ISSN 2070--2515

FAO
PLANT
PRODUCTION
AND PROTECTION
PAPER

124

Food and Agriculture
Organization of the
United Nations

World Health
Organization

Pesticide residues in food 1993

Joint FAO/WHO Meeting on Pesticide Residues

EVALUATIONS

1993

PART I - RESIDUES

Rome, 1994

CONTENTS

[1] *=first evaluation; **=re-evaluation in CCPR periodic review programme

1993 FAO/WHO JOINT MEETING ON PESTICIDE RESIDUES

Geneva, 20-29 September 1993

PARTICIPANTS

WHO Expert Group on Pesticide Residues

Prof. U.G. Ahlborg
Division of Environmental Toxicology
Karolinska Institutet
Institute of Environmental Medicine
Stockholm, Sweden

Dr A.L. Black
Medical Services Adviser in Toxicology
Department of Health, Housing, Local Government
and Community Services
Canberra, Australia

Professor J.F. Borzelleca (Chairman)
Pharmacology, Toxicology
Medical College of Virginia
Virginia Commonwealth University
Richmond, Virginia, USA

Mr D.J. Clegg
Ontario, Canada

Dr Penelope A. Fenner-Crisp (Rapporteur)
Director, Health Effects Division
Office of Pesticide Programs (H7509C)
US Environmental Protection Agency
Washington, D.C., USA

Dr E.M. den Tonkelaar
Toxicologist
Toxicology Advisory Centre
National Institute of Public Health and Environmental Protection
Bilthoven, The Netherlands

Dr Peipei Yao
Professsor of Toxicology
Institute of Occupational Medicine, CAPM
Ministry of Public Health
Beijing, China

FAO Panel of Experts on Pesticide Residues in Food and the Environment

Dr. D.C. Abbott
AOAC International, Liaison Representative
Ashtead, Surrey, United Kingdom

Dr. A. Ambrus (Rapporteur)
Budapest Plant Health & Soil Conservation Station
Budapest, Hungary

Dr. Ursula Banasiak
Stahnsdorfer Damm 81
14532 Kleinmachnow
Germany

Mr. D.J. Hamilton
Senior Principal Scientist
Agricultural Chemistry
Queensland Department of Primary Industries
Brisbane, Queensland, Australia

Mr. N.F. Ives (Vice-Chairman)
Health Effects Division (H7509C)
US Environmental Protection Agency
Washington, D.C., USA

Dr. J.-R. Lundehn
Federal Biological Research Centre for Agriculture and Forestry (BBA)
Braunschweig, Germany

Mr. K. Voldum-Clausen
VarlÖse, Denmark

Ms. H.F. Yeoh
Residue Section
Pesticides Control Branch
Dept. of Agriculture
Kuala Lumpur, Malaysia

Secretariat

Mrs P.H. Arentzen (WHO Temporary Adviser)
National Institute of Public Health and Environmental Protection
Bilthoven, The Netherlands

Dr Elisabeth Bosshard (WHO Temporary Adviser)
Federal Office of Public Health
Division of Food Science
Institute of Toxicology
Schwerzenbach, Switzerland

Mrs M. Caris (WHO Temporary Adviser)
Toxicological Evaluation Division, Bureau of Chemical Safety
Health and Welfare Canada
Ottawa, Ontario, Canada

Dr K. Fujimori (WHO Temporary Adviser)
Division of Pharmacology
Biological Safety Research Center
National Institute of Health Sciences
Tokyo, Japan

Dr H. Galal-Gorchev
International Programme on Chemical Safety
World Health Organization

Geneva, Switzerland

Dr J.L. Herrman (WHO Joint Secretary)
International Programme on Chemical Safety
World Health Organization
Geneva, Switzerland

Dr A. Kocialski (WHO Temporary Adviser)
Health Effects Division
Office of Pesticide Programs (H7509C)
US Environmental Protection Agency
Washington, D.C., USA

Dr Jens-JÖrgen Larsen (WHO Temporary Adviser)
Head, Department of General Toxicology
Institute of Toxicology
National Food Agency of Denmark
SÖborg, Denmark

Prof. M. Lotti (WHO Temporary Adviser)
Università di Padova
Istituto di Medicina del Lavoro
Padova, Italy

Mr A.F. Machin (FAO Consultant)
London, UK

Dr T.C. Marrs (WHO Temporary Adviser)
Medical Toxicology and Environmental Health
Department of Health
London, UK

Dr D. McGregor
Unit of Carcinogen Identification and Evaluation
International Agency for Research on Cancer
Lyon, France

Dr A. Moretto (WHO Temporary Adviser)
Università di Padova
Istituto di Medicina del Lavoro
Padova, Italy

Dr G. Moy
Food Safety
World Health Organization
Geneva, Switzerland

Mr W.J. Murray (FAO Joint Secretary)
Plant Protection Service
Plant Production & Protection Division
Food and Agriculture Organization of the United Nations (FAO)
Rome, Italy

Dr R. Plestina
International Programme on Chemical Safety
World Health Organization

Geneva, Switzerland

Dr A. Takanaka (WHO Temporary Adviser)
Head, Division of Pharmacology
Biological Safety Research Center
National Institute of Health Sciences
Tokyo, Japan

Dr W.H. van Eck
Chairman, Codex Committee on Pesticide Residues
Food and Product Safety Division
Ministry of Welfare, Health and Cultural Affairs
Rijswijk, The Netherlands

Dr G. Vettorazzi (WHO Temporary Adviser)
International Toxicology Information Centre (ITIC)
San Sebastian, Spain

Mr M. Walsh (WHO Temporary Adviser)
Principal Administrator EEC
Commission of the European Communities
Législation des produits végétaux et de nutrition animale
Brussels, Belgium

Mr M. Watson (WHO Temporary Adviser)
Head, Risk Evaluation Branch
Pesticide Safety Directorate
Ministry of Agriculture, Fisheries and Food
Rothamsted, Harpenden, UK

Dr Y. Yamada
Food Standards Officer
Joint FAO/WHO Food Standards Programme Group
Food and Agriculture Organization of the United Nations (FAO)
Rome, Italy

ABBREVIATIONS WHICH MAY BE USED

AChE	acetylcholinesterase
ADI	acceptable daily intake
AFI(D)	alkali flame-ionisation (detector)
ai	active ingredient
ALAT	alanine aminotransferase
approx.	approximate
ASAT	aspartate aminotransferase
at. wt.	atomic weight
b.p.	boiling point
bw	body weight
c	centi - (x 10^{-2})
°C	degree Celsius (centigrade)
CCPR	Codex Committee on Pesticide Residues
ChE	cholinesterase
cm	centimetre
CNS	central nervous system
cu	cubic
cv	coefficient of variation
DFG	Deutsche Forschungsgemeinschaft
DL	racemic (optical configuration, a mixture of dextro- and laevo-; preceding a chemical name)
DP	dustable powder
DS	powder for dry seed treatment
EBDC	ethylenebis(dithiocarbamate)
EC	(1) emulsifiable concentrate (2) electron-capture [chromatographic detector]
ECD	electron-capture detector
EMDI	estimated maximum daily intake
EPA	Environmental Protection Agency
ERL	extraneous residue limit
ETU	ethylenethiourea
F_1	filial generation, first
F_2	filial generation, second
f.p.	freezing point
FAO	Food and Agriculture Organization of the United Nations
FDA	Food and Drug Administration
FID	flame-ionization detector
FPD	flame-photometric detector
g	gram
ìg	microgram
GAP	good agricultural practice(s)
GC-MS	gas chromatography - mass spectrometry
G.I.	gastrointestinal
GL	guideline level
GLC	gas-liquid chromatography
GPC	gel-permeation chromatography
GSH	glutathione

h	hour(s)
ha	hectare
Hb	haemoglobin
hl	hectolitre
HPLC	high-performance liquid chromatography
IBT	Industrial Bio-Test Laboratories
i.d.	internal diameter
i.m.	intramuscular
i.p.	intraperitoneal
IPCS	International Programme on Chemical Safety
IR	infrared
IRDC	International Research and Development Corporation (Mattawan, Michigan, USA)
i.v.	intravenous
JMPR	Joint FAO/WHO Meeting on Pesticide Residues (Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and a WHO Expert Group on Pesticide Residues)
k	kilo- (x 10^3)
kg	kilogram
l	litre
LC	liquid chromatography
LC_{50}	lethal concentration, 50%
LD_{50}	lethal dose, median
LOAEL	lowest observed adverse effect level
LOD	limit of determination (see also "*" at the end of the Table)
LSC	liquid scintillation counting or counter
m	metre
MFO	mixed function oxidase
mg	milligram
ìg	microgram
ìm	micrometre (micron)
min	minute(s)
ml	millilitre
MLD	minimum lethal dose
mm	millimetre
M	molar
mo	month(s)
m.p.	melting point
MRL	Maximum Residue Limit (this term replaces "tolerance")
MTD	maximum tolerated dose
n	normal (defining isomeric configuration)
NCI	National Cancer Institute (United States)
NMR	nuclear magnetic resonance
no.	number
NOAEL	no-observed-adverse-effect level
NOEL	no-observed-effect level
NP(D)	nitrogen-phosphorus (detector)
NTE	neuropathy target esterase

o	*ortho* (indicating position in a chemical name)
OP	organophosphorus pesticide
p	*para* (indicating position in a chemical name)
PHI	pre-harvest interval
ppm	parts per million (Used only with reference to the concentration of a pesticide in an experimental diet. In all other contexts the terms mg/kg or mg/1 are used).
PT	prothrombin time
PTT	partial thromboplastin time
PTU	propylenethiourea
RBC	red blood cell
s.c.	subcutaneous
SC	suspension concentrate (= flowable concentrate)
SD	standard deviation
SE	standard error
SG	water-soluble granule
SL	soluble concentrate
SP	water-soluble powder
sp./spp.	species (only after a generic name)
sp gr	specific gravity
sq	square
t	tonne (metric ton)
T_3	tri-iodothyronine
T_4	thyroxine
TADI	Temporary Acceptable Daily Intake
tert	tertiary (in a chemical name)
TLC	thin-layer chromatography
TMDI	theoretical maximum daily intake
TMRL	Temporary Maximum Residue Limit
TPTA	triphenyltin acetate
TPTH	triphenyltin hydroxide
TSH	thyroid-stimulating hormone (thyrotropin)
UDMH	1,1-dimethylhydrazine (unsymmetrical dimethylhydrazine)
USEPA	United States Environmental Protection Agency
USFDA	United States Food and Drug Administration
UV	ultraviolet
v/v	volume ratio (volume per volume)
WG	water-dispersible granule
WHO	World Health Organization
wk	week
WP	wettable powder
wt	weight
wt/vol	weight per volume
w/w	weight per weight
yr	year
<	less than
≤	less than or equal to
>	greater than

≥ greater than or equal to

* (following residue levels, e.g. 0.01* mg/kg): level at or about the limit of determination

INTRODUCTION

The report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues, held in Geneva, 20-29 September 1993, contains a summary of the evaluations of residues in foods of the various pesticides considered as well as information on the general principles followed by the Meeting. The present document contains summaries of the residues data considered, together with the recommendations made.

The Evaluations are issued in two parts:

Part I: Residues (by FAO)
Part II: Toxicology (by WHO)

For those interested in both aspects of pesticide evaluation, not only both parts but also the reports containing summaries of residue and toxicological considerations will be available. Special attention is drawn to Annex I containing updated ADIs, MRLs and temporary ADIs and MRLs, which also appears in full as part of the report of the Meeting.

Some of the compounds considered at this Meeting have been previously evaluated and reported on in earlier publications. In general only new information is summarized in the relevant monographs and reference is made to previously published evaluations, which should also be consulted. In the case of older compounds which are re-evaluated as part of the periodic review programme of the Codex Committee on Pesticide Residues (CCPR) however a comprehensive review of all available data, including data which may have previously been submitted, is carried out. Compounds evaluated for the first time are indicated by a single asterisk and those evaluated in the CCPR periodic review programme by a double asterisk in the Table of Contents.

The name of the compound appearing as the title of each monograph is followed by its Codex Classification Number in parentheses.

References to previous Reports and Evaluations of Joint Meetings are listed in Annex II.

Acknowledgements

The monographs in these Evaluations were prepared by the following participants in the 1993 JMPR for the FAO Panel of Experts on Pesticide Residues in Food and the Environment: Dr. D.C. Abbott, Dr A. Ambrus, Dr. U. Banasiak, Mr D.J. Hamilton, Mr N.F. Ives, Dr J.-R. Lundehn, Mr A.F. Machin, Mr B. Murray, Mr K. Voldum-Clausen and Ms. H.F. Yeoh.

Note: Any comments on residues in food and their evaluation should be addressed to the:

Pesticide Residue Specialist
Plant Protection Service
Plant Production and Protection Division
Food and Agriculture Organization
Via delle Terme di Caracalla
00100 Rome, Italy

2. GENERAL CONSIDERATIONS

2.1 AMENDMENTS TO THE AGENDA

Benomyl (069)*, Carbendazim (072)*, Dimethoate (027), Diquat (031)*, Ethephon (106)*, Ethion (034)*, Iprodione (111)*, Profenofos (171), Propiconazole (160) and Thiophanate-methyl (077)* were originally scheduled for review by the FAO Panel. The draft evaluations of these compounds had been completed by the Panel members, but they could not be given adequate consideration in the time available to the Meeting. In the interests of maintaining the high standard of the evaluations the review of these compounds was postponed to the 1994 Joint Meeting.

The toxicological evaluation of captan was postponed until 1995 when folpet, a closely-related compound, will be reviewed. Chlorpropham was not evaluated owing to lack of data.

* Periodic review compounds

2.2 RISK ASSESSMENT PROCEDURES USED BY THE JMPR

The Codex Alimentarius Commission, at its 20th Session, considered a working paper entitled *Risk assessment procedures used by the Codex Alimentarius Commission, and its subsidiary and advisory bodies* (ALINORM 93/37). The expert committees, including the JMPR, that advise the Commission were characterized as forming a bridge between those who carry out scientific research and the risk managers (usually Codex Committees), and it was stated that the expert committees are ideally suited to perform risk assessment.

In its discussions, the Commission reiterated the importance of increasing the transparency of the work of the Joint Meeting in, for example, the identification of hazards and the choice of safety factors used in safety evaluations. The Commission noted that the characterization of exposure relative to dietary intake suffered from a lack of information and consistent methods of risk assessment, and needed to be improved. In addition, the needs of special "at risk" groups should be taken into account. It was also recommended that more attention be given to quantifying uncertainty in specific risk assessments carried out by the Expert Committees that advise the Codex Alimentarius Commission. The Commission recommended that the paper should be brought to the attention of the JMPR.

In responding to the report of the Commission, the Joint Meeting emphasized the importance of maintaining a distinction between risk assessment, which is performed by the FAO/WHO expert committees, and risk management. In this way the Codex Alimentarius Commission will have unbiased scientific advice on which to base its decisions.

Recommendations have been made at a number of conferences in recent years that FAO and WHO expert committees should be more "transparent". The Joint Meeting has responded by describing in more detail the data that it is reviewing and explaining more clearly the basis for its decisions. With regard to toxicological assessments the safety factors that are applied to the NOAELs in the studies that are reviewed represent the uncertainty inherent in the assessments and in extrapolating results from one species to another. WHO Environmental Health Criteria (EHC) 104 [i] explains the basis of the standard safety factor, and when this is not used the reasons are explained in the individual evaluations. FAO is in the process of producing a document that describes the scientific bases for its assessments.

The Joint Meeting recognized the need for improved dietary intake data, and stressed that it is dependent upon Member States to provide them. Better intake data for specific population groups, such as infants and children, are required. On the assessment side, the International Programme on Chemical Safety (IPCS) has published an EHC document that

outlines special approaches that should be taken for infants and children [2], and other organizations have considered this issue more recently [3,4].

Intake predictions have been made in recent years using the *Guidelines for predicting dietary intake of pesticide residues* [5]. Although these guidelines are generally accepted and remain valid, IPCS is considering the possibility of convening a consultation to revise them in the light of the experience gained in using them, taking into account suggestions that have been made by the Codex Committee on Pesticide Residues (CCPR) and others. The Joint Meeting stressed that theoretical predictions of maximum dietary intake should be used only as a screen to identify those pesticides whose intake may have the potential to exceed the ADI, and that theoretical predictions should not be a substitute for more realistic intake estimates based upon actual residue levels and food intake at the national and/or local level, when such information is available.

With reference to paragraph 68 in the report of the Codex Alimentarius Commission (ALINORM 93/40) the Meeting stressed that MRLs for both pesticides and veterinary drugs are based on different data bases from those on which ADIs are based, and that there is no direct relationship between them. The purpose of MRLs is to ensure that good agricultural practice in the use of pesticides or good practice in the use of veterinary drugs has been followed. They cannot be used for making realistic estimates of pesticide or veterinary drug intake.

The Meeting agreed with recommendation 6 of the working paper (ALINORM 93/37) that "it is currently impracticable (and probably not necessary) to achieve uniformity in risk analysis activities between the expert groups, and between the Codex committees. However, it is important that the principles for risk assessment be the same, or where differences exist these should be justified."

The Meeting agreed on the importance of a paper such as this to improve transparency of the Codex/Expert Committee process. However, the Meeting recommended that the working paper (ALINORM 93/97) should not be published or distributed further in its present form. A revised paper should be prepared that would take into account new developments in the dynamic field of risk assessment and recent Codex developments. That paper should then be circulated to national and international organisations to give them the opportunity to comment before publication or wider distribution.

2.3 IMPROVING THE ASSESSMENT OF DIETARY RISK OF PESTICIDES

The understanding of the potential risk of exposure to pesticide residues in food is of critical interest internationally. National governments and other organizations with responsibilities or interests in this issue are continually striving to upgrade their information base so as to improve the quality of their assessments. While current risk assessment procedures make the best use of all the available data and there is no evidence that dietary exposure to pesticide residues as a consequence of approved uses presents a risk to human health in any population group, the JMPR continually seeks to be responsive to enhancement of the scientific basis of dietary risk assessment by incorporating valid new information into its deliberations.

---

[2] *Principles for evaluating health risks from chemicals during infancy and early childhood: the need for a special approach*. WHO Environmental Health Criteria, No. 59. Geneva, World Health Organization, 1986.

[3] *Similarities and differences between children and adults: implications for risk assessment*. Washington, International Life Sciences Institute Press, 1992.

[4] *Pesticides in the diets of infants and children*. National Academy of Sciences. Washington, National Academy Press, 1993.

[5] *Guidelines for predicting dietary intake of pesticide residues*. Geneva, World Health Organization, 1989.

The attention of the Meeting was drawn to a report recently released by the National Research Council of the National Academy of Sciences (NAS) in the United States [ii]. The approaches to evaluating health risks from chemicals during infancy and early childhood have also been discussed in an EHC document [6].

An overview of the recommendations put forth in the NAS report was presented to the Meeting. These recommendations addressed areas pertinent to the conduct of the assessment of the risk of pesticide exposures in food, with particular emphasis on the consideration of differences that may exist in sensitivities or exposures to pesticides between infants and young children and the adult population.

Particular emphasis was placed upon effecting improvements in: 1) the manner in which toxicity testing is performed to understand better the potential for hazard to the unborn and very young, 2) the magnitude and quality of information related to exposure (i.e. food consumption data and residue data on raw and processed foods) and 3) risk characterization methods for integrating the hazard and exposure data to reduce uncertainties in predicting risk. While these changes should improve the current ability to assess dietary exposure to pesticide residues, the need to gather and collate data on an international basis remains, especially for population groups of special concern such as infants and children. The Meeting strongly recommended that governments address this need by conducting appropriate dietary surveys.

The Meeting should remain aware of progress made with respect to these important issues of risk assessment. Information developed as a consequence of these recommendations by the NAS may be included in the data on individual pesticides submitted for consideration by future Joint Meetings.

2.4 ADIs BASED ON SHORT-TERM EXPOSURE

The *ad hoc* Working Group on Acceptances at the 25th Session of the Codex Committee on Pesticide Residues (CCPR), when considering the situation in which an ADI is based upon the NOAEL in a short-term exposure study (such as a teratogenicity study), concluded that estimates of the intakes of pesticides should be based upon different measures of food consumption from those appropriate to a long-term effect. The Working Group requested JMPR (WHO Group) to develop guidelines for assessing the toxicological significance of dietary exposure where adverse health effects may result from single or short-term exposure and to consider the definition of the ADI when it is based on an adverse health effect following single or short-term exposure (ALINORM 93/24A, Appendix III).

It should be emphasized that, even though the numerical value of the ADI is derived from the NOAEL in a specific study or studies, it is based on all the data on the pesticide. Even if the effect seen at the lowest dose above the NOAEL is an acute one (such as acetylcholinesterase inhibition) or the study on which the ADI is based is a short-term one (such as a teratogenicity study) the total database provides confidence that long-term exposure is safe. Therefore, the Joint Meeting did not consider it appropriate to use different terminology in such situations.

With those pesticides on which the ADI is based on acute effects or short-term exposure, it may be appropriate to compare the ADI with short-term intake. When predicting intakes for such pesticides, the basis of the ADI, which is explained in the report, should be consulted to ensure that the appropriate comparisons are made. The CCPR was invited to

---

6 *Principles for evaluating health risks from chemicals during infancy and early childhood: the need for a special approach*. WHO Environmental Health Criteria, No. 59. Geneva, World Health Organization, 1986.

request advice from the Joint Meeting in specific instances in which it is not clear which comparisons should be used. The IPCS will consider the types of food intake data that represent short-term intake when the *Guidelines for Predicting Dietary Intake of Pesticide Residues* are revised, and it is expected that guidance on evaluating acute exposures will be incorporated into them. Procedures for the evaluation of the potential for acute toxicity in the context of acute exposure scenarios should then be incorporated into efforts to improve toxicological evaluations in general.

2.5 CONCOMITANT PESTICIDE AND VETERINARY USES OF CHEMICALS

During the discussion of thiabendazole, the 25th Session of the CCPR requested the Joint FAO/WHO Expert Committee on Food Additives (JECFA) and the JMPR to discuss the potential for problems when a chemical is used both as a pesticide and for veterinary purposes. This resulted from a suggestion that the CCPR should use the more recent thiabendazole ADI estimate of the JECFA as opposed to the older JMPR estimate. The JECFA and the JMPR were also requested to discuss the need to provide participants in both the CCPR and the Codex Committee on Residues of Veterinary Drugs in Foods (CCRVDF) with information on the evaluations of both the JECFA and the JMPR in these situations and to propose a procedure for consideration by both Codex Committees (draft ALINORM 93/24A, paragraph 88).

The Joint Meeting recommended that the Codex Alimetarius Commission and the FAO/WHO Secretariats of the expert committees develop procedures to ensure consistency and appropriate exchange of information among the committees involved. To this end, when either the JMPR or JECFA is scheduled to review a chemical that has been reviewed by the other Committee, the Meeting also recommended that the Joint Secretaries of the JMPR and JECFA obtain and make available copies of pertinent evaluations to their respective Meetings and in advance to the appropriate data reviewers. In a similar manner when an MRL is at Codex Step 3 for discussion the Meeting recommended that the Codex Secretariat, with co-ordination and advice from the Joint Secretaries of the JECFA and the JMPR, arrange distribution of the pertinent evaluations to country contact points for both committees. Contact points should be requested to make copies available to the national committee delegation and/or other national officials responsible for providing comments.

The Meeting drew attention to MRLs related to veterinary uses (indicated in the MRL table by V) for compounds in the CCPR periodic review programme. In a periodic review, veterinary uses will be treated in the same way as all other uses and if information is not supplied, the FAO Panel will recommend withdrawal of the MRLs. The Meeting recommended, for periodic review compounds, that full information on approved veterinary uses and data from residue trials according to the approved uses, together with metabolism data in plants and animals, be included in the submission to the FAO Panel.

2.6 VALIDATION OF STUDIES

Reports of several studies that had been performed by Industrial Bio-Test (IBT) Laboratories were submitted to the present Meeting by sponsors of pesticides that were on the agenda. In most cases no indication was given as to whether they had been validated, either by the US Environmental Protection Agency (USEPA) or Health and Welfare Canada, the two organizations that have attempted to validate most of the IBT studies. Only those studies that were found at the Meeting to have been validated by either the US EPA or Health and Welfare Canada were included in the evaluations.

The Joint Meeting requested that in future sponsors provide the validation status of IBT studies by government agencies, when they submit them to the JMPR for review.

Future Joint Meetings should consider the appropriate course of action to be taken if similar situations arise in the future. Only official government validations of any suspect data will be considered.

2.7 GUIDELINES ON THE NEED FOR ANIMAL TRANSFER STUDIES IN ESTIMATING PESTICIDE MAXIMUM RESIDUE LEVELS

In the course of the 1992 Session of the Codex Committee on Pesticide Residues (CCPR) it was recognized that there was a need to define better the criteria to be considered in determining the need for animal transfer studies when estimating pesticide maximum residue levels. It was decided to request countries to submit information on national approaches in order that some general rules might be elaborated (ALINORM 93/24, para 189). Governments were requested by circular letter (CL 1992/12-PR) to submit this information. A single submission, from the United Sates, was received. The request for this information was reiterated at the CCPR in 1993 (ALINORM 93/24A, para 18) and two further responses were received, from Norway and the United Kingdom. This information was used as the basis for developing some general guidance in this area for the FAO Panel of the JMPR.

General guidance with respect to the role of livestock metabolism and feeding studies in the estimation of pesticide maximum residue levels by the JMPR may be found in the *Codex Guidelines on Pesticide Residue Trials to Provide Data for the Registration of Pesticides for the Establishment of Maximum Residue Limits* (FAO 1986). The document states that:

- "animal metabolism studies are required whenever a pesticide is applied directly to livestock, animal premises are to be treated, or residues occur in crops or crop parts used for feed;"

- "separate feeding studies are required for a ruminant and poultry whenever residues occur in items of feed for these animals."

Because of the development of more sensitive methods of analysis, low or trace pesticide residues are found with increasing frequency in animal feed items. The question arises as to the need for animal transfer studies where livestock are expected to be exposed to low levels of pesticide residues in feed.

The definition of what constitutes a "trace level" in an animal feed commodity or diet is not possible as the judgement involves a number of factors, not only the residue level. It is unlikely that agreement will be reached on a general cut-off level for residues in feed, below which livestock feeding studies will not be required. The definition of some trigger or guidance values would be consistent with the accepted practices of national governments. In addition this would alert industry to this potential need and give them the opportunity to consider these questions in preparing their submissions to the JMPR.

<u>Guideline on the need for animal transfer studies</u>

Livestock (ruminant and/or poultry) metabolism studies are required whenever a pesticide is applied directly to livestock, to animal premises or housing, to crops or commodities used in animal feed, to forage crops, or to any plant parts that could be used in animal feeds.

Livestock transfer studies are required when detectable residues are found in feed items from crop field trials reflecting the proposed use of the pesticide (maximum rate, minimum pre-harvest interval) and the metabolism studies indicate that significant residues (>0.01 mg/kg) may occur in edible tissues (taking into account estimated residue intake from the total livestock diet) and/or the potential for bioaccumulation.

Livestock transfer studies are generally required where significant residues (generally >0.1 mg/kg) occur in crops or commodities fed to animals.

When only low levels (<0.1 mg/kg) of residues are found in feed items the anticipated dietary burden and the results of the metabolism studies must be considered. The latter may indicate that residues in animal commodities would be well below detection limits and thus serve as feeding studies.

2.8 GUIDELINES ON THE PREPARATION OF DATA SUBMISSIONS TO THE FAO PANEL OF THE JMPR

The Meeting was provided with a copy of a working paper concerning the preparation of data submissions to the FAO Panel of the Joint Meeting. The proposal outlined the format and information to be provided to the Joint Meeting. This should include an index or directory of the information to be submitted and a working paper. It will be used as the basis for the submission of information to the FAO Panel of the 1994 Joint Meeting. The formats proposed for the index or directory, the working paper and the organization of the submission are intended as guides and will be amended on the basis of comments received and experience gained in their implementation. Copies of these guidelines are available from the FAO Joint Secretary.

2.9 CHOICE OF DOSE LEVELS IN TOXICOLOGY STUDIES

The Meeting was asked to evaluate a number of toxicological studies in which one or two very low dose levels were used together with one or two high dose levels. Examples in this report are a long-term study in rats (dose levels 0, 0.1, 1.5, 125 or 250 ppm diazinon), a one-year study in dogs (0, 0.1, 0.5, 150 or 300 ppm diazinon), a long-term study in rats (0, 0.5, 2.5, 5 or 125 ppm ethylenethiourea) and a one-year study in dogs (0, 30, 80, 1000 or 3000 ppm metiram).

From a scientific point of view, the selection of doses with such a wide span between the low and high levels can create problems in establishing NOAELs. One of the main factors in interpreting toxicological data is the dose-response relationship. The determination of this becomes difficult when the doses employed in the study under review are not relatively evenly spaced across the range. The determination of the NOAEL, then, is very imprecise if effects are observed at the high doses, but no effects of concern are observed at the low doses. The "true" NOAEL would be somewhere in the broad, untested region between the low and high doses. The dose range should be selected in such a way that the highest dose gives a clear effect on the target organ, the middle dose(s) give some effect(s) and the lowest dose is the NOAEL.

It should be stressed again (see 1987 JMPR report, Section 2.5) that at very high dose levels the kinetics and nature of the biotransformation of a substance may be substantially different from those that occur at low dose levels. This can also have an influence on the evaluation of toxicological data in which only very low and very high doses have been tested because different forms of toxicity might arise as a result of these differences.

2.10 MODE OF ACTION, MECHANISM OF TOXICITY AND TOXICOLOGICAL EVALUATION

The Meeting drew attention to previous comments (1986 JMPR Report, Section 2.3) on the importance of an understanding of the mode of action of a pesticide in the evaluation of its toxicity. This understanding can provide insight into the biological activity of a pesticide and assist in the assessment of its toxicity to non-target species, even when the mechanism differs. For this reason, the Meeting recommended that, where known, information on the mode of pesticidal action as well as on the mechanism of toxicity in non-target species should be made available for consideration in the overall toxicological evaluation of the pesticide.

2.11 DIETARY INTAKE OF PESTICIDE RESIDUES

Following the methods described in *Guidelines for Predicting Dietary Intake of Pesticide Residues* [iii], Theoretical Maximum Daily Intake (TMDI) calculations have been performed for the Joint Meeting by WHO. The results are summarized in Annex III. Processing factors must be reviewed before Estimated Maximum Daily Intake (EMDI) calculations can be performed on those pesticides for which the TMDI exceeded the ADI.

3. SPECIFIC PROBLEMS

3.1 MEASUREMENT OF RADIOACTIVE IODINE UPTAKE AS IN INDICATION OF THYROID FUNCTION

Study of the mechanisms of toxicity frequently involves measurements of dynamic processes. In order to avoid invalid conclusions from such measurements, they must be made at multiple and well-spaced time points.

An example is the measurement of the degree of ^{131}I-uptake by the thyroid in experiments with goitrogenic compounds (see amitrole, ethylenethiourea (ETU) and ethylenebis(dithiocarbamate)s (EBDCs). The time at which the iodine uptake is measured is very important. When the thyroid is enlarged and overactive initially (for instance after six hours) the uptake is high, but then it decreases very rapidly. After 48 hours the value is much lower than in the controls. After 24 hours, it can be lower, the same, or still higher than the control value. With a moderately activated thyroid the uptake remains, in general, above the control value (see the results of experiments carried out by den Tonkelaar and Kroes (1974) as described under short-term studies in the monograph on amitrole). In many experiments, measurements are made only after 24 hours. These can give a false impression of the uptake, especially at higher dose levels. The time of measurement should therefore always be taken into account when interpreting ^{131}I-uptake data.

3.2 EVALUATION OF THE DITHIOCARBAMATES

The Joint Meeting evaluated toxicologically four ethylenebis(dithiocarbamate)s (EBDCs), mancozeb, maneb, metiram and zineb, as well as ethylenethiourea (ETU), the major common metabolite, degradation product and contaminant of the EBDCs. A group ADI was established for the EBDCs, and an ADI was allocated to ETU. In addition, the Meeting evaluated propineb and propylenethiourea (PTU), the major metabolite of propineb. An ADI was allocated to propineb and a temporary ADI was allocated to PTU.

The Meeting also evaluated residue and analytical aspects of mancozeb, maneb and propineb, and of ETU and PTU. Because the individual dithiocarbamates cannot be distinguished by the regulatory analytical method used, the recommended MRLs for mancozeb, maneb and propineb are covered by recommendations for the dithiocarbamates as a group and are listed in Annex I under that heading.

Because residues of propineb, which has been allocated a substantially lower ADI than the group ADI for the EBDCs, cannot be distinguished by regulatory analytical methods from residues of the EBDCs, it is strongly recommended that the development of additional regulatory analytical methods should be aggressively pursued to differentiate propineb residues from those arising from the use of EBDCs.

In the absence of such a specific analytical method, consideration should be given to the use of the ADIs for propineb/PTU when comparing the toxicological significance of dietary exposure to residues of all pesticides giving rise to carbon disulphide on analysis.

Details of the evaluations may be found in Section 4 under the general heading DITHIOCARBAMATES (4.15) and under the individual compounds ETHYLENETHIOUREA (4.18), MANCOZEB (4.28), MANEB (4.29), METIRAM (4.30), PROPINEB (4.35), PROPYLENETHIOUREA (4.36) and ZINEB (4.39).

4. EVALUATION OF DATA FOR ACCEPTABLE DAILY INTAKE FOR HUMANS AND MAXIMUM RESIDUE LIMITS

4.1 ALDICARB (117)

RESIDUE AND ANALYTICAL ASPECTS

At the 24th Session of the CCPR (1992) a proposed MRL of 0.05 mg/kg for aldicarb in Brussels sprouts was held as a TMRL at step 7B awaiting more data from supervised trials.

Data from 5 sites in the UK were available to the Meeting. Aldicarb was applied at 3.8, 5.1 and 7.7 g ai/100 m row: 5.1 g/100 m row is the registered use in the UK. Residues were <0.01-0.03 mg/kg except in one trial with the application rate of 5.1 g/100 m, where the level was 0.1 mg/kg. In another trial with the dosage of 7.7 g ai/100m the residue was 0.09 mg/kg.

The Meeting recommended that the TMRL of 0.05 mg/kg should be replaced by an MRL of 0.1 mg/kg.

4.2 AMITROLE (079)

TOXICOLOGY

Amitrole was evaluated in 1974 when a conditional ADI of 0-0.00003 mg/kg bw was allocated. This was extended in 1977. The compound was re-evaluated by the present Meeting in the periodic review programme. The International Programme on Chemical Safety (IPCS) has reviewed amitrole recently and will soon be producing an Environmental Health Criteria document on it.

Amitrole is rapidly and almost completely absorbed from the gastro-intestinal tract following oral administration to rats and mice. It is rapidly distributed throughout most body tissues, but with a slight accumulation in tissues with a rapid cell turnover (bone marrow, spleen, thymus, gastrointestinal tract). In a study with pregnant mice it was observed that amitrole passes through the placenta into the fetus with the same distribution pattern as in the mothers. Excretion is rapid after oral exposure. Within 24 hours, 70-95% of the administered radioactivity is excreted via the urine, mainly as the parent compound.

The metabolic transformation in mammals produces two minor metabolites detectable in the urine. The metabolism of amitrole occurs mainly in the liver and involves substitution of the hydrogen atom in the 5 position. The metabolites identified were 3-amino-5-mercapto-1,2,4-triazole and 3-amino-1,2,4-triazolyl-5-mercapturic acid.

Amitrole has a low acute toxicity when tested in several species by various routes of administration. In old studies, amitrole was reported to have slight irritating effects on the skin and eyes. Evidence of a moderate sensitizing potential was observed in a Magnusson-Kligman test but not in a Klecak open epicutaneous test. WHO has classified amitrole as unlikely to present acute hazard in normal use.

Oral exposures up to four weeks in rats revealed that effects on the thyroid occurred at levels $\geq$ 60 ppm in the diet or 104 ppm in drinking water. No effects were observed at 30 ppm in the diet (equivalent to 3 mg/kg bw/day) or 10 ppm in drinking water (equivalent to 1.3 mg/kg bw/day). Furthermore, it was shown that after a recovery period the effects on the thyroid were reversible.

In a 30-day study in mice at concentrations in drinking water of 0, 5000, 10000 or 20000 mg/l, histopathological changes in the liver were observed at all dose levels.

Several short-term oral studies were performed with rats. These were mainly focused on the effects on the thyroid, as this is the target organ in rats.

Only two oral studies were suitable for assessment. In one, male rats were exposed to dietary concentrations of 0, 2, 10 or 50 ppm for 13 weeks or to 0, 0.25 or 0.50 ppm for 11 weeks. The NOAEL was 2 ppm (equivalent to 0.1 mg/kg bw/day), based on histological changes in the thyroid (appearance of follicular cells, contents of colloid and capillary density). Decreases in protein-bound iodine (PBI) were not considered to be biologically significant.

The other study consisted of four short-term experiments in female rats. Dietary concentrations in one experiment were 0, 2, 20 or 200 ppm with exposure for six weeks, in two subsequent experiments 0, 20, 50 or 200 ppm with exposure for six or 13 weeks and in the fourth experiment 0, 20, 50, 200 or 500 ppm with exposure for six weeks. From these four experiments the overall NOAEL was 2 ppm (equivalent to 0.1 mg/kg bw/day), based on increased iodine uptake (shortly after injection), increased thyroid weight and histopathological changes of the thyroid (goitre and clearly activated thyroids).

From several short-term studies in rats with administration in drinking-water, slight effects on the thyroid (moderate stimulation of the thyroid epithelium) were seen at the lowest concentration tested, 50 ppm.

In a one-year study in dogs, no effects on the thyroid were observed at the highest dose tested (12.5 mg/kg bw/day). The only effect observed at this level was pale-coloured pancreases. The test, however, was performed with a small number of animals.

Long-term and/or carcinogenicity studies have been performed in mice, rats, and golden hamsters. Studies in mice were focused on induction of liver and thyroid tumours. In a carcinogenicity study in mice with only one but a very high dose level (1000 mg/kg bw/day by gavage), survival time was significantly reduced and liver and thyroid tumours were observed in all treated mice. A slight increase in the incidence of liver tumours was observed in a special carcinogenicity study in which offspring were treated for a period of 90 weeks at a level of 500 ppm in the diet.

In a carcinogenicity study in mice at levels of 0, 1, 10 or 100 ppm in the diet, an increased incidence of tumours was not observed. In this study, a thyroid function test was also performed with a small number of animals. At 100 ppm an increase in thyroid weight and in iodine accumulation in the thyroid was observed. The NOAEL was 10 ppm (equivalent to 1.5 mg/kg bw/day).

In a carcinogenicity study in rats with levels of 0, 1, 10 or 100 ppm in the diet, a slight decrease in survival time, an increase in the incidence of thyroid tumours and an increase in the incidence of (mainly benign) pituitary tumours were observed at 100 ppm. In this study, a thyroid function test was also performed with a small number of animals. At 100 ppm, thyroid weight was increased during the whole study period as was the percentage accumulation of radioiodine in the thyroid. The NOAEL was 10 ppm (equivalent to 0.5 mg/kg bw/day).

In another limited long-term study in rats, the NOAEL was 10 ppm (equivalent to 0.5 mg/kg bw/day), based on thyroid hyperplasia. A clearly enhanced thyroid tumour incidence was found at 50 and 100 ppm. In this study, animals suffered from apparent respiratory infection.

In a third study in rats, thyroid hyperplasia and thyroid tumours were observed in animals fed 100 ppm (during the first 40 weeks of the 115-120 week study, the dose level was 5 ppm). In rats treated at pulsed intervals (alternate four week periods) at levels of 60 ppm (first 3 ppm) and 200 ppm (first 10 ppm) thyroid tumours were also observed. Slight thyroid hyperplasia was also observed at the lowest dose level of 20 ppm (first 1 ppm; intermittent dosing regimen). An NOAEL could not be established.

In a carcinogenicity study in Syrian hamsters at dietary concentrations of 0, 1, 10 or 100 ppm, the NOAEL was 10 ppm (equivalent to 1 mg/kg bw/day), based on decreased body-weight gain and increased mortality. No effects on the thyroid were observed at 100 ppm. There was no evidence of carcinogenic potential.

A well performed reproduction study was not available. From a limited study in rats at dietary concentrations ranging from 25 to 1000 ppm, effects on reproductive capability were observed at 500 ppm and above. Reduction of liver weight and thyroid hyperplasia were the most sensitive effects observed at the lowest dose level (25 ppm, equivalent to 1.3 mg/kg bw/day).

In a teratogenicity study, rats were exposed by gavage at doses of 0, 100, 300 or 1000 mg/kg bw/day on days 6 to 15 of gestation. No effects were observed in this study. The NOAEL for maternal toxicity and embryo/fetotoxicity was 1000 mg/kg bw/day.

In another teratogenicity study, rats were exposed by gavage at doses of 0, 100, 500 or 1000 mg/kg bw/day. Slight maternal toxicity (reduced weight gain and food consumption and increased thyroid weights) was observed at doses of 500 and 1000 mg/kg bw/day. Reduced fetal body weight/litter and reduced skeletal ossifications were observed in the high-dose group. Increased incidences of enlarged and/or dark thyroids were seen in fetuses at 500 and 1000 mg/kg bw/day. The NOAEL for maternal toxicity and embryo/fetotoxicity was 100 mg/kg bw/day. Amitrole was considered not to be teratogenic in rats at dose levels up to 1000 mg/kg bw/day.

In a teratogenicity study in rabbits the animals were exposed by gavage to dose levels of 0, 4, 40 or 400 mg/kg bw/day. Decreased weight gain during the gestation period was observed at 40 and 400 mg/kg bw/day and increased liver weight at 400 mg/kg bw/day. A dose-related increased incidence of abortions was observed in all treated groups. Embryo/fetotoxicity were observed at 40 and 400 mg/kg bw/day. Increased incidences of irreversible structural changes were also found at these dose levels, which involved mainly the head and limbs. The NOAEL for maternal toxicity, embryo/fetotoxicity and teratogenicity was 4 mg/kg bw/day.

In a dermal teratogenicity study in rabbits at dose levels of 0, 1000, 1500 or 2000 mg/kg bw/day, maternal toxicity (decreased body weight and food consumption, thin appearance and anorexia) was observed at 2000 mg/kg bw/day. At this level, irreversible structural changes (anencephaly and microphthalmia) were observed. The NOAEL for maternal toxicity, embryo/fetotoxicity and teratogenicity after dermal exposure was 1500 mg/kg bw/day.

Amitrole has been tested adequately in series of *in vitro* and *in vivo* genotoxicity assays. Positive responses were obtained in a number of mutation assays in bacteria, recombinogenicity assays in yeast and some mammalian cell assays for mutation, sister-chromatid exchange and cell transformation. No genotoxicity was demonstrated *in vivo*. The Meeting concluded that the genotoxic potential of amitrole was equivocal.

Amitrole is a goitrogen in mice, rats and sheep but not in Syrian hamsters, dogs, or cattle at the doses that have been tested. The mechanism of thyroid toxicity involves inhibition of thyroid peroxidase. This inhibition results in decreases in circulating levels of T_4 and T_3, which stimulate the pituitary to increase secretion of TSH which in turn may cause thyroid hypertrophy, hyperplasia and neoplasia. Threshold doses have been identified in the sensitive species. Amitrole is not genotoxic in *in vivo* assays.

The Meeting withdrew the conditional ADI and established a temporary ADI, based on the NOAEL of 0.5 mg/kg bw/day in the 24-month dietary study in rats, pending the evaluation of the required data (see below). Because of the inadequacy of the existing data a safety factor of 1000 was used.

A toxicological monograph was prepared, summarizing the data received since the previous evaluation and containing relevant data from the previous monograph and monograph addendum on amitrole.

TOXICOLOGICAL EVALUATION

Level causing no toxicological effect

Mouse:	10 ppm, equivalent to 1.5 mg/kg bw/day	(18-month study)
Rat:	10 ppm, equivalent to 0.5 mg/kg bw/day	(24-month study)
	100 mg/kg bw/day	(teratogenicity study)
Hamster:	10 ppm, equivalent to 1 mg/kg bw/day	(18-month study)
Dog:	12.5 mg/kg bw/day	(12 month study)

Estimate of temporary acceptable daily intake for humans

0-0.0005 mg/kg bw

Studies without which the determination of a full ADI is impracticable

Results to be submitted to WHO by 1996 (all known to have been initiated):

1. Two-generation reproduction study in rats.
2. One-year study in dogs.
3. Oral teratogenicity study in rabbits.
4. Metabolism study in rats.

Studies which will provide information valuable in the continued evaluation of the compound

1. Further observations in humans.
2. Comparative biotransformation (including humans).
3. Clarification of the genotoxic potential of amitrole.

RESIDUE AND ANALYTICAL ASPECTS

Amitrole was evaluated by the JMPR in 1974 and 1977 and is included in the CCPR periodic review programme. A conditional ADI was allocated in 1974 and confirmed in 1977. An MRL for raw agricultural commodities was recommended at the limit of determination in 1974, but the 17th Session of the CCPR (1987) recommended that the MRL should be withdrawn and replaced by a note that uses of amitrole should be restricted to those where residues in food would not be expected to occur.

Information on registered uses was received from Australia, Belgium, Canada, France, Germany, The Netherlands, Portugal and Spain. The compound is applied to the ground and directly on to weeds and usually with a long PHI, so residues should not be detectable in crops grown on treated soil.

The Meeting received only one report from supervised trials, but was informed that new trials on apples, grapes, and pears were in progress. Most of the studies would be supplied to the JMPR in the near future.

Several reports were available from studies on the metabolism or degradation of amitrole (aminotriazole) in plants, animals and soil. In plants after direct applications to the leaves or stem the main metabolite was aminotriazolylalanine, 3-(3-amino-1,2,4-triazol-1-yl)-D-alanine. Two other metabolites were found, but not identified. The same metabolites were present in rats. After treatment of the soil surrounding plants only small amounts of aminotriazole and its metabolites were translocated to the plant. In apples residues of the parent compound and the metabolite triazolylalanine were undetectable or very low: when present the compounds were in both free and conjugated forms. In cell suspension cultures from apples 3,5-dihydroxy-1,2,4-triazole was produced.

In soil rapid degradation occurs with CO_2 as the main degradation product. Degradation in soil is strongly influenced by the presence of micro-organisms, and does not occur under anaerobic conditions. From laboratory experiments it was possible to propose a degradation scheme for amitrole in soil. The ring is opened after metabolism to 5-hydroxyaminotriazole, and via cyanamide the compound is decomposed to CO_2 and ammonia. Because of the rapid degradation only small amounts of aminotriazole are leached into soil. Leaching is most pronounced in sandy soil with a low content of organic material.

New analytical methods for the determination of residues of amitrole have been developed using gas chromatography with a nitrogen-specific detector, thin layer chromatography and high performance liquid chromatography with fluorescence or electrochemical detection. The limits of determination are 0.01 - 0.02 mg/kg for residues in fruit, vegetables and soil.

A complete re-evaluation of amitrole has not been possible because new data from supervised trials were not available. Although the registered uses reported to the Meeting are similar to the application conditions in some supervised trials examined by the JMPR in 1974, the data from the trials currently in progress should be taken into consideration. No reports from studies of storage stability were available, but the Meeting was informed that the results of such studies will be available in 1995. Reports of animal transfer studies were also lacking, but as residues of amitrole in crops are obviously very low and usually below the limit of determination, there is a very limited need for such studies.

No Codex residue limits are established for amitrole in food commodities. The Meeting is aware that the compound is in use. A realistic limit of determination for the general monitoring of amitrole would be 0.05 mg/kg.

FURTHER WORK OR INFORMATION

Desirable

1. Residue data from supervised trials on apples, pears and grapes known to be in progress.

2. Reports from experiments on the storage stability of amitrole known to be in progress.

4.3 AZINPHOS-METHYL (002)

RESIDUE AND ANALYTICAL ASPECTS

Azinphos-methyl was evaluated in 1965 and several times since. In 1991 a re-evaluation resulted in recommendations to withdraw or change several MRLs. New residue data from trials carried out according to GAP were required for apricots, black currants, citrus fruits, strawberries, kiwifruit and bulb and spring onions. The data from trials according to GAP on apricots, citrus fruits and kiwifruit were so limited that withdrawal of the existing MRLs was proposed, and this recommendation was accepted by the 25th Session of the CCPR (1993). Residue data from trials according to GAP on blueberries, cherries and grapes were also desirable as the data available were from only one country, the USA.

The Meeting received summarized residue data from Spain from trials on mandarins and oranges according to registered use in Spain. Residue data on apricots were also available from Spain, but the dosage used was about 3 times the registered rate.

Residue data from trials on cherries were received from Denmark, but samples were taken more than 50 days after the last treatment whereas the registered PHI in Denmark is 21 days. Information was received on several trials on cherries carried out in the USA, which supported the MRL of 2 mg/kg proposed by the 1991 JMPR. The Meeting was informed that data from trials on grapes in Germany and Italy, including processing studies, would be available in 1995.

At the 1991 JMPR a temporary residue limit was proposed for wheat straw and fodder. As no supplementary data were received the Meeting proposes that the temporary limit should be withdrawn.

FURTHER WORK OR INFORMATION

Desirable

1. Detailed information from trials on citrus fruits carried out in Spain.

2. Residue data from trials on citrus fruits from other countries.

4.4 BENALAXYL (155)

RESIDUE AND ANALYTICAL ASPECTS

Benalaxyl was first reviewed for residues by the 1986 JMPR, which estimated Guideline Levels and listed desirable information. Guideline Levels were changed to MRLs when an ADI was allocated by the 1987 JMPR. Over several years various limits (in particular on grapes) were questioned at the CCPR. Submission of additional unspecified data has been promised. Several submissions were made to the Meeting in response to requests of the 1986 JMPR or concerns expressed at the CCPR, some with and some without the detailed reports.

Grapes. The 0.5 mg/kg limit estimated by the 1986 JMPR was lowered to 0.2 mg/kg by the 1990 CCPR. Although no outstanding issues remained, extensive recent data from the use of benalaxyl on grapes were provided to the Meeting. Much of the summarized information could not readily be related to the more detailed reports provided owing to its code numbering format, except in the case of the Italian data.

Most of the submitted grape data do not closely reflect reported current GAP. In particular, most of the results (except at day 0) were at intervals significantly longer than the reported 7-day Italian GAP PHI (the shortest non-0-day PHI was 11 days and most PHIs were longer). The few results within GAP (GAP rates and $\geq$ 11 day PHI) were consistent with the current 0.2 mg/kg CXL. However, extrapolation from Italian residue decline curves strongly suggests that residues exceeding 1 mg/kg are likely to occur from Italian GAP at a 7-day PHI. Extrapolation of previously provided and additional data from German supervised trials also suggests that residues may approach 1 mg/kg when related to Italian GAP. However, the Meeting was informed that the manufacturer is to request that the 7-day Italian PHI be revised to 10-28 days. With that revision residues would be within the current limit. The Meeting was also informed that applications are only on small immature fruit.

Residues in must and wine were ≤0.02 mg/kg, mostly ≤0.01 mg/kg. No data were provided for grape pomace, a possible animal feed item.

Potatoes. The adequacy of previously submitted analytical methods to support the current 0.01 mg/kg CXL for potatoes has repeatedly been questioned at the CCPR. The Meeting concluded (see below) that 0.02 mg/kg is a reasonable limit of determination for the new analytical method reported, and noted that the limit of "detection" for much of the additional summary data reported (but not reviewed) is 0.02 mg/kg. The Meeting therefore proposed that the MRL should be increased to 0.02 mg/kg.

In addition to substantial supervised trials data for grapes, the Meeting received summary data on benalaxyl residues in cucumber, potatoes and tomatoes. Because summary data without accompanying detailed reports are not suitable for estimating maximum residue levels the Meeting did not review these summaries apart from considering the limit of determination for potatoes. The Meeting was informed that the detailed reports would be submitted for review at a future meeting.

The Meeting also received a limited response to the 1986 request for additional information on levels of metabolites in plants. Noting unsuccessful efforts to analyze the GX1A and GX1B glucoside metabolites in crops, the Meeting was informed of a method for the determination of these metabolites in white wine (unsuccessful in red wine). No data were provided except the results of recovery studies.

Residues in animal products. In response to a JMPR request for information on residues in cattle and pigs the manufacturer expressed the view that metabolism studies and the low residues expected in feed items would make residues in meat from cattle and pigs unlikely. Since (1) significant residues could occur: they have been found in the offal of goats and hens in metabolism studies (e.g. up to 1 and 1.8 mg/kg in the liver of goats and hens fed at 50 ppm in the feed); (2) information on the possible concentration of residues in feed items derived from processing was lacking; and (3) the duration of the metabolism study feeding periods (7 days for cattle, 14 days for hens) was relatively short, the Meeting could not with certainty come to the same conclusion. While the Meeting agreed that residues in animals would be likely to be low, there is the potential for finite residues.

Processing. Apart from data on residues of benalaxyl in wine and must, no information was provided in response to the 1986 JMPR request for information on the effect of processing on residues in crops. Processing studies would also provide insight into the likelihood of residues in animal products. The Meeting was informed that processing studies would be scheduled for 1994.

Analytical methods. A published analytical method based on acetone extraction, liquid-liquid partitioning, alumina clean-up and GLC with NPD detection was provided in response to CCPR concerns that no published enforcement method was available and doubt concerning the reported 0.01 mg/kg limit of determination in potatoes in the method previously reviewed. The published method was tested on several crops, wine, must and water. Recoveries of $\geq$95% were reported.

The Meeting received excellent documentation of what appears to be a suitable enforcement method. While the reported limits of determination (0.01 mg/kg in crops and 0.01 mg/l in wine and must) may be attainable in the author's laboratory, on the basis of sample chromatograms, reported control values and fortification levels, the Meeting concluded that a more realistic limit of determination for Codex purposes would be of the order of 0.05 mg/kg in crops (0.02 mg/kg in potatoes) and 0.05 mg/l in wine and must. Detection is possible at lower levels.

A description of an analytical method for the determination of the glucoside metabolites GX1A and GX1B in white wines was also provided (chromatograms suggest that routine analyses down to 0.05 mg/kg should be feasible). An analytical method based on column chromatography clean-up and GLC with AFID detection for determining benalaxyl in wine was also provided, with the capability of analyses at 0.02 to 0.05 mg/kg.

FURTHER WORK OR INFORMATION

Desirable

1. Submission of revised Italian GAP for grapes for the next scheduled review.

2. Submission of detailed reports of trials on cucumber, potato and tomato corresponding to summary data provided to the 1993 Meeting, reported in a manner to permit easy comparison of the summary data and the detailed reports and in the working language of the Meeting.

3. Submission on completion of processing studies which are scheduled for 1994.

(From 1986 JMPR)

4. Information on residues in meat from pigs and cattle fed a diet containing benalaxyl.

4.5 BROMOPROPYLATE (070)

TOXICOLOGY

Bromopropylate was previously evaluated by the Joint Meeting in 1973, when an ADI of 0-0.008 mg/kg bw was allocated. That Meeting recommended further desirable work as follows: 1) studies to elucidate the effects on survival rate of rats in long-term feeding studies; 2) long-term studies in a second animal species; 3) studies on the effects of bromopropylate on the liver. The results of these studies and additional data were submitted for the present evaluation.

After oral administration of [U-^{14}C]phenyl bromopropylate, most of the radioactivity was eliminated in the faeces, with lower amounts of radioactivity excreted in the urine. The routes of elimination were sex- dependent.

Bromopropylate was metabolized preferentially by cleavage of the isopropyl ester linkage, and to a minor extent by oxidation reactions attacking the phenyl ring and the isopropyl group.

Bromopropylate has the properties of a phenobarbital-like inducer of cytochrome P-450 in the mouse liver. In a DNA-binding study conducted with mice no radioactivity was detectable in liver DNA, indicating that bromopropylate is devoid of genotoxic potential in this organ.

Bromopropylate has a low acute toxicity in rats and rabbits. WHO has classified bromopropylate as unlikely to present acute hazard in normal use.

In a one-year study in dogs at dietary concentrations of 0, 100, 400 or 2,000 ppm, the NOAEL was 100 ppm (equal to 2.7 mg/kg bw/day), based on depressed body-weight gain at 400 ppm and above.

In a study in mice using dietary concentrations of 0, 30, 150, 1,000 or 3,000 ppm for 24 months, the NOAEL was 150 ppm (equal to 16 mg/kg bw/day), based on increased absolute

and relative liver weights and hepatocellular neoplastic lesions at 1,000 ppm and above.

Long-term toxicity/carcinogenicity studies in rats were reviewed. The study considered by the 1973 Joint Meeting was found to be unacceptable. In a new study at dietary concentrations of 0, 100, 700 or 5,000 ppm, the NOAEL was 100 ppm (equal to 3.7 mg/kg bw/day), based on increased water consumption and increased relative liver and thyroid weights at 700 ppm and above. Increased incidences of focal hepatocellular hypertrophy and fatty changes and pigmentation of hepatocytes were also observed at 700 ppm and above.

In a reproduction study in rats using dietary concentrations of 0, 165, 750 or 2,250 ppm, the NOAEL was 165 ppm (equal to 9 mg/kg bw/day), based on increased liver weight and hypertrophy of hepatocytes in F_1 animals at 750 ppm and above.

Teratogenicity studies were conducted with rats and rabbits. In the study in rats at doses of 0, 50, 300 or 700 mg/kg bw/day, depressed maternal body-weight gain and an increased incidence of skeletal variations of fully formed 14th ribs and rudimentary 14th ribs were recorded at 300 mg/kg bw/day and above. The maternal NOAEL in this study was 50 mg/kg bw/day. There was no evidence of embryo/fetotoxicity or teratogenicity. In the study in rabbits at doses of 0, 20, 60, or 120 mg/kg bw/day, mean body-weight gain was depressed at 60 mg/kg bw/day. The NOAEL for maternal toxicity was 20 mg/kg bw/day, and no embryo/fetotoxic or teratogenic effects were found.

After reviewing the available genotoxicity data, the Meeting concluded that bromopropylate was not genotoxic.

An ADI was established, based on the NOAEL of 2.7 mg/kg bw/day in the one-year study in dogs, using a 100-fold safety factor.

A toxicological monograph was prepared, summarizing the data that have been reviewed since the previous evaluation and incorporating relevant studies from the previous monograph.

TOXICOLOGICAL EVALUATION

Level causing no toxicological effect

Mouse: 150 ppm, equal to 16 mg/kg bw/day (two-year study)
Rat: 100 ppm, equal to 3.7 mg/kg bw/day (two-year study)
Dog: 100 ppm, equal to 2.7 mg/kg bw/day (one-year study)

Estimate of acceptable daily intake for humans

0-0.03 mg/kg bw

Studies which will provide information valuable in the continued evaluation of the compound

Observations in humans.

RESIDUE AND ANALYTICAL ASPECTS

Bromopropylate was scheduled by the CCPR for periodic review at the 1993 JMPR (ALINORM 93/24A, para 93). It was first considered by the JMPR in 1973 when residue data on apples, pears, plums, grapes, bananas, strawberries, citrus, hops, tea, cotton, egg plant and tomatoes were evaluated and MRLs for apple, banana, cherry, citrus fruits, cotton seed, grapes, hops, nectarine, peach, pear, plum, strawberry, tea and vegetables were established. Since then,

more residue trials on some of the same crops as well as additional ones such as artichokes, beans, celery, cucurbits, guavas, maize, onions, papaya, peaches, peanuts, peas, sweet peppers, pineapples and sugar beet have been conducted by the manufacturer in various countries as well as the authorities of The Netherlands and Spain. Further information has also been provided by the manufacturer, Spain, The Netherlands and Australia on current uses. Australia has also indicated that the pesticide had not been marketed since 1986.

The manufacturer has indicated that there were no current uses on nectarines, bananas or cherries. The Meeting recommended the withdrawal of the MRLs for these commodities.

Additional plant metabolism studies on apples and citrus showed that the parent compound was the residue of importance, particularly in the edible parts, although minor metabolites, mainly 4,4′-dibromobenzilic acid, were found in the leaves.

No information on the fate in animals has been submitted but the Meeting noted that adequate information on animal transfer studies for dairy cows and beef cows had been reported by the 1973 JMPR.

In water, bromopropylate was found to have a half-life of 20-40 days. Bromopropylate and its metabolites were concluded to have low mobility in sandy loam, silty loam and sandy soils on the basis of leaching studies. The half-life in silty loam and sandy loam soils was about 45 days, the major metabolite being 4,4′-dibromobenzophenone.

Residues in the juice of apples and mandarin oranges, and in wine and beer were reported to be below the limit of detection, (0.02 mg/kg in all cases, except beer 0.005 mg/kg).

Bromopropylate residues in samples of tea, tomatoes, tomato puree, oranges, grapefruit, orange juice, orange oil, apples, peaches and cherries were found to remain stable up to 2 years under freezer conditions at -18^{o}C.

GAP information was not available for guavas, papayas, pineapples, onions, celery, maize, peanuts or sugar beet, so residue data on these crops could not be evaluated.

GAP information and residue trials data on peas, tomatoes, egg plants, artichokes and sweet peppers were too limited for the Meeting to estimate maximum residue levels.

The available information for cotton seed, hops and tea was also too limited to support the present MRLs. The Meeting agreed to withdraw the recommendations for these commodities.

For citrus, the trials data on residues in the pulp and peel confirmed earlier findings that most of the residues are concentrated in the peel. Results from Australia, South Africa and Morocco were not supported by GAP information, while in data from Spain, China and Israel trials rates were expressed differently from the national GAP. Six trials in Brazil on oranges and mandarin oranges were evaluated in the light of the national GAP. At 14 days after the last application, residues ranged from 0.6 to 5.8 mg/kg in the peel and 0.2 to 0.4 mg/kg in the pulp. Assuming that the peel weight is 30% of the fruit's weight the calculated residues in whole fruit would be less than 2 mg/kg. In trials on lemons in Spain rates between 1.6 and 5.2 kg ai/ha were used, which were within Spanish GAP. At 14 days, residues ranged from 0.5 to 1.3 mg/kg. The Meeting estimated a maximum residue level of 2 mg/kg for bromopropylate in citrus fruits with a PHI of 14 days, to replace the current MRL of 5 mg/kg.

The residue trials data on apples from Brazil and Canada were not evaluated because no information on registered uses was available from these countries. Trials from Chile were also not evaluated because the registered use was supplied in terms of spray concentration while the trials application rates were expressed as kg ai/ha. Trials in The Netherlands, France and Germany on apples and pears were within the GAP of The Netherlands and France, and

residues at 21 days were within the range of 0.18 - 1.6 mg/kg. Data on apples and pears were mutually supporting. The Meeting recommended an MRL of 2 mg/kg for pome fruits at a pre-harvest interval of 21 days, based on the data from France, Germany and The Netherlands.

For peaches, trials data from Brazil and Switzerland were submitted, and for plums, data from ten trials in Germany. Data from Switzerland and Germany were evaluated on the basis of the GAP of Switzerland (stone fruits) and The Netherlands (plums). At 21 days after the last application, the highest residue obtained was 1.6 mg/kg. The Meeting estimated a maximum residue level of 2 mg/kg for plums at a pre-harvest interval of 21 days. Data from peach trials were insufficient to recommend an MRL but provided some additional support for the plum estimate.

Additional data on grapes from trials in Australia, France, Hungary, Israel, South Africa and Switzerland were submitted. As there was no information on registered uses in Australia and South Africa, the data from these countries could not be evaluated. Trials rates in Hungary were expressed differently from GAP in Hungary. The trials rates in France, Israel and Switzerland covered the national recommendations. The data from France showed that by 27 days residues in grapes were all less than 2 mg/kg, and in wine <0.02 mg/kg. The Meeting recommended an MRL of 2 mg/kg for grapes at a pre-harvest interval of 28 days.

Residue trials data on strawberries had been submitted from Brazil, Israel, Italy, Japan and Spain. Although trial rates in Spain and Italy did not cover the maximum rates under the GAP of the two countries, on the basis of the trials data the Meeting recommended an MRL of 2 mg/kg for strawberries with a PHI of 14-21 days.

For beans, evaluation of the residue trials data from Italy and Spain was based on the GAP of Spain. Residue levels at 7 days ranged from 0.26 to 2.5 mg/kg. The Meeting recommended an MRL of 3 mg/kg for common beans at a pre-harvest interval of 7 days.

Residue data from Italy on cucumbers, melons and summer squash were evaluated on the basis of the GAP of Spain. The Meeting recommended an MRL of 0.5 mg/kg for cucumber, melons and summer squash at a pre-harvest interval of 7 days.

The Meeting recommended withdrawal of the MRL for vegetables, to be replaced by MRLs for the specific commodities beans, cucumber, melons and summer squash.

FURTHER WORK OR INFORMATION

Desirable

1. Information on the occurrence of bromopropylate residues in food in commerce or at consumption.

2. Information on residues in the pomace of citrus fruit, apples and grapes, which may be used as animal feeds.

4.6 CARBOFURAN (096)

RESIDUE AND ANALYTICAL ASPECTS

The 1992 CCPR held the 2 mg/kg carbofuran citrus limit at Step 7B pending a JMPR review of country comments to be submitted. The Meeting considered comments from Germany on the proposed citrus limit, as well as supervised trials data for potatoes, onions and tomatoes. In addition to being only summarized, the data for potato did not reflect the GAP PHI. No GAP

information was provided for onions. For tomato important information was missing for two of four trials. The Meeting did not consider the summary data with no additional supporting information from limited trials sufficient for estimating maximum residue levels.

The Meeting took note of its recommendation to withdraw the 2 mg/kg temporary limit for carbosulfan in citrus fruits (see 4.7 below), and further noted that the current 2 mg/kg citrus limit for carbofuran was recommended to accommodate residues resulting from the use of carbosulfan. The Meeting therefore recommended withdrawal of the current temporary limit for carbofuran in citrus fruits.

4.7 CARBOSULFAN (145)

RESIDUE AND ANALYTICAL ASPECTS

Carbosulfan was first reviewed for residues by the 1984 JMPR. Although extensive data were reviewed for a number of commodities, the Meeting recommended only a temporary limit for citrus fruits pending the submission of required and desirable information, in particular critical supporting studies and GAP information.

The 1991 Meeting recommended that limits for carbosulfan should be for carbosulfan *per se* and that separate limits should be set for the sum of carbofuran and 3-hydroxy-carbofuran to accommodate residues resulting from the use of carbosulfan or carbofuran. Keto-carbofuran was deleted from the definition. The 1991 JMPR decided not to propose a new limit for citrus fruits, pending the review of additional data received too late for consideration. Additional requirements were added to those listed by the 1984 Meeting.

The Meeting received a discussion paper, summary comments and selected pages and summary tables from a number of reports on the identification and fate of residues in animal products in response to the 1984 JMPR requirements. The Meeting did not review these documents in the absence of the complete reports from which the summaries came. The required information on brassica vegetables was not provided. GAP information and trials data on a number of commodities were also provided, much but not all of which had been reviewed by the 1984 JMPR.

Citrus. The 1984 JMPR recommended a 2 mg/kg temporary limit for citrus fruits, pending the submission of required information. The definition of the residue was revised to separate the limits for carbosulfan from those of its metabolites by the 1991 JMPR, resulting in separate limits at 2 mg/kg for carbosulfan *per se* and 2 mg/kg for the sum of carbofuran and 3-hydroxy-carbofuran. No change in the carbosulfan numerical level was proposed, pending the review of additional information. Clarification of apparent discrepancies in Spanish GAP was also requested but the GAP has not been fully clarified. One Codex delegation provided written comments explaining its view that a 2 mg/kg limit was not justified for carbosulfan or carbofuran, citing the general lack of data reflecting GAP and the low residues of carbosulfan in particular. In order to address the issues the Meeting considered newly submitted information on GAP and new and re-submitted residue trials data, and took into account data reviewed by the 1984 JMPR as well as the Codex comments.

The most extensive data (1984 JMPR) are those from trials in the USA. However, the use is not yet GAP in the United States and the Meeting could not with confidence relate the data to the GAP of other countries. Further, new data on peel and pulp residues in Spanish trials did not include important information, precluding a determination of whether the data reflected Spanish GAP (which is itself still in question). If future submissions show that all of the Spanish data reflect GAP, maximum whole fruit residues of <0.05 mg/kg carbosulfan and 0.3 mg/kg carbofuran plus 3-hydroxy-carbofuran would be indicated, assuming a peel to pulp ratio of 30:70. Re-submitted Brazilian data (<0.05 mg/kg carbosulfan, carbofuran, or 3-hydroxy-

carbofuran) could not be related to the Brazilian GAP information provided.

The only data that could be related to GAP with some confidence were from Italian trials reviewed in 1984 which could be compared to Spanish GAP and Israeli data which could be compared to Israeli GAP. The Italian trials according to Spanish GAP resulted in maximum residues of 0.7 mg/kg carbosulfan and 1.7 mg/kg for the sum of carbofuran and 3-hydroxy-carbofuran. Maximum residues reflecting Israeli GAP were 0.05 mg/kg carbosulfan and 0.2 mg/kg for the sum of carbofuran and 3-hydroxy-carbofuran, although requested information on the interval from sampling to analysis to give greater confidence in the results was not provided.

Because the requested clarification of GAP and the sampling-to-analysis intervals still have not been supplied, because only a relatively small data base is available which can be compared GAP, and because of a large discrepancy between the two sets of results which were comparable to GAP, the Meeting concluded that insufficient information had been provided to support a citrus limit for carbosulfan or carbofuran. The Meeting was informed that additional supervised citrus trials would be conducted in Spain, Brazil and Mexico.

Hops. German data for green and dry hops, spent hops and beer that were reviewed by the 1984 JMPR were re-submitted to the Meeting, together with GAP for Spain and Germany. No GAP information was provided to the 1984 Meeting. The data indicate that dry hop residues are unlikely to exceed 3 mg/kg for carbosulfan and 7 mg/kg for combined residues of carbofuran and 3-hydroxy-carbofuran after the 28-day Spanish PHI from the 37.5 g ai/hl used in the supervised trials. No data were available for the maximum 75 g ai/hl reported to be Spanish GAP. Maximum residues 21 days (German GAP PHI) after 6 applications at 37.5 ai/hl resulted in carbosulfan residues of 0.04-3.4 mg/g in dry hops and 0-2.2 mg/kg in green hops. Corresponding residues of carbofuran plus 3-hydroxy-carbofuran were 1.4-11.3 mg/kg in dry hops and 1-9.9 mg/kg in green hops. Because 6 applications were used, compared to GAP of one application, the Meeting was unable to estimate a limit for hops.

Melons. Data (indoor) were available from only two trials in one country (two results at the GAP PHI) with only one trial at the maximum application rate. No analyses were conducted for 3-hydroxy-carbofuran and critical supporting information on sample storage conditions and the interval from sampling to analysis were lacking. It was therefore concluded that the data were insufficient to support an MRL for melons. The Meeting was informed that supervised trials on melons are being conducted in Spain.

Pome fruit. The 1984 JMPR reviewed data on apples and pears from three countries, but did not estimate a limit for pome fruit because the data base could not be related to available GAP and critical supporting information (e.g. storage conditions and intervals) was not provided. Summaries of some of the data reviewed by the 1984 JMPR were provided to the present Meeting as well as additional summary data not previously reviewed. Because only summary information was provided, because most of the old and new data could not be compared to the available Spanish GAP, and because the desirable critical supporting information still had not been provided, the Meeting concluded that the data were insufficient to support limits for pome fruit.

Potatoes. Fairly extensive data from Italy, France and the UK were reviewed by the 1984 JMPR. Summaries of these data were provided to the Meeting, but 11 of the 13 trials did not reflect the (Spanish) GAP PHI of 28 days. A discussion document and selected pages from a sugar beet metabolism study were provided in response to the 1984 JMPR requirement for a root metabolism study from both foliar and soil treatments. The Meeting concluded that data reflecting GAP were not adequate to recommend an MRL and that submission of the complete metabolism study would be needed before that requirement could be regarded as satisfied.

Stone fruit. Summary data from a substantial number of trials were available, but were not

adequately reported and many of the trials did not reflect GAP PHIs for use in recommending MRLs. The trials most adequately reported were based on an 83-day PHI, whereas GAP is 28 days. The Meeting concluded that data reflecting GAP were inadequate and inadequately reported to recommend MRLs.

Sugar beet. Extensive data were reviewed by the 1984 JMPR but critical supporting information was sketchy or in some cases missing, data were not relevant to available GAP information and a root metabolism study was required. Information provided to the Meeting indicated that Spanish GAP was similar to that used in the French and UK trials reviewed in 1984, except that the Spanish PHI is 60 days whereas most of the trials results were at $\geq$104 days. Summary discussion information and selected pages from a sugar beet root metabolism study provided to the Meeting did not meet a 1984 JMPR root metabolism requirement. The full reports need to be submitted. Summary Italian data from trials at application rates greater than the Spanish GAP rates and at longer intervals than the minimum Spanish PHI were also provided. The Meeting concluded that data reflecting GAP were still inadequate to support an MRL for sugar beets. When the complete metabolism study and the detailed Italian data are provided the Meeting can reconsider the position.

Metabolism. The Meeting was informed that metabolism studies on oranges, rats, and goats and an animal transfer study on cows are being conducted.

4.8 CHLOROTHALONIL (081)

RESIDUE AND ANALYTICAL ASPECTS

Chlorothalonil was first evaluated in 1974. This evaluation has been prepared as part of the programme of periodic reviews agreed by the CCPR.

Information on current GAP and residue trials data were made available to the Meeting by one of the manufacturers; GAP information was also provided by Australia, Canada and the EC.

At the initiation of this review there were 35 MRLs for chlorothalonil; all were CXLs except the MRL for grapes which was at step 7B.

The fate of chlorothalonil has been studied in lettuce, tomato, carrot and celery. Chlorothalonil was the major characterised component of the residue in all cases; small amounts of 4-hydroxy-2, 5, 6-trichloroisophthalonitrile (SDS-3701) were also found.

Data from supervised residue trials carried out in a number of countries and on a range of crops were available.

No GAP was reported for citrus fruit, so the Meeting recommended that the CXL of 5mg/kg should be withdrawn.

The CXL of 10 mg/kg for cherries was proposed in 1974; it was based on US GAP with a 7-day pre-harvest interval and residue data from trials carried out in the USA. Since use so close to harvest is no longer GAP in the USA the CXL is obsolete and the Meeting considered that it should be withdrawn. Results from a series of trials carried out in accordance with current GAP in the USA were available at the Meeting. Residues up to 0.5 mg/kg were found. The Meeting recommended that an MRL of 0.5 mg/kg was appropriate for this use.

For peaches, the CXL of 25 mg/kg was again based on US GAP permitting use up to 7 days before harvest and residue trials data from the USA. This US GAP is now obsolete and therefore the CXL was not acceptable. Supervised trial data on peaches from Italy, Spain and

the USA were made available to the Meeting. Residues up to 0.12 mg/kg were found when chlorothalonil was used according to current US GAP, and up to 0.98 mg/kg in Italian trials within Spanish and Greek (1.5kg ai/ha and 14-15 days PHI) and Italian (1.0 kg ai/ha and 21-day PHI) GAP. The Meeting recommended an MRL of 1 mg/kg for peaches.

Chlorothalonil residues up to 4.1 mg/kg were found in cranberries harvested 50-70 days after treatment at 5.9 kg ai/ha (within US GAP) in a series of trials in the USA in the 1980s. The Meeting recommended an MRL of 5 mg/kg for cranberry.

The CXLs of 25 mg/kg for raspberries (red and black) and currants (black, red and white) and 10 mg/kg for blackberries were based on GAP and trials in the USA. Since this GAP is no longer current the Meeting recommended that these CXLs should be withdrawn.

For grapes, the draft MRL, at step 7B, is 10 mg/kg. This proposal was based on Austrian GAP of 0.11 kg ai/ha with a PHI of 7 days and on data from supervised trials carried out in Germany; this GAP is no longer current. A 1.6 kg ai/ha, 7-day PHI GAP has been reported for Australia and in one trial in 1973/4 chlorothalonil residues up to 5.6 mg/kg were found in supervised trials after treatment within this GAP. However, GAP in France (0.4 kg ai/ha, 30 days PHI) yielded much more recent data that were consistent and were deemed more suitable as the basis for a recommendation. The Meeting therefore recommended an MRL of 0.5 mg/kg, based on the data from France.

For banana the GAP on which the CXL of 0.2 mg/kg was based is not clearly described in the 1973 evaluations. The data base considered by the present Meeting was not sufficient to support a soundly based MRL and the Meeting recommended that the CXL should be withdrawn.

The CXL of 5 mg/kg for bulb onions was based on trials data for green onions; the Meeting therefore concluded that it needed revision. Chlorothalonil residues up to 0.57 mg/kg were found in bulb onions harvested 7 days after treatment at 1.5 - 1.75 kg ai/ha (within US GAP) and up to 0.52 mg/kg 14 days after treatment at 1.5 kg ai/ha (within other countries' GAP), although most results were lower than these. The Meeting recommended an MRL of 0.5 mg/kg.

The CXL of 5 mg/kg for cabbages was based on residue data from US trials where crops were harvested on the day of the last treatment. Since current US GAP specifies a minimum PHI of 7 days the CXL should be revised. Chlorothalonil residues up to 0.7 mg/kg were reported from trials using treatment regimes within US, UK and Irish GAP. The Meeting recommended an MRL of 1 mg/kg.

For broccoli, the CXL of 5 mg/kg is based on a 7-day PHI and results from US trials. This GAP is still current in the USA and Canada but although results were reported from two further US trials where treatments were within GAP, the Meeting considered the data were inadequate and recommended that the CXL of 5 mg/kg should be withdrawn.

The CXL of 5 mg/kg for Brussels sprouts was based on a PHI of 7 days and data from the USA. Chlorothalonil residues up to 4.3 mg/kg were reported for samples harvested 6-7 days after treatment at 1.3-2.5 kg ai/ha. The Meeting recommended that the CXL should be maintained.

For cauliflower, the CXL of 5 mg/kg was based on a PHI of 7 days and residue data from the USA. Chlorothalonil residues up to 0.47 mg/kg were reported from trials where treatments were within current GAP in the USA, UK and Ireland. The Meeting recommended an MRL of 1 mg/kg.

For kale, the CXL of 10 mg/kg was based on US GAP and residue data. Since this GAP is

no longer current the Meeting recommended withdrawal of the CXL.

The CXL of 5 mg/kg for melons except watermelon was based on US trials data and a 1-day PHI. Chlorothalonil residues up to 1.45 mg/kg were found in samples treated in accordance with US GAP. The Meeting recommended an MRL of 2 mg/kg but recognised that additional data on residues on different types of melons would be desirable.

For cucumbers, the CXL of 5 mg/kg is based on a 1-day PHI. Chlorothalonil residues up to 4.3 mg/kg were reported from trials where treatments were in accordance with US GAP and the Meeting recommended that the CXL should be maintained.

The CXLs of 5 mg/kg for summer and winter squash and pumpkins were based on a 1-day PHI. Chlorothalonil residues up to 3.6 mg/kg were found in samples of summer and winter squash treated in accordance with current US GAP. The Meeting recommended that MRLs of 5 mg/kg were appropriate for summer and winter squash. No residue data were presented for pumpkins and therefore that CXL should be withdrawn, although pumpkins appear to be covered in the Codex Classification by the MRL for winter squash.

For sweet corn, the CXL of 1 mg/kg was based on a 1-day PHI which is no longer GAP. Residue data reflecting current US GAP were available from only one trial; these were not sufficient to estimate a maximum residue level. The Meeting recommended withdrawal of the CXL.

The CXL for tomato is 5 mg/kg, based on US data and GAP. Chlorothalonil residues up to 4.6 mg/kg were found in trials where treatments were within GAP. The Meeting recommended that the CXL should be maintained.

The CXL of 10 mg/kg for peppers was based on US GAP and residue data. Since use on peppers is no longer GAP in the USA the Meeting recommended withdrawal of this recommendation.

The CXLs for endive, lettuce and witloof chicory (sprouts) were based on US GAP and residue data. Since use on these crops is no longer GAP in the USA the Meeting recommended withdrawal of these CXLs.

The CXL of 5 mg/kg for common bean (pods and/or immature seeds) was based on US GAP and residue data. In supervised trials residue levels in crops treated in accordance with GAP were up to 3.1 mg/kg. The Meeting recommended that the CXL should be maintained.

The CXL for lima beans (dry) was based on US GAP and residue data. Since this use is no longer GAP in the USA the Meeting recommended withdrawal of the CXL.

The CXL of 1 mg/kg for carrots was based on GAP and residue data from the USA. Residues up to 0.96 mg/kg were reported from trials where treatments were within GAP. The Meeting recommended that the CXL should be maintained.

The CXL of 0.1 mg/kg for potato was based on a 0-day PHI. Residues up to 0.18 mg/kg were reported from trials where treatments were within GAP although only one result exceeded 0.1 mg/kg. The Meeting recommended an MRL of 0.2 mg/kg.

The CXL of 1 mg/kg for sugar beet was based on a 1-day PHI; this is no longer GAP. Residues reflecting current GAP were up to 0.1 mg/kg in the root. The Meeting recommended an MRL of 0.2 mg/kg for sugar beet root. Corresponding residues in the leaves reached 14 mg/kg. The Meeting recommended an MRL of 20 mg/kg for sugar beet leaves or tops but realised that appropriate animal transfer studies were lacking.

The CXL of 15 mg/kg for celery was based on a 7-day PHI. Chlorothalonil residues up to 9.8 mg/kg were found in trials where treatments reflected current GAP. The Meeting recommended an MRL of 10 mg/kg.

Barley grain from crops treated in accordance with GAP contained up to 1.4 mg chlorothalonil/kg. Most results however were much lower than this. The Meeting decided that the data reflecting use up to 1.4 kg ai/ha were not sufficient to support a soundly based MRL and recommended an MRL of 0.1 mg/kg for grain, based on application rates up to 1.0 kg ai/ha. The Meeting also recommended an MRL of 20 mg/kg for barley straw; animal transfer studies are desirable.

Wheat grain from crops treated in accordance with GAP contained up to 0.09 mg chlorothalonil/kg. The Meeting recommended that the MRL should be established at 0.1 mg/kg for grain and 20 mg/kg for wheat straw, recognising that animal transfer studies were desirable.

GAP was not reported for any other cereal grain. The Meeting recommended that the CXL for cereal grains should be withdrawn.

The CXLs for whole peanut and peanut kernels were based on a 1-day PHI; this is no longer GAP. Chlorothalonil residues up to 0.03 mg/kg were found in crops treated in accordance with current GAP. The Meeting recommended an MRL of 0.05 mg/kg for peanut and withdrawal of the CXL for whole peanuts.

Information on residue distribution between the inedible and edible portions of the commodity was available for banana; chlorothalonil is essentially a surface residue and transfer to pulp was insignificant.

Processing studies are available for cherry, peach, grape, cabbage, cucumber, squash, tomato, snap bean, carrot, potato, celery and peanut.

Washing cherries, peaches, cucumbers, tomatoes and snap beans removed 45-95% of the residue. Residue reductions of 75-98% occurred in cabbages, cucumbers, tomatoes and celery during distribution from the farm gate to retail outlets. Residue levels in canned cherries, canned pickled cucumber and tomato juice made from treated crops were very low (1-2% of initial residues). Residues were not found in canned peach puree, wine, squash-based baby food, tomato paste, canned or frozen snap beans, carrot-based baby food, potato crisps, dried potato or refined peanut oil prepared from crops with incurred residues.

Chlorothalonil residues were stable during freezer storage for one year in cherries, cucumbers, tomatoes, carrots, potatoes, celery and wheat grain.

FURTHER WORK OR INFORMATION

Desirable

1. Additional residue data from supervised trials on different types of melons.

2. Animal transfer studies assuming a residue equivalent to the recommended MRL of 20 mg/kg in sugar beet leaves or tops, barley straw and wheat straw.

3. Additional residue data on grapes treated according to GAP in Australia.

4.9 CHLORPYRIFOS-METHYL (090)

RESIDUE AND ANALYTICAL ASPECTS

Data evaluated by the 1991 JMPR showed residues of chlorpyrifos-methyl in crude and refined maize oil as high as approximately 100 mg/kg, produced from maize containing only 3.8 mg/kg. The 1991 JMPR therefore required further information on the influence of commercial refining processes on residues of chlorpyrifos-methyl in oil from maize and a full description of the processes used.

As a maximum residue limit of 10 mg/kg is established for chlorpyrifos-methyl in rape seed, and it would be expected that residue levels in oil produced from treated rape seed would also be high, information was required on the levels of residues occurring in rape seed oil.

In response to the requirement for information about commercial processes used for producing and refining maize oil a detailed description of the milling, refining and deodorization procedures generally used in the USA was received. Two studies of the fate of residues of chlorpyrifos-methyl during the processes of milling, refining and deodorization were also supplied. The compound is concentrated in the oil produced from maize grain and it does not disappear during the process of refining, but it disappears almost completely when the oil is deodorized. The procedure used for deodorizing is to heat the oil to 175-230° C in a vacuum. In this process chlorpyrifos-methyl is volatilized, and in one of the experiments the vapours were trapped and 95% of the chlorpyrifos-methyl originally present in the oil was collected as the unchanged compound.

No information was available to the Meeting on the levels of chlorpyrifos-methyl in rape seed oil either from trials or from monitoring. The Meeting therefore recommends withdrawal of the existing temporary MRL of 10 mg/kg for chlorpyrifos-methyl in rape seed.

Information was received from Spain on registered uses of chlorpyrifos-methyl and summarized residue data from trials on lemons, mandarins and oranges. The applications in the trials were in accordance with registered uses in Spain, except those on mandarins where the dosage was a little lower in the trials. All residues were low, between 0.01 and 0.13 mg/kg after 14 days, and lower than the proposed limit of 0.5 mg/kg for oranges. The proposed residue limit for oranges was confirmed, but the Meeting was unable to propose a residue limit for the whole group of citrus fruits as details from the trials in Spain were not available.

FURTHER WORK OR INFORMATION

Desirable

Submission of details from trials on citrus fruits in Spain and further information on GAP for citrus fruits in Spain.

4.10 CYCLOXIDIM (179)

RESIDUE AND ANALYTICAL ASPECTS

Cycloxidim, a systemic cyclohexanedione herbicide, was reviewed for the first time by the 1992 JMPR. However, the time available did not allow adequate evaluation of the extensive residue data provided by the manufacturer. These data have been reviewed by the present Meeting.

Residue data were reported from supervised trials of cycloxidim carried out in 15 countries and on over 40 commodities. Although many of these trials were according to registered and/or recommended use patterns, some crop/ application rate combinations were not registered or

the resultant data were very limited. In addition, residue data on bulb onions, parsnip, sunflower seed and hay were obtained using an analytical procedure with the comparatively high limit of determination of 0.5 mg/kg while that for Brussels sprouts was 0.25 mg/kg; determinations on all other commodities could be made down to 0.05 mg/kg.

Cycloxidim is applied as a foliar spray directly to the growing crop and also to soil as a surface application. Owing to its systemic properties some uptake and distribution is to be expected although the extent is likely to be variable as it is dependent on the growth stage; this is borne out by the wide variations observed in the residue data presented. As there is little alteration in residue level with time after application, the PHIs are of little real significance.

These factors combined to make the estimation of suitable maximum residue levels for this compound rather complicated. However, despite these potential drawbacks the residue data were deemed to be sufficient to allow recommendations for MRLs to be made for 16 commodities. They were regarded as being inadequate, for various reasons, to support recommendations for the other commodities for which residue data were available. Data on citrus, pome and stone fruits were sparse, as were those on tropical fruits, cucurbits and some root and stem vegetables. Despite the absence of processing data on grapes, potatoes and sugar beet the Meeting felt able to recommend MRLs for those crops.

Residue data on beans (dry), rape seed and soya bean (dry) were deemed adequate and mutually supportive for a maximum residue level of 2 mg/kg to be estimated for each commodity. Similarly, despite the inherent variability, residue data on Brussels sprouts, cabbage and cauliflower were taken together to estimate a maximum level of 2 mg/kg for brassica vegetables. Data on common bean together with those for peas (in pod) supported a maximum residue level of 1 mg/kg for each, while for shelled peas (green) a level of 2 mg/kg was suggested; for peas (dry) the results were too variable to interpret with any degree of assurance. Residue data on carrot, leek, lettuce (head and leaf) and strawberry were also found to be adequate for the recommendations given in Annex I to be made.

FURTHER WORK OR INFORMATION

Desirable

1. Residue data from supervised trials on bulb onions, parsnip, sunflower seed and hay, using an analytical procedure with a lower limit of determination of about 0.05 mg/kg.

2. Processing studies on grapes, potatoes and sugar beet treated with cycloxidim in supervised trials.

4.11 DDT (021)

RESIDUE AND ANALYTICAL ASPECTS

At the 22nd and 23rd Sessions of the CCPR (1991 and 1992) it was agreed that countries should be requested to provide information on registered or recommended uses of DDT and also on residue levels from trials with registered uses or from monitoring. At the 23rd Session the existing Extraneous Residue Limits for DDT in cereal grains, eggs, meat and milk were converted to temporary limits, and the general MRL for DDT in fruits and vegetables was withdrawn. Residue data were necessary to support the existing ERLs and possibly to develop ERLs for DDT in commodities in the fruits and vegetables groups.

The Meeting has received no information about registered or recommended uses of DDT on crops or animals. Monitoring data for residues of DDT in fruits, vegetables, cereal grains

and products of animal origin were received from the governments of Canada, Denmark, The Netherlands and the USA.

It was obvious from the monitoring data that residues are not often present in fruit, vegetables and cereal grains, but the incidence observed is of course dependent on the limit of determination used in the monitoring. In Canada, The Netherlands and the USA the limit of determination was 0.01 mg/kg, while the limit in Denmark was 0.02 mg/kg. In Canada DDT was present in 29 of 1100 samples. In Denmark no residues were found in any of 4300 samples. In The Netherlands DDT was present in 54 of 37,500 samples, and in the USA in 369 of 15,445 samples. In most cases residues were low and mostly below 0.1 mg/kg. The frequency of residues in fruit and vegetables seems to be highest in carrots, probably owing to the occurrence of DDT in the soil from earlier uses and the ability of carrots to take up pesticides from the surrounding soil. In Canada and the USA DDT was present in 64 of a total of 452 samples of carrots, which is approximately 14%. It is more surprising that DDT residues were also present in several samples of apples.

Residue data from monitoring cereal grains were available only from the USA. In 579 samples of cereal grains DDT was present in 5 samples with the highest residue at 0.09 mg/kg.

Residue data were available from many samples of animal products such as butter, milk, cheese, eggs and the fat of cattle, pigs, poultry, sheep, goats and horses. Residues of DDT and its metabolites occurred more frequently in animal products than plant products. Residues were mostly at very low levels, but in some samples up to 0.5 mg/kg and in a few samples even higher with a maximum of 1.8 mg/ kg in fat from cattle.

Residues in the monitoring data available to the Meeting were usually considerably lower than the existing temporary ERLs. Residues in the fat of meat were as mentioned above much lower than the existing limit of 5 mg/kg. In eggs residues with a few exceptions were below the limit of determination (0.01 or 0.02 mg/kg), and were at the level of 0.11 and 0.20 mg/kg in only two samples of egg powder. For milk the existing TMRL is 0.05 mg/kg, approximately 1 mg/kg in milk fat. All residues in samples from the monitoring studies were considerably lower, and generally below the limit of determination. Residues in butter and cheese, calculated as the levels in milk fat, were always considerably lower than 1 mg/kg.

The incidence of detection of environmental contaminants is expected to increase if lower limits of determination are employed. The Meeting noted the remarks made at the 24th Session of the CCPR (ALINORM 93/24, 29) concerning realistic limits of determination, that using methods with low limits of determination was costly and not the best use of resources. The Meeting concluded that for the general monitoring of DDT and the metabolites included in the definition, a suitable limit of determination for the total residue would be 0.02 mg/kg.

As the production of the compound ceases and environmental residues decrease, extraneous residues in food will also decrease. The Meeting therefore recommended that monitoring data should be evaluated again in 1998, with the possibility of lowering the ERLs for DDT.

FURTHER WORK OR INFORMATION

Desirable

Residue data from monitoring DDT in fruit and vegetables in other countries.

4.12 DIAZINON (022)

TOXICOLOGY

Diazinon was previously evaluated by the Joint Meeting in 1963, 1965, 1966 and 1970. An ADI of 0-0.002 mg/kg bw was allocated in 1966, based on an NOAEL of 0.02 mg/kg bw/day in human volunteers. The compound was reviewed at the present Meeting on the basis of the CCPR periodic review programme.

Following oral administration to rats, diazinon was almost completely absorbed and eliminated, mainly in the urine.

The main degradative pathway includes the oxidase/hydrolase-mediated cleavage of the ester bond leading to the pyrimidinol derivative 4-hydroxy-2-isopropyl-6-methylpyrimidine, which is further oxidized to more polar metabolites.

Diazinon has moderate acute oral toxicity to mice and rats. The clinical signs observed were consistent with cholinesterase inhibition and included sedation, tremors, convulsions and ataxia. It is classified by WHO as moderately hazardous.

In an oral 90-day feeding study in rats at dietary concentrations of 0, 0.5, 5, 250 or 2500 ppm, the NOAEL was 5 ppm (equal to 0.4 mg/kg bw/day), based on erythrocyte and brain cholinesterase inhibition at 250 ppm and above.

In short-term studies in dogs, diazinon was administered at dietary concentrations of 0, 0.1, 0.5, 150 or 300 ppm for either 90 days or 52 weeks. In both studies, the NOAEL was 0.5 ppm (equal to 0.02 mg/kg bw/day), based on erythrocyte and brain cholinesterase inhibition at 150 ppm and above.

In a carcinogenicity study in mice, diazinon was administered at dietary concentrations of 0, 100 or 200 ppm over 103 weeks. There was no evidence of carcinogenicity.

In a carcinogenicity study in rats diazinon was administered at dietary concentrations of 0, 400 or 800 ppm for 103 weeks. There was no evidence of carcinogenicity.

In a long-term toxicity/carcinogenicity study, rats were maintained on a diet containing diazinon at concentrations of 0, 0.1, 1.5, 125 or 250 ppm for up to 99 weeks. The NOAEL was 1.5 ppm (equal to 0.07 mg/kg bw/day), based on inhibition of erythrocyte and brain cholinesterase at 125 ppm and above. There was no evidence of carcinogenicity.

A multigeneration reproduction study was conducted in rats using dietary concentrations of 0, 10, 100 or 500 ppm. The NOAEL was 10 ppm (equivalent to 0.5 mg/kg bw/day), based on a reduction in parental body-weight gain in the F_1 generation and a reduced survival rate and reduced body weight of F_1 pups at 100 ppm.

In a teratogenicity study in rats, diazinon was orally administered at dose levels of 0, 10, 20 or 100 mg/kg bw/day. Maternal toxicity, indicated by weight loss correlated with reduced food consumption, became evident at 100 mg/kg bw/day. Effects on the fetuses at this dose level consisted of retarded ossification and an increased incidence of rudimentary ribs. The NOAEL was 20 mg/kg bw/day, based on maternal toxicity and fetotoxicity. There was no evidence of teratogenicity.

A teratogenicity study in rabbits conducted with oral dose levels of 0, 7, 25 or 100 mg/kg bw/day revealed clinical signs of maternal toxicity, increased mortality and reduced body-weight gain at 100 mg/kg bw/day. The NOAEL was 25 mg/kg bw/day. There was no evidence of teratogenicity.

A neurotoxicity study performed with hens treated at dose levels of 13 or 28 mg/kg bw/day (protected by atropine pre-treatment) did not reveal evidence of delayed neurotoxicity.

Diazinon has been adequately tested in a series of genotoxicity assays. Chromosomal aberrations were induced in cultured mammalian cells, but there were no other indications of genotoxicity. The Meeting concluded that diazinon was not genotoxic.

Diazinon was evaluated in four human male volunteers who received 0.025 mg/kg bw/day of diazinon in capsules for 34-36 days. There were no consistent treatment-related effects on plasma or erythrocyte cholinesterase activity, blood chemistry or urinalysis. No clinical effects were reported. The NOAEL was 0.025 mg/kg bw/day.

The ADI of 0-0.002 mg/kg bw was maintained, which is based on the NOAEL of 0.025 mg/kg bw/day in the study in humans, using a 10-fold safety factor.

A toxicological monograph summarizing the data received since the previous evaluation and containing relevant data from the previous monograph and monograph addenda was prepared.

TOXICOLOGICAL EVALUATION

Level causing no toxicological effect

Rat: 5 ppm, equal to 0.4 mg/kg bw/day (90-day study)
1.5 ppm, equal to 0.07 mg/kg bw/day (99-week study)
10 ppm, equivalent to 0.5 mg/kg bw/day (reproduction study)
20 mg/kg bw/day (maternal toxicity in teratogenicity study)

Rabbit: 25 mg/kg bw/day (maternal toxicity in teratogenicity study)

Dog: 0.5 ppm, equal to 0.02 mg/kg bw/day (one-year study)

Human: 0.025 mg/kg bw/day (34-36-day study)

Estimate of acceptable daily intake for humans

0-0.002 mg/kg bw

Studies which will provide information valuable in the continued evaluation of the compound

Further observations in humans.

RESIDUE AND ANALYTICAL ASPECTS

Diazinon, originally evaluated by the JMPR in 1967 and re-evaluated for residues several times up to 1979, is included in the CCPR periodic review programme.

The general CXLs for fruits and vegetables (0.5 mg/kg) were retained by the 1990 CCPR, to await review by the 1993 JMPR.

Information on current world-wide GAP and extensive residue data were provided by one manufacturer and several countries.

Diazinon is an organophosphorus insecticide with a broad spectrum of activity against a

wide range of pests: sucking, chewing and boring insects, including soil-living insects. It is effective mainly by contact and stomach action. The product has been introduced world-wide in many countries and is used on numerous crop groups or commodities. It is generally used as a foliar or soil spray or applied as a granule to the soil.

Major target crops are leafy, fruiting, stem and root vegetables, deciduous fruit, rice and maize. Minor crops include berries, cereals, citrus, grapes, mushrooms, nut trees, olives and sugar beet. Additional uses for non-food crops are on ornamentals, grass and turf, and in nurseries.

Citrus fruits

Only limited data were available for oranges and mandarins which the Meeting felt were not sufficient to estimate a maximum residue level. Evidently the PHI (14-21 days) does not much influence residue levels. The Meeting recommended withdrawal of the CXL (0.7 mg/kg).

Pome fruits

The Meeting estimated a maximum residue level of 2 mg/kg (PHI 14 days), based on available trials from Germany, Switzerland and the USA on apples and pears.

Stone fruits

Cherries. Trials at dosage rates of 3.3 kg ai/ha applied 5 times showed residues up to 0.73 mg/kg 10 days after the last application. The Meeting recommended an MRL of 1 mg/kg.

Peaches. Taking into account data from Germany (PHI 14 days) and the USA (PHI 20 days), the Meeting estimated a maximum residue level of 0.2 mg/kg.

Plums (including Prunes). Trials at dosage rates of 3.3 kg ai/ha applied 5 times showed residues up to 0.78 mg/kg 10 days after the last application. The Meeting proposed an MRL of 1 mg/kg.

Prunes [dry]. US trials with dried prunes showed residues up to 1.9 mg/kg. The Meeting estimated a maximum residue level of 2 mg/kg.

Berries

Grapes. On the basis of the data available the Meeting felt unable to estimate a maximum residue level because the data do not match relevant GAP.

Strawberry. After observing the recommended PHI of 5 days the residues were below 0.1 mg/kg except one of 35 results, at 0.12 mg/kg. A maximum residue level of 0.1 mg/kg was estimated.

Cranberry. No results were reported within the reported GAP.

Currants, Black, Red, White. Results of trials in Germany and Switzerland showed residues up to 0.21 mg/kg at the recommended PHI of 14 days. The Meeting estimated a maximum residue level of 0.2 mg/kg.

Blackberries, Boysenberry, Raspberries. On the basis of US data and GAP for caneberries (PHI 7 days) the Meeting recommended an MRL of 0.1 mg/kg for blackberries and boysenberry, and 0.2 mg/kg for raspberries.

Olives. Because too few of the available residue data reflect current GAP the Meeting felt

unable to estimate a maximum residue level on olives or olive oil although processing studies are available which indicate an accumulation of diazinon in crude oil by a factor of 3-5. The Meeting recommended withdrawal of the CXLs for olives (2 mg/kg) and olive oil, virgin (2 mg/kg).

Tropical fruits

Persimmons. Only one trial reflected the reported GAP in New Zealand.

Banana. Only two trials reflected the current GAP in Costa Rica. Although no residues were found the Meeting felt unable to estimate a maximum residue level on such limited data.

Kiwifruit. On the basis of 6 new trials from New Zealand (PHI 28 days) the Meeting estimated a maximum residue level of 0.2 mg/kg.

Pineapple. Seven days after application (the PHI in Costa Rica) trials in Honduras and Costa Rica showed residues up to 0.07 mg/kg. Reported results from the USA were not taken into account because exaggerated application rates were used. The Meeting estimated a maximum residue level of 0.1 mg/kg.

Bulb vegetables

Onion, Bulb. At the recommended rate of application and the recommended PHI of 10 days residues were below 0.05 mg/kg, which was estimated as the maximum residue level.

Spring onion. The range of residue levels found in a series of trials was wide, between <0.01 and 0.65 mg/kg. A maximum residue level of 1 mg/kg, after the recommended PHI of 10 days, was estimated.

Brassica vegetables

Broccoli. Results of 10 trials from the USA within recommended GAP (PHI 7 days) showed residues up to 0.23 mg/kg. The Meeting estimated a maximum residue level of 0.5 mg/kg.

Cabbages, Head. On the basis of several trials from the USA within the recommended GAP (PHI 7 days) the Meeting estimated a maximum residue level of 2 mg/kg.

Cauliflower. The Meeting concluded that the reported data were insufficient to estimate a maximum residue level. Most of the trials were in Germany where there is no GAP. These results could be related to GAP in Switzerland (PHI 14 days), but only three trials included this PHI.

Kohlrabi. On the basis of the available trials and GAP in Switzerland (14 days PHI) the Meeting estimated a maximum residue level of 0.2 mg/kg.

Fruiting vegetables

Cucumber. On the basis of a PHI of 7 days (GAP in the USA) the Meeting estimated an MRL of 0.1 mg/kg. One value of 0.4 mg/kg was assumed to be an outlier because in that trial only 0.2 mg/kg was found 3 days after application.

Cantaloupe. Seven days after application (GAP in he USA) residues up to 0.18 mg/kg were found in the reported USA trials. The Meeting estimated a maximum residue level of 0.2 mg/kg.

Squash, Summer. In US trials residues up to 0.05 mg/kg were found 7 days after application.

An MRL of 0.05 mg/kg was recommended by the Meeting.

Mushrooms. Limited data from The Netherlands do not reflect the current PHI. The Meeting felt unable to estimate a maximum residue level.

Peppers, Sweet. Although no data were available for the recommended PHI of 5 days in the USA, the data from USA trials at PHIs of 3-7 days clearly show that 5 days after application residues would not exceed 0.05 mg/kg. The Meeting estimated a maximum residue level of 0.05 mg/kg for sweet peppers.

Sweet corn (corn-on-the-cob). No residues of diazinon were detectable (<0.01 mg/kg) 10-14 days after the last application. Seven days after the last application residues were small, <0.01-0.02 mg/kg. The Meeting proposed an MRL of 0.02 mg/kg.

Maize forage. Residues were detectable at levels of 0.04-7.95 and <0.01-4.95 mg/kg 7 and 14 days after the last application. The Meeting estimated a maximum residue level of 10 mg/kg.

Leafy vegetables

Tomato. On the basis of reported US data and a PHI of 1 day, the Meeting estimated a maximum residue level of 0.5 mg/kg.

Chinese cabbage; Kale, Chinese. Reported trials from Thailand on Chinese cabbage did not include the national PHI of 14 days but data from Thailand on Chinese kale showed residues below 0.02 mg/kg 14 days after application. Taking all the data on both commodities into account, the Meeting felt able to estimate a maximum residue level of 0.05 mg/kg for both Chinese cabbage and kale.

Lettuce, Head; Lettuce, Leaf. No data were available covering the PHI of 10 days registered in the USA where most of the trials were done. However, the great number of results at sampling intervals of 7-14 days clearly show that 10 days after application residues will be below 0.5 mg/kg. The Meeting estimated a maximum residue level of 0.5 mg/kg for both head and leaf lettuce.

Spinach. The Meeting concluded that the residue situation is comparable to that of lettuce and proposed an MRL of 0.5 mg/kg (PHI 10 days).

Legume vegetables

Common beans (pods and/or immature seeds). At a PHI of 7 days residues were at levels up to 0.2 mg/kg, which was estimated as a maximum residue level.

Garden pea, shelled. Residue results from the USA showed a comparable situation to that of common beans. The Meeting estimated a maximum residue level of 0.2 mg/kg (PHI 7 days).

Root vegetables

Carrot. After PHIs of 7 and 14 days residues were below 0.5 and 0.3 mg/kg, respectively. A maximum residue level of 0.5 mg/kg was estimated, covering the recommended PHI of 7 days.

Celeriac; Parsley, Turnip rooted. The results of only one trial were reported for each commodity. The Meeting therefore did not propose an MRL.

Potatoes. After the recommended PHI of 14 days residues were below the reported limit of determination. A maximum residue level of 0.01* mg/kg was estimated.

Radish. After PHIs of 7 and 14 days residues were below 0.1 mg/kg. A maximum residue level of 0.1 mg/kg was estimated, covering the recommended PHI of 10 days.

Sugar beet. At a PHI of 14 days residues were at levels up to 0.1 mg/kg, which was estimated as a maximum residue level.

Sugar beet leaves or tops. On the basis of the reported data and a PHI of 14 days a maximum residue level of 5 mg/kg was estimated.

Stalk and stem vegetables

Artichoke. Limited data available from Spain (3 results, one location) were insufficient to recommend an MRL.

Witloof chicory (sprouts). In the one trial reported no residues were detectable (<0.01 mg/kg) 14 days after application. No MRL was proposed.

Cereal grains

Maize. In all reported trials there were no detectable residues (<0.02 mg/kg) in the grain. Taking into account the reported results on sweet corn, the Meeting estimated a maximum residue level of 0.02* mg/kg.

Rice. Seven trials were conducted in India, Indonesia and Pakistan. No residues were detectable (<0.02 or <0.03 mg/kg) in the grain or the unhusked grain 23-59 days after the last application. Low residues were detectable in the straw, up to 0.04 mg/kg. Because the data did not match GAP in most of the countries the Meeting agreed not to recommend an MRL. The Meeting recommended withdrawal of the CXL for polished rice (0.1 mg/kg).

Tree nuts

Almonds. All results from the USA clearly show that normally there will be no residues in the nuts (<0.01 mg/kg). However in several cases residues up to 0.03 mg/kg occurred owing to contamination. The Meeting proposed an MRL of 0.05 mg/kg.

Almonds, hull. The available data were at PHIs of 14-45 days (and some at 0 days). The Meeting estimated a maximum residue level of 5 mg/kg (PHI 14-45 days).

Walnuts. On the basis of the available results of 24 supervised trials it is clear that no residues occur in nuts. The Meeting proposed the limit of determination as the MRL (0.01* mg/kg).

Oilseed

Cotton seed. On the basis of only four trials from one year and one country the Meeting felt unable to estimate a maximum residue level and recommended withdrawal of the CXL for cotton seed (0.1 mg/kg).

Animal products

Meat and milks. Residues in the milk and tissues of cattle were reported after applying ear tags to the animals (registered use in Canada).

Two ear tags (6% cypermethrin, 11% diazinon), one per ear, were attached to each of three Holstein dairy cows. Milk samples were taken five hours before application, and 5 h and 1, 3, 7, 14, 21, and 28 days after application.

Residues of diazinon in milk samples were not detectable (<0.0005 mg/kg) until three days after tag application. The residues remained consistently less than 0.002 mg/kg for the entire residue study.

Three Hereford steers were treated with two ear tags (6% cypermethrin, 9.6% diazinon), one per ear. After 14 days, one animal was slaughtered and samples of blood, liver, tongue, muscle, back fat, and kidney fat were analysed; after 100 days the remaining two treated animals were killed and similar samples were analysed.

Diazinon was found on the hair, but in the analysed tissues it was detectable only in the back fat and kidney fat of the animal killed 14 days after tag attachment. The levels were low, 0.032 and 0.035 mg/kg respectively. No residues (<0.01 mg/kg) were found in the back fat, kidney fat, liver, muscle and tongue 100 days after treatment, indicating that there was no accumulation.

Four Hereford steers were treated with two ear tags (20% diazinon) - one per ear. After 7 days, one animal was killed and samples of blood, liver, tongue, muscle, centre back fat, and kidney fat were analysed; a second steer was killed after 14 days and the remaining two after 28 days, all animals being sampled in the same fashion.

Residues were detectable in the centre back and kidney fat of all the animals. The highest levels were 0.045 and 0.041 mg/kg, respectively, on day 14. There were still detectable residue levels of diazinon in the centre back and kidney fat on day 28 at 0.02-0.03 mg/kg.

Data from planned animal transfer studies are not yet available. The Meeting recommended withdrawal of the established MRLs.

Withdrawal of the CXLS for Barley, Fruits (except ...), Hazelnuts, Leafy vegetables, Meat of cattle, pigs and sheep, Milks, Peanut, Pecan, Safflower seed, Sunflower seed, Vegetables (except ...), and Wheat is recommended because available residue data are insufficient although GAP is reported.

Metabolism in plants

Metabolism studies have been carried out on apples, beans, sweet corn, lettuce, potatoes and rice.

In processing

Processed fractions were prepared from apples, grapes, lettuce, endive, maize, pineapples, potatoes, sugar beet and tomatoes. Wine was made in some cases from harvest grapes and olive oil (crude) was prepared from olives in one trial. Generally residues of diazinon are reduced or not detectable in processed commodities with importance for human consumption: juice, sugar, and wine.

A concentration of residues was observed in crude olive oil and in pomace-type fractions, with the latter having a potential use as animal feed.

Residues in the edible portion of food commodities

No information was available about the partition of residues between the pulp and peel in citrus fruit, pineapple or cantaloupe.

No residues were detectable in the whole fruit or in separate samples of pulp and peel when diazinon was applied to bananas.

Residues in almonds (kernels) were low, ranging from <0.01 to 0.03 mg/kg 28-45 days after the last application.

Stability of pesticide residues in stored analytical samples

The stability of diazinon and the metabolites diazoxon (G-24576) and hydroxydiazinon (CGA 14128) under freezer storage conditions was determined in maize, tomatoes, potatoes, apples, strawberries, lettuce, soya beans (dry), refined corn oil, tomato paste and sugar beet molasses.

Residues of diazinon are generally stable in crops and processed commodities for a minimum of twenty-six months of freezer storage. A slight decline was observed in strawberries after three months storage, which continued at a much slower rate through twenty-six months.

Residues of diazoxon are unstable in crop and processed fraction substrates, but are stable in maize oil.

Residues of hydroxydiazinon are generally stable in crop substrates and processed fractions. A decline in residues was observed in apples and strawberries after three months of storage and continued at a much slower rate through twenty-six months.

The stability of diazinon under freezer storage conditions was further tested in some animal tissues, namely muscle, liver, kidney and fat of sheep. Diazinon was found to be stable for at least 8 months of storage.

Methods of residue analysis have been described for commodities of plant and animal origin with a limit of determination of 0.01 mg/kg in most commodities. Diazinon is classified as fat-soluble (octanol/water partition coefficient 3.95). In a large number of studies residues of the potential metabolites diazoxon (G 24576) and hydroxydiazinon (CGA 14128) were determined besides parent diazinon. Since these two compounds were practically not found in crops at harvest or in processed commodities it can be concluded that the use of the product according to GAP may be reliably monitored by determining the parent compound alone.

The data now available from the various metabolism studies show that major metabolites identified in plant and soil metabolism, namely 4-hydroxy-2-isopropyl-6-methylpyrimidine (G 27550), 4-hydroxy-2-(2-hydroxyprop-2-yl)-6-methylpyrimidine (GS 31144), and diazoxon (G 24576), also occur in animal metabolism (rat). Thus there is no need to include compounds other than diazinon in the residue definition.

FURTHER WORK OR INFORMATION

Desirable

1. Results of ongoing residue studies on citrus fruits, hazelnuts, hops, pecans and peanuts.

2. Data from dairy cattle transfer studies reported to be completed.

3. Additional information on registered veterinary uses including residue data.

4.13 DICHLORVOS (025)

TOXICOLOGY

Dichlorvos has been previously evaluated by the Joint Meeting in 1965, 1966, 1967, 1970 and 1977. An ADI of 0-0.004 mg/kg bw was allocated in 1966 and was maintained at subsequent Meetings. The compound was re-evaluated by the present Meeting in the CCPR periodic review programme.

Dichlorvos is rapidly absorbed by all routes of exposure and rapidly degraded. The metabolic pathways of dichlorvos are similar in mammalian species, including humans. Metabolites are rapidly excreted or incorporated into natural enzymatic pathways.

Dichlorvos has a marked acute oral toxicity with typical cholinergic signs and has been classified by WHO as highly hazardous.

Rat erythrocyte and brain cholinesterase inhibited by dichlorvos spontaneously reactivates with a half-life of about two hours both *in vitro* and *in vivo*.

Several carcinogenicity studies in mice and rats using routes other than gavage were negative, even when doses causing signs of toxicity were used. It should be noted that two squamous-cell carcinomas of the oesophagus have been observed in treated mice in one study.

In a two-year feeding study in rats (0, 0.1, 1, 10, 100 or 500 ppm), no neoplastic lesions were attributed to treatment. The NOAEL, based on brain cholinesterase inhibition, was 100 ppm (actual concentration 47 ppm, equivalent to 2.4 mg/kg bw/day).

In a carcinogenicity study in mice dichlorvos, administered by corn oil gavage (0, 10 or 20 mg/kg bw/day to males, and 0, 20 or 40 mg/kg bw/day to females), caused forestomach papillomas (statistically-significant positive trend with increased incidence in the high-dose female group). Elements of the mechanism by which these papillomas might arise have not been established, but the induction of hyperplasia in the forestomach was demonstrated. Additionally, genotoxic effects might occur at high local concentrations of dichlorvos (see below) as can be obtained in gavage dosing but not in dietary exposure. On the basis of the increased incidence of forestomach papillomas, the NOAEL was 10 mg/kg bw/day.

In a carcinogenicity study in Fisher 344 rats, dichlorvos administered by corn oil gavage (0, 4 or 8 mg/kg bw/day) caused an increased incidence of pancreatic adenomas (statistically significant in males only), mononuclear cell leukaemias (statistically significant in males only, no dose-response relation) and mammary gland adenomas or fibroadenomas (females only, no dose-response relation, statistically significant in the low dose group only). The Meeting observed that the incidence of pancreatic acinar adenomas in male control rats was unusually high and therefore the higher incidence found in treated animals was considered of questionable biological significance. The increased incidence of mononuclear cell leukaemia, which is usually high and variable in this strain of rat, was also of questionable biological significance. The doses used significantly inhibited plasma, but not erythrocyte, cholinesterase activity when measured three hours after treatment. However, given the rapid recovery of erythrocyte cholinesterase activity after inhibition by dichlorvos, the timing might have underestimated the inhibition.

Dichlorvos has been adequately tested in a series of *in vitro* and *in vivo* genotoxicity assays. The data indicate that dichlorvos is genotoxic in bacteria and cultured mammalian cells, but that it is not clastogenic *in vivo* except under conditions where an unusually high tissue dose can be attained. Dichloroacetaldehyde, a major metabolite of dichlorvos, is a weak bacterial mutagen. Positive results have been reported in mice given a dose of dichloroacetaldehyde far greater than that which could derive from sublethal doses of dichlorvos. Dichlorvos has been shown to methylate DNA *in vitro* at a rate that is 8-9 orders of magnitude lower than the rate of phosphorylation. DNA alkylation is therefore not likely to occur at doses of dichlorvos which are not inhibitory to erythrocyte/brain cholinesterase.

A three-generation reproduction study in rats was negative at doses up to 235 ppm in the diet, equivalent to 12 mg/kg bw/day. A one-litter, one-generation study in mice in which dichlorvos was administered by inhalation at doses which caused >90% plasma cholinesterase inhibition, but no signs of toxicity, was negative. Dichlorvos caused reversible damage of seminiferous tubules, Leydig and Sertoli cells at oral doses of 10 mg/kg bw daily for 18 days in mice and at 5 mg/kg bw and above every other day for 8 weeks in rats.

Dichlorvos appeared not to be teratogenic in mice, rats and rabbits at doses which caused maternal toxicity.

Dichlorvos caused delayed polyneuropathy in hens at doses much higher than the unprotected LD_{50}. Cases of delayed polyneuropathy also have been reported in humans after severe intoxications.

In humans, the rate of dichlorvos hydrolysis by plasma is similar to that in rats. The rate of recovery of inhibited erythrocyte and plasma cholinesterase activity in humans given dichlorvos is much slower than in rats. Half-lives of recovery are about 15 days in humans and about two hours in rats. A daily dose of 1 mg/kg bw to male human volunteers for seven days caused 5-30% inhibition of erythrocyte cholinesterase. The NOAEL in humans, based on the absence of erythrocyte cholinesterase inhibition in 12 volunteer males for 21 days, was 0.04 mg/kg bw/day.

In 1986, the Joint Meeting discussed the significance of carcinogenicity studies for organophosphorus pesticides and the requirements for further studies (Section 3.1 of the report). At that time none of the organophosphorus pesticides had caused a carcinogenic response in experimental animals. The 1986 Meeting recommended that, depending upon future evaluation on a case-by-case basis, further consideration should be given to the need for carcinogenicity studies for organophosphorus compounds.

In assessing the potential hazard to humans of residues of dichlorvos, the following considerations were taken into account in view of the weakly positive results in the gavage carcinogenicity study in mice.

Organophosphorus esters used as insecticides react with biological molecules by phosphorylation of serine hydrolases and alkylation of macromolecules. Phosphorylation of acetylcholinesterase and alkylation of DNA are considered to account for the acute cholinergic toxicity and initiation of the carcinogenic process, respectively. These biochemical reactions occur at different rates. When the rate of phosphorylation is substantially higher than the rate of alkylation, *in vivo* genotoxic effects are unlikely to occur because effective doses cannot be achieved owing to acute toxicity. Dichlorvos meets these criteria, the rate of phosphorylation of acetylcholinesterase being much faster (eight orders of magnitude) than that of alkylation of several macromolecules, including DNA. Hence positive mutagenicity tests were seen only *in vitro* and, as indicated in the 1986 Joint Meeting report, carcinogenicity studies are unlikely to give more information. The weak carcinogenic response of dichlorvos obtained in mice in a corn oil gavage study should be interpreted as a local effect of dichlorvos.

Information on comparative cholinergic toxicity might be of critical relevance for the extrapolation of toxic effects (other than acute effects) of organophosphates in experimental animals to humans. The characteristics of the interactions of a given compound with acetylcholinesterase (rates of phosphorylation, spontaneous reactivation and ageing) from different species can be compared *in vitro*. Also, the *in vivo* rate of reappearance of blood acetylcholinesterase activity can be measured. In some cases, metabolic degradation of organophosphates can be assessed comparatively by measuring the level of serum A esterase which hydrolyses a given compound. All these data make possible an improved assessment of the cholinergic toxicity of organophosphorus compounds in different species.

This knowledge may be of special significance in the case of dimethyl phosphates since the rates of *in vivo* reactivation vary substantially across species. Therefore, chronic dosing is more critical for extrapolation from animal data to humans. In a repeated dose regime, the longer the half-life of reactivation the more rapid and/or more toxic will be the resulting effect (hence in a chronic dosing regime, humans will be intoxicated by doses of dichlorvos which, when given alone, cause much less inhibition of erythrocyte/brain cholinesterase than rodents can tolerate). Therefore, comparison between the *in vivo* rates of recovery of enzyme activity will allow an assessment of the repeated doses of compounds and the resulting cholinesterase inhibition, which would represent the limiting factors for other toxicities (including mutagenicity and carcinogenicity).

In the case of dichlorvos the Meeting considered the relevance of carcinogenicity data derived from rodents to human safety, and concluded that the compound would not cause chronic human health hazards at doses below those which inhibit acetylcholinesterase.

The Meeting maintained the ADI, which is based on studies in humans with an NOAEL of 0.04 mg/kg bw/day, using 10-fold safety factor.

Some of the data that served as the basis for this review are summarized in WHO Environmental Health Criteria 79. A toxicological monograph summarizing new or not previously reviewed data as well as relevant data from previous monographs and monograph addenda on dichlorvos was prepared.

TOXICOLOGICAL EVALUATION

Level causing no toxicological effect

Mouse: 10 mg/kg bw/day (two-year study)

Rat: 47 ppm in the diet, equivalent to 2.4 mg/kg bw/day (two-year study)

Human: 0.04 mg/kg bw/day (21-day study)

Estimate of acceptable daily intake for humans

0-0.004 mg/kg bw

Studies which will provide information valuable in the continued evaluation of the compound

Further observations in humans.

RESIDUE AND ANALYTICAL ASPECTS

Dichlorvos was previously evaluated at the 1965, 1966, 1967, 1969, 1970 and 1974 Joint Meetings and it is included in the CCPR periodic review programme.

Dichlorvos, an organophosphorus insecticide, combines both contact and stomach action and has a marked vapour action. It is effective against a broad spectrum of insect pests in the field and in stored products. In addition to plant and stored product protection, it is often used in public health vector control and in animal health for ectoparasite control. The compound is registered for use on over 30 commodities in many countries.

Residues from supervised field trials or specific studies were evaluated for 45 commodities.

Following foliar application at recommended or double rates, the residues in most of the plant commodities tested (apples, avocados, beans, Brussels sprouts, cacao beans, cabbages, cauliflower, chilli peppers, cotton seed, cucumbers, dates, egg plants, lettuce, onions, peanuts, peas, rice, sorghum, soya beans, strawberries, tea, tomatoes, witloof chicory) were below the limit of determination (0.01-0.04 mg/kg, except tea 0.1 mg/kg). Detectable residue were reported in four commodities with maximum values of 0.05 mg/kg in cherries, 0.15 mg/kg in peaches, 0.08 mg/kg in grapes and 0.04 mg/kg in kale.

Residues in samples of endive, celery, spinach, cauliflower, leeks, cucumbers, paprika, tomatoes, radishes and blackberries moving in commerce in The Netherlands were below the limit of determination (<0.05 mg/kg). A residue of 0.1 mg/kg was found in one of 10 lettuce samples.

Following the post-harvest or indoor application of dichlorvos, residues were present in detectable amounts in beans (1.85 and 1.48 mg/kg after 14 and 21 days), wheat (0.41-5.0 mg/kg after 1-2 months), barley (0.14-0.74 mg/kg after 21 days), rice (0.12-0.23 mg/kg after 23-24 days), lettuce (0.2-0.4 mg/kg after 3-4 days) and mushroom (0.02-0.12 mg/kg after 1 day). The results of supervised trials on barley, wheat and rice were supported by the extensive studies in India on a wide variety of stored commodities including cereals.

The very limited data base for most of the crops and/or the discrepancy between the trial conditions and current GAP for beans, cacao beans, coffee beans, cotton seed, egg plant, indoor lettuce, peanuts, peppers and soya beans did not allow the estimation of maximum residue levels, although the Meeting took into account that the results are mutually supportive for many commodities.

The Meeting was also concerned about the lack of frozen storage stability tests of residues in fruit and vegetable samples in view of the period of several months which often elapsed between sampling and analysis, and the substantial decrease of dichlorvos residues in wheat samples stored at -15^{o}C.

In the absence of this information and as a result of the limited residue data, the previous recommendations for fruits and vegetables were withdrawn.

The animal metabolism studies showed that dichlorvos is readily absorbed, hydrolysed and effectively eliminated. The metabolic pathway was identical in the mammalian species studied. The studies on laboratory animals are discussed as part of the toxicological evaluation.

Residues of dichlorvos in the eggs of hens receiving a mist spray three times at a rate of 50 mg/m^3 were first detected 2 days after the first treatment. The residue concentration varied from <0.03 to 0.11 mg/kg during the trial. There was no increase in the concentration following consecutive treatments, and the residue declined below the limit of determination 3 days after the last application. Three birds were killed 18 hours after the 3rd treatment and the breast and leg muscle analysed. The residues found were between <0.01 and 0.05 mg/kg. Egg production was not affected by the treatments.

Various tissues of pigs which had received a single oral dose of vinyl-1-^{14}C-dichlorvos (ca. 40 mg dichlorvos/kg feed) were analysed at 2, 7 and 14 days after the treatment. The ^{14}C content of the tissues expressed as dichlorvos varied from 2.5 mg/kg (in brain) to 33 mg/kg (in liver) after 2 days, and from 1.9 mg/kg to 9.7 mg/kg after 14 days, but no dichlorvos, demethyl-dichlorvos, dichloroacetaldehyde or dichloroacetic acid could be detected.

Three cows were sprayed for 31 consecutive days with 59 ml of 1% dichlorvos solution for the control of horn fly and mosquitoes. Milk samples were collected at 2 hours and 1, 2, 4, 8, 16, 24 and 31 days. Tissue and blood samples were taken one day after the final treatment.

No dichlorvos was detected in any milk samples (<0.003 mg/kg) or body tissues (<0.002 mg/kg) from the treated cows.

Dairy cows were dosed orally at rates of 1.3, 1.8 and 2.6 mg/kg body weight with dichlorvos in the form of polyvinyl chloride pellets. The PVC formulation prevents substantial absorption by the animal, but may release the compound only much later in the manure. As the absorbed amount is unknown the non-detectable dichlorvos residues (<0.04 mg/kg) in milk samples, collected between 1 and 14 days, cannot be related to the dose.

In a dermal application experiment, six cattle and two dairy cows were sprayed once with 15 litres of an emulsion of NUVAN 100 EC at a concentration of 1500 mg/kg. Cattle were slaughtered 1, 3 and 7 days after treatment. Milk samples from two dairy cows were taken 6 h and 1, 3 and 7 days after treatment (control 1 day before treatment). No measurable residues of dichlorvos were found in milk (<0.005 mg/kg) or in muscle, liver, kidney or fat (<0.02 mg/kg).

In plants, the main routes of degradation of dichlorvos were found to be cleavage of the P-C bond to form the major metabolite dimethyl phosphate, demethylation to monomethyl phosphate and phosphoric acid, demethylation to demethyl-dichlorvos (a minor pathway), and loss by volatilization.

Cotton plants of the Deltapine Smooth leaf variety were grown in a glasshouse and treated by injecting aqueous solutions of ^{32}P-labelled dichlorvos (100 µg) into the petioles of individual, fully expanded leaves. Leaf samples were collected 1, 24 and 48 hours after the treatment. The analyses showed 81.2% loss as volatiles, 12.3% as dimethyl phosphate (the major metabolite), 2.2% as phosphoric acid + methyl phosphate and 0.1% as demethyl-dichlorvos (minor metabolites) within 48 hours of application. No parent residue was detectable at this time. Non-extractable residues corresponded to less than 5% of the applied radioactivity.

Greenhouse-grown bean, potato, and tomato plants of 15 cm height, with or without roots, were placed in formulated 0.1-0,2% ^{32}P-dichlorvos. Dichlorvos was degraded with a half-life of 6.8, 4.6 and 6.8 hours on beans, tomatoes and potatoes, respectively. Dimethyl phosphate was a major metabolite detected during the 24-hour test period. Volatile radioactivity amounted to about 60% in all three plant species.

Wheat grain at moisture levels of 18% and 10.6% was topically treated with ^{14}C-dichlorvos at a rate of 40 ìg ai/10 g sample. Treated samples were stored in sealed glass jars in darkness at 20°C and sampled over a period of 10 days. the uptake of dichlorvos was rapid at the higher moisture level. Within 2 days the aqueous-extractable bound activity reached a maximum and remained stable for 7 days, after which the phosphorylated protein was converted to a more stable demethyl form. The uptake rate decreased once all the protein had been phosphorylated, so excess dichlorvos would be lost from the grain by volatilization.

Dichlorvos broke down rapidly on grain to give mainly dimethyl phosphate (about 2 mg/kg at day 10) and phosphorylated protein derivatives, which are mainly water-soluble. Lesser amounts of demethyl-dichlorvos (about 0.5 mg/kg at day 10), monomethyl phosphate (about 0.2-0.25 mg/kg at day 10) and traces of phosphoric acid (undetectable-0.05 mg/kg at day 10) were also found.

The hydrolysis of dichlorvos at a concentration of 10 mg/l in aqueous media follows first order kinetics. The rate of hydrolysis appears to be strongly influenced by the ionic strength of the solution. Half-lives at 30°C determined at pH 1, 5, 7, 9 and 13 are of the order 74, 50, 18, 16 and 0.65 hours respectively. At 20°C and pH 13, the half-life value is 1.3×10^{-2} hours.

In micro-ecosystems containing sediments from a recultivated gravel pit and the drainage ditch of a fruit orchard, dichlorvos was very rapidly degraded to CO_2. After 16 days incubation, 76 and 69% of the applied 1.0 mg ^{14}C-dichlorvos/l was mineralized to $^{14}CO_2$ in the two

systems, respectively. Unchanged parent compound could be detected until day 7 (0.5%) and day 3 (3.1%), respectively. The following dichlorvos metabolites occurred as intermediary products: phosphoric acid, mono(2,2-dichloroethyl) monomethyl ester, sodium salt, demethyl-dichlorvos, 2,2-dichloroethanol, 2,2-dichloroacetaldehyde and dichloroacetic acid. Contamination of surface waters by dichlorvos is unlikely owing to rapid and thorough metabolism.

The photolytic degradation of dichlorvos was studied in water and aqueous methanolic solutions at 20 °C. It was degraded with a half-life of about 6 hours in water but was stable in methanolic solution.

Dichlorvos was applied to soil at a rate corresponding to l kg ai/ha and aged for 0, 2, 8 and 12 days. In a column leaching study 35%, 10%, 2% and <0.1% of the applied radioactivity was detected in the leachate respectively. Up to 15% of the radioactivity in the leachate was dissolved $^{14}CO_2$. 2,2-dichloroethanol and demethyl-dichlorvos corresponded to about 10% and 1% respectively. Parent dichlorvos was rapidly mineralized in the soil, and it was not detectable in any leachates. Within 2 days of ageing, 60-65% of the parent compound was detected as $^{14}CO_2$.

Dichlorvos was very rapidly degraded to the final mineralization product CO_2 in non-sterile standard soil 2.1 of the BBA and in a natural biologically active soil. After an incubation period of 2 days, 1.2% of the unchanged parent compound could be detected in soil 2.1 and none in the second system. During the same period 60-61% of the radioactivity applied as the parent compound was measured as $^{14}CO_2$. The decomposition of the active ingredient was slower in sterile standard soil 2.1. The half-life was 8.7 days. Less than 1% $^{14}CO_2$ was formed.

Dichlorvos was converted to dichloroethanol, dichloroacetic acid and ethyl dichloroacetate by a microbial enrichment derived from sewage.

The persistence of dichlorvos in stored commodities is strongly dependent on the temperature and moisture content or relative humidity (RH). A moisture content of 12% in wheat is roughly equivalent to 60-65% relative humidity. Reported half-lives are 10 days at 25°C and 12% moisture content, 25 days at 21°C and 9.3% moisture content, and 1.8 days at 35°C and 13.7%. The Australian Grain Industry uses an estimated half-life of 7 days at 30°C and 50% RH, and 28 days at 20°C and 50% RH.

Stored wheat lots treated with 6, 12 and 20 mg/kg dichlorvos were processed and the residues were analysed in milling fractions and baked products. The results indicate that the loss in processing may be largely attributable to the scouring and conditioning process. The ratios of dichlorvos residues in bran, germ and flour to wheat were on average 1.5, 1.0 and 0.1, respectively. The residues (mg/kg) detected were as follows: in white bread <0.02-0.2, in wholemeal bread and steamed bread 0.2-0.3, and in Arabic flat bread 0.3-0.6. In calculating the reduction in residue in the cooked products the differences in moisture content were taken into account by the following factors: 1.5 for white, wholemeal and steamed bread, 1.14 for flat bread, and 1.0 for noodles. This gives a loss of 75% of the dichlorvos residue in the production of pan breads, about 65% in the case of Arabic flat bread, which tends to retain a higher residue owing to the short (30 second) heat treatment, and 55% in white noodles. There is a 100% loss in yellow noodles owing to the alkali treatment (1% sodium carbonate by flour weight). There were no detectable residues in any cooked products produced from wheat stored for 3 months after treatment even at an application rate of 20 g/tonne.

In cacao beans about 99% of the residue was lost after roasting. Cacao butter contained 1.3-3.3% of the residues detected in the un-roasted cacao beans.

Washing removed 97%, 24% and 6% of the initial dichlorvos residues from potatoes, lettuce and endive. Cooking endive resulted in an 84% loss of residues.

Whole soya beans containing 0.92-1.5 mg/kg residues were processed into hulls, toasted hulls, flakes, crude oil and refined oil. Residues were 5.4 - 6.0 mg/kg, <0.02 mg/kg, 0.2 mg/kg, 0.55 mg/kg and <0.02 mg/kg respectively.

Storage stability tests of dichlorvos in wheat samples with moisture contents ranging between 13% and 17% indicated that 2 months after treatment 62.5% of the initial concentration (20 mg/kg) was lost at -15°C. When stored at 5°C, 50% and 80% was lost after seven and thirty days respectively. In another study, 50 mg/kg dichlorvos was added to wheat samples of 9.3-13.7% moisture content and stored at -15°C for 11 months. The losses were between only 2% and 22% at different moisture levels.

Although these two experiments showed two different levels of loss, they indicated that the breakdown of dichlorvos cannot be completely prevented even under cold storage conditions.

Dichlorvos can be determined by many published multi-residue procedures.

The samples from supervised trials before 1972 were analysed by an automated cholinesterase-inhibition method described in the 1970 Evaluations. Since then all samples have been analysed by gas chromatography using phosphorus-specific flame-photometric detectors.

FURTHER WORK OR INFORMATION

Desirable

1. Storage stability tests carried out on major commodities at or below -18°C.

2. Residue data on milled products of cereals other than wheat.

3. An animal transfer study on poultry.

4.14 DIQUAT (031)

TOXICOLOGY

Diquat was previously evaluated by the Joint Meeting in 1970, 1972, and 1977. An ADI of 0-0.008 mg diquat ion/kg bw was allocated in 1977.

When administered orally, ^{14}C-diquat was poorly absorbed from the gastrointestinal tract of rats, cows and goats and mainly eliminated via the faeces during the first 24 hours, the small part absorbed being principally eliminated via the urine. The total percentages of the administered doses eliminated via the faeces were 94, 91 and 94 for the rat, cow and goat respectively; 3.1% and 0.4% were eliminated in the urine of rats and cows, respectively, and very small percentages of radioactivity were found in the milk of cows and goats (0.004% and 0.0175% respectively).

After oral administration of ^{14}C-diquat to rats (45 mg ion/kg bw) the major excreted product was diquat in both urine (5% of dose) and faeces (>57% of dose): diquat monopyridone was the main metabolite in the faeces (5% of dose), but a minor one in the urine. In another oral study in rats (100 mg ion/kg bw), small amounts of diquat dipyridone and picolinic acid were found in addition to the monopyridone. After subcutaneous injection (10 mg ion/kg bw) in the rat, 75% of the dose was present in the urine as diquat, about 3% as the monopyridone and 6% as the dipyridone.

Unlike paraquat, diquat is not actively taken up by lung slices and lung toxicity is not characteristic of diquat poisoning.

The acute oral toxicity of diquat varies with species, but is between 125 and 250 mg ion/kg bw in rodents. It is classified by WHO as moderately hazardous.

In a 90-day feeding study in rats, using dietary concentrations of 0, 20, 100 or 500 ppm, the NOAEL was 100 ppm, equal to 8.5 mg ion/kg bw/day, based upon reduction in body-weight gain, food consumption and plasma protein at the next higher dose.

In a one-year feeding study in dogs, doses of 0, 0.5, 2.5 or 12.5 mg/kg bw/day were added to the feed. The NOAEL was 0.5 mg ion/kg bw/day, based upon lens opacity in females at the next higher dose.

Two long-term toxicity/carcinogenicity studies were conducted in mice. The first (80 weeks) used dietary concentrations of diquat ion of 0, 30, 150 or 500 ppm. The NOAEL was 30 ppm, equivalent to 4.5 mg ion/kg bw/day, based upon reduced growth rates at the next higher dose together with hepatic vacuolation in males. In a 2-year study in mice, in which dietary concentrations of 0, 30, 100 and 300 ppm were used, the NOAEL was 30 ppm, equal to 3.6 mg ion/kg bw/day, based on reduction in body-weight gain and increased relative kidney weights at the next higher dose. There was no evidence of carcinogenicity in mice.

Two two-year feeding studies have been conducted in rats. In the first, diquat dibromide was administered in the diet at concentrations of 0, 5, 15, 75 or 375 ppm. The NOAEL was 5 ppm, equal to 0.2 mg ion/kg bw/day, based upon cataract formation in the 15 ppm group. In the second study, dietary concentrations of 0, 15, 25 or 75 ppm diquat ion were used. The NOAEL was 25 ppm (equivalent to 1.3 mg ion/kg bw/day), based on cataract formation at the next higher dose. There was no evidence of carcinogenicity in rats.

Numerous teratogenicity studies have been conducted. NOAELs could not be determined in two mouse studies. There were three teratogenicity studies in rats; in the first study dietary concentrations of 0, 125 or 500 ppm diquat ion were used. A dose-related increase in subcutaneous fetal haemorrhages compared to the controls was observed. An NOAEL could not be derived from this study. In the second study, diquat was administered at oral doses of 0, 4, 12, 24 or 40 mg ion/kg bw/day. For fetal toxicity, the NOAEL was 24 mg ion/kg bw/day but maternal toxicity was observed in all test groups (reduced weight gain and food consumption). In the third study, diquat was administered by gavage at doses of 0, 4, 12 or 40 mg ion/kg bw/day. The NOAEL for both maternal and fetal toxicity was 12 mg ion/kg bw/day, based in the case of the dams on reduced body weight and food consumption and in the case of the fetuses on reduced fetal weight and defects in fetal ossification at the highest dose.

In a study in rabbits, diquat was given orally at doses of 0, 1.25, 2.5 or 5.0 mg ion/kg bw/day. There was no evidence of any effects on embryonic or fetal development. The NOAEL was 2.5 mg ion/kg bw/day, based on mild maternal toxicity at the highest dose. In a second study in rabbits, doses of 0, 1, 3, 7 or 10 mg ion/kg bw/day were administered by gavage. Doses of 3 mg ion/kg bw/day or above were associated with maternal toxicity as manifested by weight loss or reduced weight gain and reduced food intake. No evidence of fetotoxicity was observed. The NOAEL was 1 mg ion/kg bw/day, based upon maternal toxicity. In a third study in rabbits, doses of 0, 1, 3 or 10 mg ion/kg bw/day diquat were given by gavage. The NOAEL was 1 mg ion/kg bw/day, based upon maternal toxicity (reduced weight gain and food consumption) and skeletal effects in the fetuses at doses of 3 mg ion/kg bw/day.

Two multigeneration reproduction studies were conducted in rats. In the first study, diquat was given at dietary concentrations of 0, 125 or 500 ppm. This study did not exhibit an NOAEL, since there was decreased weight gain in F_0 and F_1 animals at the lowest dose, but the effects

observed at this dose (125 ppm, equivalent to 6.3 mg ion/kg bw/day) were trivial. In the second study, rats were fed diquat at dietary concentrations of 0, 16, 80 or 400 ppm. The NOAEL was 16 ppm (equivalent to 0.8 mg/kg bw/day), based upon a low incidence of partial cataract formation at 80 ppm.

Diquat has been adequately tested in a series of genotoxicity assays *in vitro* and *in vivo*. Chromosomal aberrations were induced *in vitro* but there was no other evidence of genotoxicity. The Meeting concluded that diquat was not genotoxic.

An ADI of 0-0.002 mg/kg bw was established, based upon an NOAEL OF 0.19 mg ion/kg bw/day identified in a two-year study in rats, using a safety factor of 100.

A toxicological monograph summarizing the data received since the previous evaluation and incorporating relevant sections from the previous monograph and monograph addenda was prepared.

TOXICOLOGICAL EVALUATION

Level causing no toxicological effects

Mouse: 30 ppm, equal to 3.6 mg ion/kg bw/day (two-year study)

Rat: 5 ppm, equal to 0.2 mg ion/kg bw/day (two-year study)
12 mg ion/kg bw/day (teratogenicity study)
16 ppm, equivalent to 0.8 mg ion/kg bw/day (multigeneration reproduction study)

Rabbit: 1 mg ion/kg bw/day (teratogenicity study)

Dog: 0.5 mg ion/kg bw/day (one-year study)

Estimate of acceptable daily intake for humans

0-0.002 mg ion/kg bw

Studies which will provide information in the continued evaluation of the compound

Observations in humans.

4.15 DITHIOCARBAMATES (105)

TOXICOLOGY

Based on data on the individual compounds, ADIs of 0-0.05 mg/kg bw could have been established for mancozeb and maneb and an ADI of 0-0.03 mg/kg bw could have been established for metiram. The Meeting concluded that the data base for zineb was inadequate to determine its own ADI.

Analytical methods for the determination of parent residues are non-specific. Residues are measured by the evolution of carbon disulphide, which is currently the only suitable regulatory method of analysis. Therefore, no distinction can be made as to the identification of the specific parent compound. Since no differentiation can be made between the parent EBDC residues of maneb, mancozeb, zineb and metiram the Joint Meeting established a group ADI of 0-0.03 mg/kg bw for these EBDCs, which was based on the lowest value within the group,

the ADI for metiram. Given the similarity of the chemical structure of zineb with the other EBDCs, and the comparable toxicological profiles of the EBDCs based on the toxic effects of ethylenethiourea (ETU), the Meeting included zineb in the group ADI. See the separate sections on ETU, mancozeb, maneb, metiram and zineb.

A separate ADI was established for ETU, the major common metabolite, degradation product and contaminant of all the EBDCs, based on its well-documented ability to inhibit thyroid function in rats, generally considered to be the most sensitive species.

The Meeting established an ADI of 0-0.007 mg/kg bw for propineb and a temporary ADI of 0-0.0002 mg/kg bw for propylenethiourea (PTU). The ADI for propineb is considerably lower than that allocated for the EBDCs, a consequence of which is discussed in Section 3.2.

RESIDUE AND ANALYTICAL ASPECTS

Mancozeb, maneb, propineb and their derivatives ethylenethiourea and propylenethiourea were scheduled for periodic re-evaluations at the 1993 JMPR (ALINORM 93/24A, para 133).

Extensive information on mancozeb, maneb and propineb was made available to the Meeting, and has been reviewed under those headings. Because the uses of the dithiocarbamates lead to a common residue, measured and expressed as CS_2, recommendations for MRLs are consolidated under DITHIOCARBAMATES (105), and listed in Annex I.

Information and recommendations on ethylenethiourea (ETU) are summarized under that heading.

4.16 ENDOSULFAN (032)

RESIDUE AND ANALYTICAL ASPECTS

Endosulfan has been reviewed by the JMPR eight times since 1967, including a major re-evaluation in 1989. At the 24th (1992) Session of the CCPR it was pointed out (ALINORM 93/24, paras 81-86) that the MRLs for head cabbages, Savoy cabbage and cauliflower did not reflect the residues expected from GAP in the USA. MRLs for broccoli, Brussels sprouts, head cabbage and Savoy cabbage were therefore held at Step 7B pending review by the present Meeting. The proposed deletion of the general MRLs for "fruit" and "vegetables, except as otherwise listed", as recommended by the 1989 JMPR, was also delayed until after this review.

Information on the current GAP of 21 countries was made available to the Meeting by the manufacturer, including full details from the USA. A large quantity of residue data that had not previously been submitted for review was also provided.

Residue data from supervised trials on many fruits, vegetables, cereals, oilseeds and beverage seeds which had not been reviewed previously were also provided and are recorded in the monograph on this compound. The Meeting confirmed that the data emphasised the desirability of withdrawing the current general MRLs for fruit and vegetables and replacing them with MRLS for individual commodities, usually at a lower level. It was also possible to make recommendations for MRLs on some additional crops.

The residue data on oranges were adequate to allow an MRL of 0.5 mg/kg to be recommended but the data on clementines and lemons were only in summary form and thus not sufficient to extend the MRL to the citrus fruit group.

Residues from trials on apples, cherries and plums were within the current MRLs of 1 mg/kg. Data for peach residues supported a similar MRL of 1 mg/kg. Residue data on grapes also allowed an MRL of 1 mg/kg to be recommended but the strawberry data were only summaries and were thus inadequate.

A dip treatment of pineapples with endosulfan is required in Australia for export quarantine purposes. The resultant residues are up to 2 mg/kg, within the CXL for "Fruits", and so an MRL of 2 mg/kg was recommended to cover this post-harvest use.

Unfortunately, the only residue data on brassica crops treated in the USA concerned two trials on Brussels sprouts that were carried out in 1964 and had been reported previously; at a 14-day PHI a maximum residue of 1.2 mg/kg was observed. In one trial on Brussels sprouts in the UK in 1976, 0.1 mg/kg was found after 14 days and 0.06 mg/kg after 21 days but the data were not adequate to support an MRL recommendation. Data from other countries under their GAP conditions were available for broccoli, head cabbage, Savoy cabbage and cauliflower; these results were consistent with the existing draft MRLs of 0.5, 1, 2 and 0.5 mg/kg, respectively.

For some other vegetables, currently covered by the CXL for "Vegetables, except as otherwise listed", the data presented were sufficient to allow recommendations to be made for broad bean, cucumber, melons except watermelon, summer squash and tomato, all at 0.5 mg/kg, and for soya bean at 1 mg/kg. Data for sweet peppers were inadequate.

Residues on celery (2 mg/kg), common bean (0.5 mg/kg), head lettuce (1 mg/kg) and potato (0.2 mg/kg) were within the respective CXLs.

For cereals, residue data were presented for maize and wheat, allowing recommendations of 0.1 mg/kg and 0.2 mg/kg, respectively, to be made.

Trials on some oilseeds gave sufficient residue data for MRLs to be recommended for cotton seed (1 mg/kg), rape seed (0.5 mg/kg) and sunflower seed (1 mg/kg).

New residue data were also available which allowed recommendations to be made for MRLs on cacao beans and coffee beans, both at 0.1 mg/kg.

Processing data were available for apples (juice and pomace), grapes (wine and must) and common beans (washing and cooking).

4.17 ETHEPHON (106)

TOXICOLOGY

Ethephon was evaluated at the 1978 JMPR, but an ADI was not allocated since the available toxicological data were insufficient. It is a plant growth regulator that acts by release of ethylene, influencing directly several physiological processes such as ripening and maturation and stimulating the production of endogenous ethylene. Since the compound in high concentration (>87%) is a waxy solid and difficult to handle, a technical product that contains 71% ethephon and 21% water is marketed and also used for most toxicological studies. Although ethephon is a dibasic phosphonic acid, its commercial formulation exhibits some anticholinesterase activity.

Following the oral administration of ethephon to rats, about 90% of the administered radioactivity was recovered, principally in urine (50%), expired air (19%), and faeces (6%) during 120 h after dosing. Most of the dose was recovered within 24 h.

After oral administration of ethephon to dogs, radioactivity was found in urine (40%),

expired air (30%) and faeces (5%). Total body retention was 1%. Peak plasma and red blood cell concentrations were observed 2 h after dosing. Only traces were observed after 22 h.

After oral administration to rats, ethephon was excreted in urine and faeces as the mono- and disodium salts and some unidentified metabolites, and metabolized to ethylene and eliminated in the expired air. In dogs, ethephon is partly metabolized to ethylene and eliminated in expired air and also excreted unchanged in the urine.

In dogs dosed orally with ethephon, plasma cholinesterase activity was inhibited at 2 h with recovery starting within a few hours. Erythrocyte cholinesterase levels responded more slowly with signs of recovery at 72 h.

Ethephon has a low oral acute toxicity in mice, rats, and rabbits. WHO has classified ethephon as unlikely to present acute hazard in normal use. Ethephon is corrosive to the skin of rabbits.

In a four-week study in mice at dietary concentrations of 0, 30, 100, 300, 1000 or 3000 ppm, the NOAEL was 300 ppm (equal to 51 mg/kg bw/day), based on inhibition of erythrocyte cholinesterase activity.

In a four-week study in rats at dietary concentrations of 0, 625, 1250, 2500, 5000 or 10000 ppm, the NOAEL was 625 ppm (equal to 52 mg/kg bw/day), based on inhibition of erythrocyte cholinesterase activity.

In a one-year study in dogs at dietary concentrations of 0, 100, 300, 1000 or 2000 ppm, the NOAEL was 1000 ppm (equal to 27 mg/kg bw/day), based on soft stools and changes in body and spleen weight. However, cholinesterase activities were not determined.

In a two-year study in dogs at dietary concentrations of 0, 30, 300, or 1500 ppm, the NOAEL was 30 ppm (equal to 0.86 mg/kg bw/day), based on inhibition of erythrocyte cholinesterase activity and smooth muscle hypertrophy in the stomach and small intestine.

In two 78-week studies in mice at dietary concentrations of 0, 30, 100, 300, 1000 or 10000 ppm, the NOAEL was 100 ppm (equal to 14 mg/kg bw/day), based on inhibition of erythrocyte cholinesterase activity. There was no evidence of carcinogenicity.

In two 104-week studies in rats at dietary concentrations of 0, 30, 300, 3000, 10000 or 30000 ppm, the NOAEL was 30 ppm (equal to 1.2 mg/kg bw/day), based on inhibition of erythrocyte cholinesterase activity. There was no evidence of carcinogenicity.

Brain cholinesterase was not depressed in any studies.

In a two-generation reproduction study in rats at dietary concentrations of 0, 300, 3000 or 30000 ppm, the NOAEL for maternal and filial toxicity was 300 ppm (equal to 22 mg/kg bw/day), based on reduced food intake, body weight and weight gain. There was no adverse effect on reproduction.

In two studies in hens for delayed neurotoxicity, no evidence of delayed neurotoxicity was observed.

In two oral teratogenicity studies in rats at dose levels of 0, 125, 200, 250, 500, 600, or 1800 mg/kg bw/day, the NOAEL was 600 mg/kg bw/day, based on maternal toxicity. There were no teratogenic effects.

In two teratogenicity studies in rabbits at oral dose levels of 0, 50, 62.5, 100, 125, or 250 mg/kg bw/day, the NOAEL was 50 mg/kg bw/day, based on maternal and embryo/fetotoxicity.

There were no teratogenic effects.

After reviewing the *in vitro* and *in vivo* genotoxicity data, the Meeting concluded that there was no evidence of genotoxicity.

In 16 male and female human volunteers treated orally with 3 x 40 mg/day of ethephon (approximately 1.5 and 2.2 mg/kg bw/day in males and females, respectively) for 28 consecutive days, no significant inhibitory effect on human plasma or erythrocyte cholinesterase activity was observed. Subjective complaints of urinary urgency, diarrhoea of sudden onset, effect on appetite and dyspepsia were recorded. An NOAEL could not be determined on the basis of the clinical symptoms.

In 30 male and female human volunteers treated orally with 0.5 mg/kg bw/day of ethephon (divided into 3 doses) for 16 consecutive days, plasma cholinesterase activity was inhibited but returned to its initial activity within the recovery period of 29 days. The NOAEL was 0.5 mg/kg bw/day, based on the lack of inhibition of erythrocyte cholinesterase.

In 20 male and female human volunteers receiving 0.17 or 0.33 mg/kg bw/day of ethephon orally (divided into 3 doses) for 22 consecutive days, plasma cholinesterase activity was inhibited and did not return within the recovery period of 14 days. An NOAEL of 0.33 mg/kg bw/day in both males and females was determined based upon the lack of inhibition of erythrocyte cholinesterase.

An ADI of 0-0.05 mg/kg bw was established, based on the NOAEL in the 16-day study in humans of 0.5 mg/kg bw/day, using a 10-fold safety factor.

A toxicological monograph was prepared.

TOXICOLOGICAL EVALUATION

Level causing no toxicological effect

Mouse: 100 ppm, equal to 14 mg/kg bw/day (78-week study)

Rat: 30 ppm, equal to 1.2 mg/kg bw/day (104-week study)

Dog: 30 ppm, equal to 0.86 mg/kg bw/day (two-year study)

Human: 0.5 mg/kg bw/day

Estimate of acceptable daily intake for humans

0-0.05 mg/kg bw

Studies which will provide information valuable in the continued evaluation of the compound

1. The Meeting noted that ethephon is a dibasic phosphonic acid and therefore not able to phosphorylate hydrolases at the serine residue. However, *in vivo* data showed inhibition of plasma and erythrocyte, but not brain, cholinesterase. Neither data with the pure compound nor *in vitro* studies were available. The Meeting considered that these effects on cholinesterases need clarification and recommended re-evaluation of the compound in 1995.

2. Further observations in humans.

4.18 ETHYLENETHIOUREA (ETU) (108)

TOXICOLOGY

Ethylenethiourea (ETU) was reviewed in conjunction with the ethylenebis(dithiocarbamate)s (EBDCs) by the Joint Meeting in 1963, 1965, 1967, 1970, 1974, 1977, 1980, 1986 and 1988. In 1988 the Joint Meeting extended the temporary ADI of 0-0.002 mg/kg bw pending the submission of additional data. ETU is also of interest because it forms part of the terminal residue to which consumers of produce treated with the EBDCs are exposed and because the levels of ETU in treated produce generally increase during food processing as the levels of the EBDC parent compounds decrease.

Following oral administration to mice essentially all of the ETU was recovered in the excreta within 48 hours; none was recovered as carbon dioxide. Approximately 50% of the administered dose was found in urine as unchanged ETU.

After the oral administration of radiolabelled ETU, its concentration in both pregnant mice and rats peaked at about the same time (1.4 hours), with concentrations in maternal and fetal tissues similar at 3 hours. The half-life of elimination from mice and rats was 5.5 hours and 9.4 hours respectively. Approximately 70% of the ETU was found in the urine in both species at 48 hours.

Mice metabolize ETU primarily by the flavin-monooxygenase (FMO) system and rats by the P-450 system of enzymes.

ETU is slightly toxic after acute oral administration, with the (LD_{50}) ranging from 545 mg/kg bw in pregnant rats to 4000 mg/kg bw in adult mice.

In a 13-week study in mice at dietary concentrations of 0, 125, 250, 500, 1000 or 2000 ppm the NOAEL was 250 ppm (equivalent to 38 mg/kg bw/day). Diffuse follicular cell hyperplasia of the thyroid and hepatocellular cytomegaly were observed at 500 ppm.

In a three-month study in mice at dietary concentrations of 0, 1, 10, 100 or 1000 ppm the NOAEL was 10 ppm, equal to 1.7 mg/kg bw/day. ETU produced thyroid follicular cell hyperplasia and decreased colloid density at 100 ppm.

The NOAEL in a study in which rats were fed dietary concentrations of ETU at 0, 0.63, 1.25, 2.5, 5.0 or 25 ppm for 8 weeks 25 ppm (equal to 2.6 mg/kg bw/day), the highest dose tested.

In a 13-week study in rats ETU was administered in the diet at concentrations of 0, 60, 125, 250, 500 or 750 ppm. The NOAEL was less than 60 ppm (equal to 3.0 mg/kg bw/day), based on histopathological findings of diffuse follicular cell hyperplasia in the thyroid.

In a 90-day study in rats ETU was administered in the diet at concentrations of 0, 1, 5, 25, 125 or 625 ppm. The NOAEL was 25 ppm (equal to 1.7 mg/kg bw/day), based on hyperaemia of the thyroids with and without enlargement, thyroid follicular cell hyperplasia, increased thyroid-to-brain-weight ratio, decreased ^{125}I thyroid uptake, decreased T_3 and decreased thyroxine at 125 ppm.

In a four-week feeding study in dogs at dietary concentrations of 0, 200, 980 or 4900 ppm, the NOAEL was 200 ppm, equal to 6.7 mg/kg bw/day. Decreased body-weight gain, decreased thyroxine and T_3 levels and enlarged thyroids were observed at 980 ppm.

In a 13-week feeding study in dogs at dietary concentrations of 0, 10, 150 or 2000 ppm the NOAEL was 10 ppm, equal to 0.39 mg/kg bw/day. At 150 ppm haemoglobin, packed cell volume, and red blood cell count were decreased, and cholesterol was increased. Effects on the thyroid were found only at 2000 ppm.

In a 52-week feeding study in dogs at dietary concentrations of 0, 5, 50, or 500 ppm, the NOAEL was 5 ppm, equal to 0.18 mg/kg bw/day. At 50 ppm a reduction in body-weight gain, hypertrophy of the thyroid with colloid retention, a slight increase in thyroid weight and pigment accumulation in the liver were observed.

Male and female mice received perinatal (F_0) and adult (F_1) exposure to ETU at the following dietary concentrations (F_0,F_1); 0,0; 0,330; 0,1000; 330,0; 330,330; 330,1000; 110,330; or 33,100 ppm. Mice receiving perinatal exposure only (330,0 ppm) showed no effect on the incidences of neoplasms after 2 years. Cytoplasmic vacuolization of follicular cells of the thyroid was evident in males and females at 33,100 ppm, but no increases in neoplasms of the liver, pituitary or thyroid were observed. T_4 values were significantly decreased in both sexes and TSH was slightly elevated. Animals receiving 330 ppm during adulthood showed tumours of either the liver, pituitary or thyroid. Increasing perinatal exposure from 0 to 330 ppm was associated with an increased incidence of thyroid and pituitary lesions in female mice receiving adult exposure to 330 ppm, but there were no enhancing effects of perinatal exposure in mice receiving adult exposures of 1000 ppm when compared to adults in the 0,1000 ppm group.

Rats were fed dietary concentrations of ETU at levels of 0, 5, 25, 125, 250 or 500 ppm for 2 years. The NOAEL was 5 ppm, equivalent to 0.25 mg/kg bw/day. Vascularity and hyperplasia of the thyroid were seen at 25 ppm.

In a two-year feeding study in rats using dietary concentrations of 0, 0.5, 2.5, 5 or 125 ppm the NOAEL was 5 ppm (equal to 0.37 mg/kg bw/day), based on changes in clinical chemistry, increased T_3, decreased T_4, increased thyroid weight, increased liver weight and an increased incidence and severity of diffuse thyroid follicular cell hyperplasia at 125 ppm.

In a two-year carcinogenicity study in rats using dietary concentrations of 0, 175 or 350 ppm, thyroid carcinomas and hyperplastic goitres were observed in both sexes at 175 ppm (equivalent to 8.8 mg/kg bw/day).

Male and female rats received perinatal (F_0) and adult (F_1) exposure to ETU at the following dietary concentrations (F_0,F_1); 0,0; 0,83; 0,250; 90,0; 90,83; 90,250; 30,83; or 9,25 ppm. Rats receiving perinatal and adult exposure of 9,25 ppm showed no increase in tumours and no apparent biologically meaningful changes in thyroid hormone function at two years when compared to 0,0 ppm controls. Thyroid hyperplasia was evident in both sexes. At 9 months, animals given 9,25 ppm manifested decreased T_3 and T_4 values and increased TSH without evidence of thyroid follicular cell hyperplasia. Males and females receiving a dose of 90,0 ppm showed no hormonal changes and no tumours at 2 years. Thyroid follicular cell hyperplasia was, however, evident. Animals receiving adult exposure showed a significant increase in thyroid follicular cell tumours at 83 and 250 ppm (males) and 250 ppm (females). Males and females showed no significant differences in the number of tumours between dose groups of 0,83; 30,83; and 90,83 ppm. Males and females receiving 90,250 ppm showed increases in thyroid follicular cell tumours when compared to 0,250 ppm. At the end of 2 years males and females receiving 0,83 or 0,250 manifested increased numbers of thyroid tumours when compared to 0,0 ppm controls.

In a two-generation reproduction study in rats at dietary concentrations of 0, 2.5, 25 or 125 ppm the NOAEL was 2.5 ppm, equal to a range of 0.16-0.38 mg/kg bw/day, based on thyroid gland follicular cell hyperplasia and hypertrophy at 25 ppm.

An oral teratogenicity study conducted in rats at dose levels of 0, 5, 10, 20, 40 or 80 mg/kg bw/day indicated no maternal toxicity at 40 mg/kg bw/day (NOAEL). Maternal lethality was observed at 80 mg/kg bw/day. The NOAEL for embryo/fetotoxicity effects was 5.0 mg/kg bw/day, based on teratogenic effects observed at 10 mg/kg bw/day.

An oral teratogenicity study in rats at dose levels of 0, 15, 25 or 35 mg/kg bw/day was conducted. No maternal toxicity was observed at 35 mg/kg bw/day (NOAEL). The NOAEL for embryo/fetotoxicity and teratogenicity was 15 mg/kg bw/day, based on higher incidences of dilated brain ventricles at 25 mg/kg bw/day.

Oral teratogenicity studies in rats (0, 10, 20, 30, 40 or 50 mg/kg bw/day), mice (0, 200, 400 or 800 mg/kg bw/day) and hamsters (0, 90, 270 or 810 mg/kg bw/day) revealed no maternal toxicity at the doses tested. The NOAEL for embryo/fetotoxicity in the rat was 10 mg/kg bw/day, based on dilation of the lateral or fourth ventricle at 20 mg/kg bw/day. The NOAEL for embryo/fetotoxicity in the hamster was 90 mg/kg bw/day, based on a decrease in fetal body weight at 270 mg/kg bw/day. The NOAEL for mice was higher than 800 mg/kg bw/day.

In an oral teratogenicity study, rabbits received 0, 5, 10, 20, 40 or 80 mg/kg bw/day of ETU. The NOAEL for maternal toxicity was 80 mg/kg bw/day. The NOAEL for embryo/fetotoxicity was 40 mg/kg bw/day, based on an increase in resorption sites, decreased brain weight and a degeneration of the proximal convoluted tubules in the kidneys of fetuses at 80 mg/kg bw/day. Malformations were not observed at the highest dose.

A study with pregnant rats administered ETU, T_3/T_4 and sodium iodide in combination indicated a reduction in some of the teratogenic responses when compared with groups administered ETU alone. These results indicate that the teratogenic potential of ETU may in part be secondary to the thyroid toxicity of ETU.

ETU has been the subject of many *in vitro* and *in vivo* studies for genotoxicity. It induces mutations in bacteria at very high doses but variable responses have been obtained in other types of mutation assays. Acceptable assays for other genotoxicity endpoints *in vitro* were generally negative, while all *in vivo* assays were negative. The Meeting concluded that ethylenethiourea was not genotoxic.

The ADI is based upon an NOAEL of 0.39 mg/kg bw/day in the 13-week dog study since this dose level is between the NOAEL of 5 ppm (equal to 0.18 mg/kg bw/day) and the middle dose (effect level) of 50 ppm (equal to 1.79 mg/kg bw/day) of the 52-week dog study. A 100-fold safety factor was applied.

A toxicological monograph was prepared, summarizing the data that have been received since the previous evaluation and incorporating the previous monograph and monograph addenda on ETU.

TOXICOLOGICAL EVALUATION

Level causing no toxicological effects

Mouse: 10 ppm, equal to 1.7 mg/kg bw/day (3-month study)

Rat: 5 ppm, equal to 0.37 mg/kg bw/day (two-year study)
2.5 ppm, equal to a range of 0.16-0.38 mg/kg bw/day (reproduction study)

Dog: 10 ppm, equal to 0.39 mg/kg bw/day (13-week study)

5 ppm, equal to 0.18 mg/kg bw/day (52-week study)

Estimate of acceptable daily intake for humans

0-0.004 mg/kg bw.

Studies which will provide valuable information in the continued evaluation of the compound

Observations in humans.

RESIDUE AND ANALYTICAL ASPECTS

Ethylenethiourea (ETU) is a metabolite and decomposition product of the ethylenebis(dithiocarbamate) (EBDC) fungicides. MRLs have been established to reflect maximum residue levels in raw agricultural commodities at harvest. ETU was scheduled (ALINORM 93/24A, Appendix IV, Annex I) for periodic (toxicological and residue) re-evaluation by the 1993 JMPR.

Extensive data were made available to the Meeting on ETU residues in raw agricultural commodities from supervised trials, in processed foods from supervised trials, and in raw and processed commodities in trade, and on the production of ETU in the plant and animal metabolism of mancozeb and maneb.

ETU residues in raw agricultural commodities were generally low (0.1 mg/kg or less) or undetectable (LOD mostly 0.01-0.02 mg/kg). Some reported ETU residues could be an artefact of the analysis, because a small percentage of the ethylenebis(dithiocarbamate) residues can be converted to ETU during the determination.

Animal metabolism and animal transfer studies with mancozeb and maneb on lactating dairy cows, lactating goats and laying hens showed that ETU was a minor metabolite and that ETU residues in milk, eggs and tissues arising from ethylenebis(dithiocarbamate) (EBDC) feed residues would normally be very low or undetectable.

ETU was either undetectable or a minor residue in plant metabolism studies with applied mancozeb or maneb. Where ETU was detected, it was mostly in surface rinsings.

ETU was generally short-lived when applied to plant leaves or soil. It was rapidly degraded by UV light.

Ethylenebis(dithiocarbamate) residues are readily converted in part to ETU if processing includes a heating step. Levels of ETU in processed products bear no relationship to the ETU levels in the raw commodities. ETU levels in processed commodities depend on the levels of EBDC which are present at crucial stages where heating takes place and the duration and temperature of that heating.

Under a US Food and Drug Administration monitoring programme (1990-1991) a variety of baby foods (864 samples) were monitored for pesticide residues. ETU residues were detected in 65 samples; the highest levels detected were 0.06 mg/kg. In 1989-90 in the USA a large survey of food items (approximately 300 samples each of 19 different raw and processed commodities) was conducted for dithiocarbamate and ETU residues. No measurable residues of ETU (LOD 0.001 mg/kg) were found in 82% of the samples. All ETU residue levels were less than 0.1 mg/kg. ETU was not detected (LOD 0.005 mg/kg) in any of 100 commercial grape juice samples in the USA taken from producers using grapes from areas where dithiocarbamate fungicides were used.

The Meeting agreed that MRLs for ETU did not assist in deciding whether GAP in the use of EBDCs was being followed. The Meeting agreed to recommend the withdrawal of all MRLs for ETU.

Normally the regulation of a residue in the raw agricultural commodity sets a limit on the levels in processed food because some or all of the residue is lost during the process. The levels of ETU in the processed commodity bear no relation to the levels in the raw agricultural commodity. ETU is more likely to occur in processed food where it can be generated by the heating of EBDC residues during the process.

Processing trials demonstrate that under some conditions considerable conversion of EBDCs to ETU can occur. Processing studies available to the Meeting showed that an initial commercial washing and cleaning of the raw agricultural commodity removes much of the EBDC, which is a surface residue, and reduces the potential for ETU formation.

The extensive food surveys in the USA, which have included many processed foods, have generally found only low levels of ETU (less than 0.1 mg/kg) and only in a minority of samples (fewer than 20%). The data suggest that, if good processing practices are followed, ETU residues in processed food would rarely exceed 0.1 mg/kg.

The 1990 JMPR reported results of the monitoring of food in commerce or at consumption for ETU in Canada for 1975-1985. Residues in a number of processed products were all below the limit of detection (0.05 mg/kg). Limited 1989-1990 data from Canada on a variety of fruit juices and drinks showed residues to be below 1 µg/kg.

4.19 ETOFENPROX (184)

2-(4-ethoxyphenyl)-2-methylpropyl 3-phenoxybenzyl ether

The compound was considered for the first time by the present Meeting.

Etofenprox is an insecticide with an action similar to the pyrethroids. It is active against a wide range of insect pests and is effective against strains of rice green leafhopper and planthoppers resistant to organophosphorus and carbamate insecticides.

Etofenprox is mostly formulated as 20% wettable powders or 20% emulsifiable concentrates.

TOXICOLOGY

After oral administration to rats, total excretion was 85-90% in faeces and 7-9% in urine. Excretion in bile was found to be 10 to 30%. Unchanged etofenprox was not found in the bile. Total retention in the body after 5 days was 3-4%. In the gastrointestinal tract from 48 to 93% was absorbed. Absorption tended to be dose-dependent. Tissue concentrations were highest in fat; this residue was present as unchanged parent compound. Etofenprox was excreted (as the unchanged compound) in milk. The major biotransformation routes involve *O*-de-ethylation of the ethylphenyl moiety and hydroxylation of the phenoxybenzyl moiety followed by conjugation with glucuronide or sulphate. Oxidation of the á-CH_2 group followed by hydrolysis represents an additional route. The available results for dogs indicate a lower gastrointestinal absorption rate than in rats. The major biotransformation routes were the same as in rats.

Etofenprox has a low acute oral toxicity in mice, rats and dogs. WHO has classified etofenprox as unlikely to present acute hazard in normal use.

In a 13-week study in mice, using dietary concentrations of 0, 50, 500, 3000 or 15000 ppm, the NOAEL was 500 ppm, equal to 60 mg/kg bw/day. The main effects seen were mortality, growth retardation, increased weights of liver (with enlarged hepatocytes) and kidneys (with tubular basophilia and dilatation) and decreases in red blood cell counts and haemoglobin concentration.

In a 13-week study in rats using dietary concentrations of 0, 50, 300, 1800 or 10800 ppm, the NOAEL was 300 ppm, equal to 20 mg/kg bw/day, based on effects on growth and the liver. In addition, increased thyroid weight with increased incidence of microfollicles in this organ was observed.

In a 52-week study in dogs using dietary concentrations of 0, 100, 1000 or 10,000 ppm, the NOAEL was 1000 ppm (equal to 32 mg/kg bw/day), based on decreased red blood cell counts, haemoglobin concentration and packed cell volume, increased serum alkaline phosphatase and increased liver weight (with swelling of hepatocytes).

In a two-year toxicity/carcinogenicity study in mice using dietary concentrations of 0, 30, 100, 700 or 4900 ppm, the NOAEL was 30 ppm (equal to 3.1 mg/kg bw/day), based on an increased incidence of tubular lesions in the kidneys at ≥100 ppm. There was no evidence of carcinogenicity.

A two-year toxicity/carcinogenicity study in rats also used dietary concentrations of 0, 30, 100, 700 or 4900 ppm. The NOAEL was 100 ppm (equal to 3.7 mg/kg bw/day), based on increased weights of thyroid and kidneys and microscopic liver changes at ≥700 ppm. The incidence of cystic follicles in the thyroid was increased only at 4900 ppm. There was an increased incidence of thyroid follicular adenomas among the 4900 ppm animals, which was statistically significantly increased only in females. The absence of genotoxicity of etofenprox (see below) in combination with the observed activation of the thyroid gland, which might be related to the effects on the liver (the latter probably leading to increased breakdown of thyroid hormones), is a strong indication for a non-genotoxic mechanism of induction of the thyroid tumours.

In a two-generation study in rats using dietary concentrations of 0, 100, 700 or 4900 ppm, the NOAEL was 100 ppm (equivalent to 5 mg/kg bw/day). No effects on reproduction were observed. The main effects seen in parents as well as young were decreased growth and effects on the weights and histopathology of liver and kidneys. The effects on the offspring were consistent with exposure to unchanged etofenprox via milk.

Embryo/fetotoxicity and teratogenicity were studied in rats (3 studies, segment I, II & III, respectively) and rabbits (1 study). In the three studies in the rat, etofenprox was administered by gavage at dose levels of 0, 12.5, 250 or 5000 mg/kg bw/day. In each of the studies, dose-related maternal toxicity (clinical signs, growth retardation) was observed at 5000 mg/kg bw/day. In the two studies in rats with dosing before or during pregnancy, no effects on offspring/fetuses were seen. In the study in rats with dosing during lactation (segment III), toxic effects developed in the offspring, most likely as a result of exposure to etofenprox via milk. In none of the studies in rats were irreversible structural malformations found. The NOAEL for maternal or parental toxicity in each of these studies was 250 mg/kg bw/day. For fetotoxicity, the NOAEL was 5000 mg/kg bw/day in the segment I and II studies. The NOAEL for neonatal effects in the segment III study was 250 mg/kg bw/day. In the study in rabbits the NOAEL for maternal toxicity was 10 mg/kg bw/day, based on decreased growth at 50 and 250 mg/kg bw/day. Incidences of late abortions and early-embryonal mortality were increased only at 250 mg/kg bw/day. The NOAEL for embryo/fetotoxicity was 50 mg/kg bw/day. No irreversible structural malformations were noted in this study.

On the basis of the results of the available *in vitro* and *in vivo* genotoxicity data there was no evidence that etofenprox is genotoxic.

The most sensitive species in the animal studies presently available appear to be rodents, with NOAELs of 3.1 and 3.7 mg/kg bw/day for mice and rats, respectively, in the long-term studies. The ADI was based on the long-term study in mice, using a 100-fold safety factor.

A toxicological monograph was prepared.

TOXICOLOGICAL EVALUATION

Level causing no toxicological effects

Mouse: 30 ppm, equal to 3.1 mg/kg bw/day (long-term toxicity/carcinogenicity study).

Rat: 100 ppm, equal to 3.7 mg/kg bw/day (long-term toxicity/carcinogenicity study).

Rabbit: 10 mg/kg bw/day (maternal toxicity in a teratogenicity study).

Dog: 1000 ppm, equal to 32 mg/kg bw/day (52-week study).

Estimate of acceptable daily intake for humans.

0 - 0.03 mg/kg bw.

Studies which will provide information valuable in the continued evaluation of the compound.

1. Clarification of the dose-response relation for thyroid effects in the rat, including evaluation of T_3, T_4, TSH and other relevant parameters.

2. Observations in humans with adequate information on exposure levels.

RESIDUE AND ANALYTICAL ASPECTS

Degradation studies were carried out on etofenprox in plants (beans and rice) and soil. Metabolic studies were also carried out in animals (rats and dogs), but information about these was only available to the Meeting in a summarized form.

The metabolism of etofenprox in bean and rice plants was examined by applying á-^{14}C-benzyl-labelled and 1-^{14}C-propyl-labelled etofenprox to leaves of the plants under laboratory conditions. There was very limited translocation of the parent compound and its metabolites to other parts of the plants, including the seeds in rice. Etofenprox was gradually decomposed on and in the treated leaves and was reduced to approximately 50% after 3 weeks. The main metabolite from the oxidization of etofenprox was 2-(4-ethoxyphenyl)-2-methylpropyl 3-phenoxybenzoate. Residues of other metabolites, mainly 3-phenoxybenzoic acid and 2-(4-ethoxyphenyl)-2-methylpropan-1-ol, were also present but in small quantities. The half-life of etofenprox on beans was determined to be 3 weeks for both labelled forms. At that time the main metabolite accounted for 11-15% and unrecovered compounds for 14-18% of the radioactivity applied. Experiments have shown that all metabolites observed on the bean leaves, except conjugates, were very similar to products formed by photodegradation, implying that the metabolism on plant leaves is affected by light.

Degradation studies on etofenprox in soil were carried out with three different soil types using the same two ^{14}C-labelled forms as in the experiments on plants. The half-life of etofenprox in soil was determined to be 6-9 days and largely independent of the soil types and labelled forms used. The main products formed after oxidation were 2-(4-ethoxyphenyl)-2-methylpropyl 3-phenoxybenzoate and 2-(4-ethoxyphenyl)-2-methylpropyl 3-hydroxybenzyl eth-

er. The liberation of $^{14}CO_2$ from the degradation of ^{14}C-labelled etofenprox was examined. After 2 and 8 weeks the radioactivity originating from CO_2 was 8-12 and 32-44%, respectively. The degradation of etofenprox in soil was evidently caused by micro-organisms as no degradation occurred in sterilized soil.

Field studies were carried out to examine the rate of degradation of etofenprox in paddy and upland soils. The half-lives in the two paddy soil types examined were 79 and 62 days, while the half-lives in the two upland soils were 39 and 9 days. Etofenprox is strongly adsorbed to soil, and little leaching takes place. No residues of the parent compound and only small quantities of metabolites were detectable in the effluents from three soil types after 2 weeks of leaching.

Supervised trials were carried out on several kinds of fruits and vegetables in Japan, on apples and potatoes in Hungary, and on potatoes and rape in Poland. In Japan trials were also carried out on rice, wheat, corn and tea. The application rates were different in the three countries. In Japan the rates were generally of the order of 0.5-1.5 kg/ha, while the highest rate in Hungary was 0.15 kg ai/ha and in Poland 0.09 kg ai/ha. Residues in apples were 0.4-0.8 mg/kg in Japan and 0.1-0.2 mg/kg in Hungary. Residues in potatoes were below the limit of determination in all the trials, including those in Japan at the highest dose rate.

Residues were determined in animal products after feeding experiments on dairy cows. Cows were fed with etofenprox at levels of 10, 30 and 1000 mg/animal/day, where 1000 mg/day represents a considerably higher level of intake than would occur in practice. After a feeding period of 28 days residues in milk from cows fed with 10 and 30 mg/day were at or below the limit of determination (0.05 mg/kg), but residues from 1000 mg/day were up to 2 mg/kg. Residues in tissues were also examined after the feeding period. Residues in liver, kidney and skeletal muscle from 10 and 30 mg/day were at or below the limit of determination, but in the peritoneal and subcutaneous fat were quite high and up to 0.84 mg/kg. For cows fed with 1000 mg/day, residues were up to 14 mg/kg in peritoneal fat and up to 3.5 mg/kg in subcutaneous fat, and were also present in kidney, liver and muscle.

Residues of etofenprox in plant material and soil are determined by gas chromatography with an electron capture detector after extraction with acetone and clean-up by partitioning with water/n-hexane and by column chromatography on Florisil or alumina/silica gel. For most crops and soil the purified extract is reacted with trimethylsilyl iodide to form 3-phenoxybenzyl iodide. The limit of determination is 0.01 mg/kg. For milk and animal tissues the method is similar, but ethyl acetate/hexane is used for the extraction and a silica sep-pak is used for the chromatographic clean-up. The limit of determination for residues in animal products is 0.05 mg/kg.

The manufacturer informed the Meeting that the analytical methods described, including the chromatographic clean-up step, are specific for the parent compound etofenprox and do not determine other compounds containing the 3-phenoxybenzyl moiety.

Supervised trials for most crops were carried out in only one country, Japan, and although they were at two sites they took place within the same year. The Meeting was therefore able to propose maximum residue limits for etofenprox in only two crops.

FURTHER WORK OR INFORMATION

Desirable

1. Submission of documentation for the specificity of the analytical methods for the determination of etofenprox.

2. Supervised trials on crops from more than one year and trials carried out in more than one country.

3. Studies on the processing of crops containing residues of etofenprox

4. Residues in straw from wheat and other crops used as animal feedingstuffs.

4.20 FENBUTATIN OXIDE (109)

RESIDUE AND ANALYTICAL ASPECTS

Fenbutatin oxide, a miticide registered for use on many crops world-wide, was first reviewed by the 1977 JMPR for both toxicology and residues. A toxicological re-evaluation in the periodic review programme of the CCPR was conducted in 1992, but the corresponding residue review was postponed to 1993 owing to the late arrival of data. Although the present Meeting reviewed over 250 individual reports or studies containing residue data and GAP information, little or no information was provided on some critical supporting studies (e.g. plant, goat and hen metabolism studies, processing studies for tomatoes, freezer storage stability of analytical samples, analytical methods, etc.). The Meeting received processing studies for apples, grapes and citrus and, on request, cow metabolism and transfer studies as well as a chicken feeding study. The Meeting was informed that rat, hen and goat metabolism studies had been submitted to WHO and could be submitted to FAO for future review. A proposed LC analytical method for fenbutatin was also reviewed.

GAP and summary residue data received from Spain and The Netherlands for a number of commodities were received too late for full consideration. The GAP information has been added to the 1993 Monograph. Most if not all of the summary residue information appears to have been included in earlier submissions for 1993 review and has therefore been considered.

There was still a lack of critical supporting information, with the exception of the cow and chicken feeding studies and processing studies provided. Accordingly, the Meeting limited this periodic review primarily to evaluating supervised trials data and/or evaluating data in the context of the available information on current GAP.

Supervised trials data show fenbutatin oxide residues in general to be primarily on the surface or in the peel. Residues in banana pulp are 1-2% of the level in the whole banana; peeled cucumber residues are $\leq$33% of whole cucumber residues; citrus pulp residues are <5% of the whole fruit residues. In nuts residues in the shell are typically 25 times those in the nut meat, although in a few cases only 3 to 4 times. In almonds the hull residues were of the order of 60 times the level in nut meat.

Processing information was provided on a number of commodities. Washing may remove 20 to 40% of the residues on fruits and an even higher proportion in some cases on citrus fruits. Concentration occurs in some processed fractions, with concentration factors of 1.7 in wet apple pomace, 6 in dry apple pomace, 5 in dry citrus pulp, 6.7 in citrus oil, 4.3 in wet grape pomace, 18 in dry grape pomace, 4.3 in dried grapes, 2.4 in dried prunes, and up to 9 in dried peaches. Residues from GAP applications to grapes are $\leq$0.02 mg/kg in wine or grape juice.

In a number of studies samples were also analysed for residues of the metabolites dihydroxybis(2-methyl-2-phenylpropyl)stannane (SD31723) and 2-methyl-2-phenylpropylstannoic acid (SD33608). The former is with few exceptions $\leq$10% of the fenbutatin oxide residue and the latter usually $\leq$1/2 the level of the SD31723. There is some evidence that the canning process may reduce residues near the MRL to non-detectable levels, at least in stone fruit.

Avocado. There is no MRL for avocado. Because data were available only for the flesh and no GAP was available for the countries in which trials were conducted, the Meeting concluded that information was insufficient to support a limit.

Banana. There is currently no MRL for bananas. Maximum residues reflecting GAP were 6.3 mg/kg at 7 days, 3.4 mg/kg at 2 days and 5.7 mg/kg at 0 days. The GAP PHI is 1 day. Approximately 1-8% of the whole fruit residue has been found in the pulp (maximum level 0.14 mg/kg), although ≤2% is likely to be a more reliable estimate taking into account analytical factors. Although few of the results were exactly at the GAP PHI, residues show little decline over 7 days from application, and the Meeting concluded that data over this period were relevant to estimating a maximum residue level. Because results were available from only one country, the Meeting considered additional data reflecting the GAP of other countries desirable. However, because results were available from three locations in three different years, the Meeting concluded that they were sufficient to estimate a 10 mg/kg limit.

Beans. There is no MRL for beans. Residues in green beans from a single application in a single trial in one country were 0.5 mg/kg after 3 days. Data were available from another country at a slightly exaggerated application rate (75 g ai/hl instead of 50 g ai/hl) from a formulation which is not recognised as GAP. The plot size was only 12 M^2. The Meeting could not recommend a limit for green beans.

Residues in French beans from 2 applications at GAP rates under glasshouse conditions in one country resulted in maximum residues of 0.4 mg/kg after 6 days compared to a GAP PHI of 7 days. At twice the 25 g ai/hl GAP rate residues were 0.15 mg/kg after 7 days. The Meeting concluded that the data were insufficient to support a limit for beans.

Citrus. The CXL for citrus fruits is 5 mg/kg. Although 43 trials were conducted in 5 countries, 3/4 of these were in one country and only 12 of the trials represented current GAP (6 on oranges, 2 on grapefruit, 3 on lemons and one on mandarins). Maximum residues resulting from GAP were 1.5 mg/kg in grapefruit, 2.4 mg/kg in mandarins, 3.3 mg/kg in oranges and 4 mg/kg in lemons, the last at a 21-day PHI compared to the GAP 7 day PHI. Other trials at GAP application rates, but at twice the GAP number of applications resulted in residues up to 14 mg/kg, but generally less than 10 mg/kg. Residues of the metabolite SD31723 were typically 2-10% of the parent compound in whole oranges and in all the fruits the residue of SD33608 tends to be about half that of SD31723.

While additional data reflecting GAP for oranges, grapefruit and mandarins are desirable, the Meeting concluded that the available results were marginally sufficient in a mutually supportive way to confirm the existing 5 mg/kg citrus group CXL for these individual citrus fruits. This does not apply to lemons or limes, because relatively few trials representing GAP were available for lemons. As noted, the highest GAP residue (4 mg/kg) in lemons was at a 21-day PHI compared to a GAP 7-day PHI, and the next highest residue of 2.4 mg/kg was from only single applications whereas two are permitted. The Meeting concluded that additional data reflecting current GAP for lemons and/or limes with the minimum PHI and maximum application rates would be required before a limit could be recommended for lemons or limes or for citrus fruits as a group. Additional data reflecting GAP for oranges, grapefruit and mandarins are also desirable.

The available information from the simulated commercial processing of oranges with field-incurred residues indicates that residues in dry orange pulp are 2-4.6 times those in whole unwashed oranges. Assuming a fivefold concentration and a residue at the 5 mg/kg MRL level in the unprocessed fruit, the maximum dry pulp residue would be 25 mg/kg compared to the current 7 mg/kg limit. Although there is currently no Codex MRL for citrus oil, the 6.6 concentration factor from whole unwashed oranges indicates that an MRL of 30 mg/kg would be needed.

Cucumbers. The CXL for cucumbers is 1 mg/kg. Data were available from 4 European countries (open and glasshouse) and the USA (open), although the US data do not correspond to current GAP. While application rates in the US trials were higher and PHI intervals shorter than required by GAP in Europe, the data are useful for illustrating the dependence of residues on the application rate and giving some indication of differences in residues between 2 and 3 applications. Residues from single applications representing European GAP ranged from 0.03 to 0.3 mg/kg. The Meeting concluded that a 0.5 mg/kg limit was supported.

Egg plant. The CXL for egg plant is 1 mg/kg. Because data were available from only a single glasshouse trial in one country, because no GAP information was provided for that country and because the trials did not conform to GAP application rates or PHIs of neighbouring countries the Meeting concluded that the information was insufficient to support the limit, and recommended its withdrawal.

Gherkins. The CXL for gherkins is 1 mg/kg. Because only summary information from a single glasshouse trial was available, the Meeting concluded that it was insufficient to support the limit, and recommended its withdrawal.

Grapes. The CXL for grapes is 5 mg/kg. Data were available from over 60 supervised trials in 5 countries. Maximum residues of approximately 2 mg/kg resulted from European GAP and 4 mg/kg from US GAP. While US GAP is comparable to that of several European countries, many more US trials were available. The Meeting confirmed the existing 5 mg/kg limit. Currently there is no Codex limit for processed grape products. Because the concentration of residues from grapes to raisins is approximately fourfold, the Meeting concluded that an MRL of 20 mg/kg for raisins would be appropriate, based on 5 mg/kg in the whole fruit. Similarly, concentration of the order of 18 times in dry pomace (or stem waste) supports a limit of 100 mg/kg for dry pomace.

Hops. No MRL is proposed for fenbutatin oxide in hops. Supervised trials data reflecting GAP were available from a single trial in one country, resulting in residues of the order of 5 mg/kg. The Meeting concluded that these data were inadequate for estimating a maximum residue level.

Melons. Currently there is a 1 mg/kg CXL for melons, except watermelons. Although no residues (<0.01 mg/kg) resulted 7 or 14 days after applications at GAP rates in a single trial in one country whose GAP PHI is 3 days, the Meeting concluded that the data were insufficient to support the limit, and recommended its withdrawal.

Nuts. No MRL is established for nuts. Maximum residues of fenbutatin oxide in the nut meats of almonds, pecans, walnuts and filberts from treatments at US GAP application rates, PHIs and number of applications included residues of $\leq$0.02, 0.04, 0.05, 0.13, 0.16, and 0.3 mg/kg (the last at a 1.5-fold application rate). Appreciably more data were provided which were at GAP application rates and PHI, but with 3 applications instead of the maximum 2 per season which is GAP. These included residues of $\leq$0.02 (10); 0.03 (4); 0.04 (2); 0.05; 0.07; 0.08; 0.1; 0.2 (3) and 0.3 mg/kg.

Although there were no side-by-side comparisons, the overall data suggest similar residues from 2 or 3 applications at GAP rates. The Meeting therefore concluded that a 0.5 mg/kg limit could be supported for almonds, pecans, and walnuts. Residues were observed up to 56 mg/kg in almond hulls (an animal feed item).

Data were too limited to support a limit for filberts, for which no GAP information was provided.

Data were also available for metabolites SD31723 and SD33608 with levels of <0.02 mg/kg in nut meats.

Peppers. The CXL for peppers (sweet) is 1 mg/kg. Data were available from two supervised trials. The single application (2 are permitted) in the Belgian trial was outdoors, although the provided Belgian GAP was for glasshouse uses. The Netherlands glasshouse trial was at 0.5 kg ai/ha, which could not be related to the GAP rate of 25 g ai/hl. Although residues in two supervised trials (1 and 0.6 mg/kg after the 3-day PHIs) suggest that residues may not exceed 1 mg/kg from GAP, the Meeting did not consider two data points reflecting GAP sufficient to support the MRL, and recommended its withdrawal.

Pome fruit. The CXLs are 5 mg/kg for apples and pears and 20 mg/kg for dry apple pomace. The data base for pome fruit included 103 supervised trials for apples and 17 for pears. In many trials residues decreased little during 2-3 weeks after application. Maximum residues in apples were 2.9 mg/kg from GAP applications in non-US trials. In US trials the highest residues from GAP applications were 4.3 mg/kg from dilute sprays, 9.6 mg/kg from concentrated SC sprays and 12 mg/kg from concentrated WP sprays, although only three of the many trials on apples were with concentrated sprays at GAP application rates. The two apple trials which resulted in the higher residues were on a different variety from those in other trials, but it could not be concluded that the variety influenced the residue. While the trial plot was a single tree, this was also true of other trials. Higher residues from concentrated spray applications are also suggested by pear trials where maximum residues reflecting US GAP were 2.3 mg/kg for dilute sprays and 5.6 and 3 mg/kg for WP and SC concentrated spray applications respectively. These dilute and concentrated spray applications were all on the same variety of pear.

While the Meeting concluded that the current 5 mg/kg Codex limit is adequate for dilute spray applications, it would not accommodate the USA concentrated spray uses. The Meeting concluded that additional data reflecting GAP would be needed to accommodate these.

The Codex limit for dry apple pomace is 20 mg/kg to accommodate the current 5 mg/kg limit on apples (fourfold concentration factor). Concentration and reduction factors in apple processed products estimated from studies provided to this Meeting varied, depending on the study: juice (unclarified) 0.6 times, wet pulp 1.7-3.5 times, and dry pulp 6-12 times. Clearly the fourfold factor previously used by the JMPR is too low in view of this information. Putting greater weight on the most comprehensive processing study provided, the Meeting concluded that for estimating maximum residue levels a factor of 7 would be reasonable for whole fruit to dry pomace. With an MRL of 5 mg/kg and a concentration factor of 7 a 40 mg/kg limit can be recommended for dry apple pomace.

Raspberries. No MRL exists. Supervised trials information was available for only one country, for which no GAP information was provided and the GAP of other countries could not be used. The Meeting concluded that insufficient information was available to estimate a maximum residue level.

Soya beans. Because residue results (<0.01 mg/kg) were available from only three supervised trials in a single country 67-80 days after application compared to the GAP 7-day PHI, and because analytical recoveries by analytical method SAMS 345-1 were highly variable at a 0.2 mg/kg fortification level (50-110%), the Meeting concluded that data reflective of GAP were insufficient to support a limit.

Stone fruit

Forty-seven studies from 8 countries were available for stone fruit, representing 76 supervised trials. Most of the results referred to de-stoned fruit. No attempt was made to calculate residue in the whole fruit including stone, since average stone weights were only about 6% of the whole fruit weight.

Cherries. The CXL is 5 mg/kg. Maximum residues reflecting approximate GAP in Germany were 0.6 mg/kg and in the USA 5.1 mg/kg (whether results were adjusted for 69% recoveries was not stated). If not corrected, a maximum residue of 7.4 mg/kg would be indicated. Other US data also did not indicate whether corrections had been made for low recoveries. If not, other residues when adjusted for recoveries would be of the order of 7 mg/kg. Results from The Netherlands data did not reflect the national 42-day PHI. However, the residues up to 1.2 mg/kg were from applications consistent with German or Italian GAP PHIs, although German GAP was reported to be due to expire in 1993. Maximum residues of SD31723 were 0.9 mg/kg and SD33608 0.04 mg/kg from GAP. The former was ≤25% of the fenbutatin oxide residue and SD33608 is usually less than half of the level of SD31723.

The Meeting was particularly concerned at the lack of information on whether results from several US studies (the major portion of the data base) were corrected for analytical recoveries less than 70% (58% in one case), and at the information that German reregistration is to expire in 1993 (the German GAP is relevant to other European trials for which no GAP was provided). The Meeting concluded that a 10 mg/kg limit could be supported for cherries.

Peaches, nectarines. The CXL is 7 mg/kg. Maximum residues in peaches reflecting approximate GAP in Australia were 2.5 mg/kg, and in the USA 8 mg/kg from a 1.5-fold rate (5.3 mg/kg adjusted to the GAP rate) and up to 5.8 mg/kg from approximate GAP rates, but with 3 instead of the permitted 2 applications. Residues were up to 3.5 mg/kg in two US trials reflecting GAP on nectarines. Maximum peach residues were 0.8 mg/kg in Canada at US GAP rates; 1.3 mg/kg in France at German GAP rates; and 3.3 mg/kg in Germany. Three trials in South Africa also resulted in residues up to 3.1 mg/kg after 10 days and 4 mg/kg after 13 days (14 day-PHIs are common in other countries) at application rates which are GAP in other countries, although GAP information for South Africa was not provided. Residues were up to 6 and 7.8 mg/kg after 13 and 10 days respectively at higher application rates. The Meeting concluded that the data supported the current 7 mg/kg limit for peaches and in a mutually supportive way could support a limit at the same level for nectarines.

Plums. The CXL is 3 mg/kg. Maximum residues approximating GAP were: German trials 0.7 mg/kg; United States trials 2.1 mg/kg; Netherlands trials <0.1 mg/kg. No GAP information was available for South Africa. Residues were 0.9 and 1 mg/kg after 14 days. From GAP applications, maximum residues of SD31723 were 0.07 mg/kg and of SD33608 0.04 mg/kg. SD31723 is usually <5% of the fenbutatin oxide residue and SD33608 is similar to or lower than SD31723. Control values for fenbutatin oxide range from <0.01 to 0.1 mg/kg, depending on the analytical method used.

Although recoveries in some US trials were below 70% and no information was provided on whether the results were corrected, recoveries were acceptable in the trial with the highest GAP residue (2.1 mg/kg). Furthermore, assuming that the results in the trials with low recoveries are uncorrected, maximum residues would be about 2.2 mg/kg. The Meeting concluded that the data were sufficient to support the CXL for plums.

There is no MRL for prunes (dried plums). Data provided indicate that fenbutatin oxide residues are concentrated in drying plums by a factor as high as 2.5. Applying this to the 3 mg/kg limit for fresh plums would imply an MRL of 7.5 or 10 mg/kg for dried prunes.

Residue levels in dried plums from trees treated in accordance with GAP were provided, although no data were included for the fresh fruit from which a concentration factor could be estimated. Maximum residues were 3.1 mg/kg. Analytical recoveries for this study were only 55% and it was not indicated whether the result had been corrected for the low recovery. If not, a residue of 5.7 mg/kg would be indicated. This would be consistent with the theoretical 7.5 mg/kg estimated above.

Strawberries. The CXL is 3 mg/kg. Twenty-seven reports were available from 7 countries

representing 47 supervised trials (32 from the USA). Data from two countries could not be related to the available GAP. Maximum residues approximating GAP were 1.3 mg/kg from Australian trials, 0.4 mg/kg from French trials, 0.5 mg/kg from UK trials, and 7 mg/kg from Mexican trials (based on US GAP). The more numerous US trials resulted in a fairly continuous distribution of residues, except for two values, up to 9.9 mg/kg (the last from a 1.2-fold application rate). The exceptions were at one site with residues of 12 and 18 mg/kg. Because information on the project history for these trials was in question and because the residues (especially 18 mg/kg) were not consistent with those found in numerous other similar trials, even at exaggerated rates, the Meeting gave little weight to these two values.

Maximum residues from GAP of the metabolites SD31723 and SD33608 were respectively 0.1 and 0.05 mg/kg after 1 day. Generally residues of SD31723 were ≤5% of fenbutatin oxide residues and SD33608 residues were about half or less of the SD31723 residues (after one day). The Meeting concluded that the data supported an increase in the current 3 mg/kg CXL to 10 mg/kg.

Tomato. The CXL is 1 mg/kg. Four of the 12 supervised trials were according to GAP, and the maximum residues in these: Denmark 0.4 mg/kg (glasshouse); Italy 0.3 mg/kg (field); the UK 0.3 mg/kg (glasshouse). Although results were available from 3 additional countries, they could not be related to the GAP information provided. Residues were up to 0.8 mg/kg after 3 or 4 days in two trials that could not be confirmed to reflect GAP. No tomato processing data were provided. No residues (<0.1 mg/kg) of metabolite SD31723 were found in the two trials in which it was determined.

The Meeting concluded that the data were adequate to confirm the current limit for tomatoes, but only for glasshouse uses.

Animals. Feeding studies with labelled fenbutatin oxide at 34 ppm dietary feeding levels indicate that the greatest potential for residues is in the kidney and liver of cattle, with possible low residues in muscle. Conventional feeding studies were also conducted at 11 or 96 ppm in the cattle diet for 21 or 22 days. No residues (<0.02 mg/kg) were found in milk, cream or tissues from the lower feeding level. Residues of fenbutatin oxide were found in all cream and tissue samples from the higher feeding level, while SD 31723 was found only in the liver and kidney. SD 33608 was not detected in any sample (<0.02 mg/kg).

Depending on the assumptions used, a dietary intake of the order of 20 ppm could be estimated, about twice the level in the lower feeding level trial. Adjusting data from the highest feeding level trial to a 20 ppm dietary burden results in maximum fenbutatin oxide residues in liver of 0.02, kidney 0.05, fat of meat 0.01, muscle 0.01 and milk fat 0.05 mg/kg.

Again depending on what assumptions are made, a case could be made for a slight lowering of the previously estimated 0.2 mg/kg limits for liver and kidney, but since the levels are not much greater than the validated limits of determination for these organs, and because more than one of the feed items could be fed at one time, the Meeting concluded that the liver and kidney limits previously estimated for cattle, goats, pigs, horses and sheep could be confirmed. They have been combined under a new proposal at the same level for edible offal.

The Meeting had some reservations about the previous estimates for cattle meat and milk of 0.02 mg/kg at the limit of determination. There was no evidence that the levels would be exceeded in practice, but the analytical method had not been validated below 0.1 mg/kg for any animal matrix in studies provided to the Meeting. For this reason the Meeting recommended increasing the stated limits of determination and hence the MRLs for these commodities to 0.05 mg/kg and limits for the meat of cattle, dogs, horses and sheep have been combined at the same level as a new proposal for meat.

The Meeting also observed that residues of SD 31723 can be about twice those of

fenbutatin oxide in cattle liver. Because residues of fenbutatin oxide are found in liver and because it is the only matrix in which SD 31723 exceeds fenbutatin oxide, the Meeting concluded that definition of the residue solely as fenbutatin oxide is satisfactory.

Residues in skim milk and cream indicate a propensity for fenbutatin oxide to accumulate in lipid rather than aqueous media, but levels in muscle do not differ from those in mesenteric or subcutaneous fat sufficiently to regard fenbutatin oxide as a fat-soluble pesticide.

Feeding chickens at 5 ppm dietary levels produced no residues of fenbutatin oxide or its two metabolites in tissues or eggs, except 0.02 mg/kg fenbutatin oxide in two whole egg samples. From the 25 ppm dietary feeding level the maximum residues of fenbutatin oxide were 0.04 mg/kg in liver, 0.03 mg/kg in kidney and 0.12 mg/kg in whole eggs. These decreased to <0.02 mg/kg in liver and kidney 3 days after cessation of feeding, but the decrease was slower in whole eggs. No residues of either parent compound or metabolites were found in other tissues or organs. As in the case of cattle, residues of SD 13723 were greater than those of fenbutatin oxide in liver (3 to 5 times as high in this case) and residues of SD 33608 were generally comparable to those of the parent compound.

If it is assumed that the greatest dietary intake from feed items for which there are MRLs would be from dry grape pomace (100 mg/kg MRL) and that it is fed at a maximum of 5% of the diet, a dietary intake of approximately 5 ppm can be estimated. Maximum fenbutatin oxide residues of 0.02 mg/kg in whole egg from the 5 mg/kg feeding level and 0.12 mg/kg from the 25 ppm level support 0.02 mg/kg as a maximum residue level for whole eggs. While SD 317243 might occur near 0.02 mg/kg in liver (0.12 mg/kg from 25 ppm feeding), residues of fenbutatin oxide *per se* would not be expected to be above 0.02 mg/kg. Although residues would be likely not to exceed 0.02 mg/kg in whole eggs, kidney or liver, the same considerations as those mentioned above regarding the levels of method validation for cattle products led the Meeting to conclude that a limit of 0.05 mg/kg (not a limit of determination, because residues around 0.02 mg/kg may occur) would be more appropriate in whole eggs and 0.05 mg/kg (as a limit of determination) in liver and kidney. There would be no compelling need for a limit in poultry meat or fat. Because limits are proposed for eggs and chicken edible offal, 0.05 mg/kg is recommended for chicken meat as a limit of determination level.

Only one of the two analytical methods used in the supervised trials was provided, although the principles were summarized and recoveries and limits of detection were usually provided with field trials data. The two basic methods were both described in earlier monographs. The first is based on chloro-derivatization in a solvent containing HCl followed by GLC determination. The second (e.g. method MMS-R-494-1 provided to the Meeting) includes methylation of fenbutatin oxide, SD 31723 and SD 33608 with methyllithium and determination by GLC with flame-photometric detection of tin. In general determination at 0.02 to 0.05 mg/kg of each compound in cream, 0.1 mg/kg of each in cow liver, and 0.1 to 0.2 mg/kg SD 31723, 0.05 mg/kg SD 33608 and probably $\geq$0.1 mg/kg parent compound in grapes appears to be supported by sample chromatograms. Recoveries from the various substrates were generally $\geq$80%, but at near MRL levels, especially for fenbutatin oxide.

The submitted method may be adequate for regulatory analysis at proposed MRL levels, although submission of all of the analytical methods with sufficient information to permit estimation of the limits of determination and of any information on multi-residue methods suitable for enforcement is desirable.

A proposed liquid chromatographic procedure was also provided, but it was not validated sufficiently for the Meeting to recommend its use.

FURTHER WORK OR INFORMATION

Desirable

1. Information on whether residues in US stone fruit trials in 1993 Monograph Table 8 references 5,6,7,9 (cherries), 21, 23, 24, (plums), and 29, 30 (peaches), were corrected for analytical recoveries.

2. Information on South African GAP for the use of fenbutatin oxide on peaches.

3. Submission of the analytical methods used in the supervised field trials and in the cow feeding study TIR-26-119-73, with validation information.

4. Current information on analytical methods suitable for enforcement for both plant and animal foods, including multi-residue methods.

5. Current information on the stability of residues in stored analytical samples.

6. Current information on the fate of residues in poultry, plants, soil and water/sediment systems. Metabolism studies on rats, goats and hens reportedly submitted to WHO are specifically requested .

7. Information on residues in food in commerce or at consumption.

8. Information on the interval between the last feeding and slaughter in cow feeding study TIR-26-119-73 (Koos, 1973).

9. Submission of Report 22-112-74 (on the fate of residues), referenced in Potter and Nugent (1978), as the basis for analyses of animal products for fenbutatin oxide, SD 31723 and SD 33608.

10. Additional pome fruit data reflecting US concentrated spray GAP.

11. Tomato processing information.

4.21 FENPROPATHRIN (185)

(*RS*)-á-Cyano-3-phenoxybenzyl-2,2,3,3-tetramethylcyclopropanecarboxylate

Fenpropathrin was considered for the first time by the present Meeting. It is an ingestion and contact synthetic pyrethroid insecticide and acaricide formulated as an EC and used against various species of Acari, Aleyrodidae, Aphididae and Lepidoptera on cotton, grapes, ornamentals, fruits, vegetables and other field crops. Most countries approve a range of application rates (e.g. apple: 0.06 kg ai/ha, 2 applications in Hungary - 0.45 kg ai/ha, 8 applications in the USA). Normally, the effects of a treatment last for 3 -4 weeks.

TOXICOLOGY

After oral administration of fenpropathrin to rats, the compound was almost completely absorbed and eliminated in urine and in faeces. The major biotransformation reactions consist of oxidation at the methyl groups of the acid moiety and at the 2′- and 4′- positions of the alcohol moiety, and cleavage of the ester linkage followed by glucuronide, sulphate or glycine conjugation.

Fenpropathrin has been tested for acute toxicity and it has been classified as moderately hazardous by WHO.

In a short-term feeding study in rats conducted at dietary concentration levels of 0, 3, 30, 100, 300 or 600 ppm over thirteen weeks, the NOAEL was 300 ppm, equal to 17 mg/kg bw/day, based on reduced body-weight gain and the appearance of clinical signs at higher dose levels. In a second 13-week rat study the NOAEL was 150 ppm, equal to 8 mg/kg bw/day, based on depression of body-weight gain at higher dose levels.

A one-year study in dogs conducted at dose levels of 0, 100, 250 or 750 ppm revealed an NOAEL of 100 ppm, equal to 3 mg/kg bw/day, based upon reduced body weight gain and clinical signs (emesis, tremors) at 250 ppm.

A long-term toxicity/carcinogenicity study was performed in mice over 104 weeks at 0, 40, 150 or 600 ppm. The NOAEL was 600 ppm the highest dose tested, 600 ppm, equal to 56 mg/kg bw/day. There was no evidence of carcinogenicity.

In a long-term toxicity/carcinogenicity study in rats conducted at dietary concentrations of 0, 1, 5, 25, 125 or 500 ppm over two years, the NOAEL was 125 ppm, equal to 5 mg/kg bw/day, based on depression in body-weight gain at 500 ppm. There was no evidence of carcinogenicity.

A second long-term toxicity/carcinogenicity study in rats performed at dietary concentrations of 0, 50, 150, 450 or 600 ppm over two years revealed an NOAEL of 150 ppm, equal to 7 mg/kg bw/day, based on the appearance of clinical signs at higher doses. There was no evidence of carcinogenicity.

In a multigeneration reproduction study in rats fenpropathrin was administered at dietary levels of 0, 5, 25 or 250 ppm. The NOAEL was 25 ppm, equal to 1.6 mg/kg bw/day, based on decreased pup weights in the F_{3A} generation at 250 ppm.

In a second multigeneration reproduction study conducted at dose levels of 0, 40, 120 or 360 ppm, the NOAEL was 40 ppm, equal to 3 mg/kg bw/day, based on depression of body-weight gain, increased mortality in females and the occurrence of tremors in pups at 120 ppm and above.

Two oral teratogenicity studies in rats were performed at dose levels of 0, 0.4, 2 or 10 mg/kg bw/day and 0, 0.4, 1.5, 2, 3, 6 or 10 mg/kg bw/day. The NOAELs were 2 and 3 mg/kg bw/day in the two studies, respectively, with respect to maternotoxic effects and 10 mg/kg bw/day in both for embryotoxicity and teratogenicity.

In an oral teratogenicity study in rabbits at dose levels of 0, 1.5, 3 or 6 mg/kg bw/day, the NOAEL was 6 mg/kg bw/day. In a second study with oral doses of 0, 4, 12 or 36 mg/kg bw/day the NOAEL was 4 mg/kg bw/day with respect to maternal toxicity.

Fenpropathrin has been adequately tested in a series of *in vitro* and *in vivo* genotoxicity assays. The Meeting concluded that fenpropathrin is not genotoxic.

On the basis of studies in hens and rats, fenpropathrin exhibited no potential for delayed neurotoxicity.

Data on observations in humans were not suitable for the estimation of an acceptable daily intake.

An ADI of 0-0.03 mg/kg bw was established, based upon an NOAEL of 3 mg/kg bw/day in

the multigeneration reproduction study in rats, the teratogenicity studies in rats and the one-year feeding study in dogs, using a safety factor of 100.

A toxicological monograph was prepared.

TOXICOLOGICAL EVALUATION

Levels causing no toxicological effect

Mouse: 600 ppm, equal to 56 mg/kg bw/day (104-week study)

Rat: 150 ppm, equal to 7 mg/kg bw/day (104-week study)
40 ppm, equal to 3 mg/kg bw/day (reproduction study)
3 mg/kg bw/day (maternal toxicity in teratogenicity study)

Rabbit: 4 mg/kg bw/day (maternal toxicity in teratogenicity study)

Dog: 100 ppm, equal to 3 mg/kg bw/day (one-year study)

Estimate of acceptable daily intake for humans

0-0.03 mg/kg bw

Studies which will provide information valuable in the continued evaluation of the compound

Further observations in humans.

RESIDUE AND ANALYTICAL ASPECTS

Residue data from supervised trials on apples, cotton seed, gherkins, grapes, pears and tomatoes were supplied to the Meeting. No data on cucumber were received.

The major biotransformation reactions of fenpropathrin in animals consist in oxidation at the methyl groups of the acid moiety and at the 2_ and 4_ positions of the alcohol moiety, cleavage of the ester linkage and conjugation of the resultant carboxylic acids and alcohols with glucuronic acid, sulphuric acid and glycine.

Studies in plants with radio-labelled fenpropathrin demonstrate that in fruit fenpropathrin itself is the primary component of the residues, whereas in leaves degradation products constitute the greater part of the residues. The major metabolic reaction of fenpropathrin in plants has been found to be the rupture of the ester linkage followed by oxidation to produce 3-phenoxybenzoic acid (PB acid) and the corresponding alcohol and aldehyde. From the acid side of the molecule, the main metabolite is 2,2,3,3-tetra-methylcyclopropanecarboxylic acid (TMPA) and this compound can give rise to 2-hydroxymethyl-2,3,3-trimethyl-cyclopropanecarboxylic acid (TMPA-CH_2OH) and 5-hydroxymethyl-6,6-dimethyl 3-oxabicyclo-[3.1.0]hexan-2-one (TMPA-CH_2OH lactone) by subsequent hydroxylation. Also PB acid can be hydroxylated at the 4′ position and the parent molecule at the 2′ or 4′ position. The hydroxylated derivatives exist in both free and conjugated forms. Breakdown products in plants did not differ greatly from those in animals. The residues of the main metabolites PB acid and TMPA in samples from supervised field trials constituted only a negligible proportion of the total residues. It is therefore considered appropriate to define the residue in crops as the parent compound.

The fate of fenpropathrin in the soil will be influenced by a combination of photo degradation and microbiological processes. It is unlikely that fenpropathrin will remain in the soil long

enough for residues to survive and affect succeeding crops. Metabolites do not accumulate in soil. Fenpropathrin is strongly adsorbed by soils, and when used as recommended will not contaminate ground water. Examination of plants grown on treated soils showed only extremely small uptake of radioactivity.

The residue data from supervised trials were evaluated as follows.

Apple. Results of 19 US trials with a maximum application rate of 0.45 kg ai/ha, a 14-day PHI and a maximum of 8 applications showed that the residues were below 5 mg/kg in whole fruit (minimum 0.06 mg/kg, maximum 4.5 mg/kg, estimated maximum residue level 5 mg/kg).

Pear. The maximum level observed in pears treated according to anticipated approved uses was 3.2 mg/kg in whole fruit in the State of Washington, USA, where the spray concentration was 0.024%, the application rate 0.9 kg ai/ha, the PHI 14 days and the crops were subjected to a total of 6 applications. In 15 supervised US trials within GAP based on 0.45 kg ai/ha, 0.012%, a 14-day PHI and 8 applications all residues were below 5 mg/kg (minimum 0.58 mg/kg, maximum 2.9 mg/kg; estimated maximum residue level 5 mg/kg).

Grapes. The maximum GAP was in US trials. There were 4 trials within GAP (0.45 kg ai/ha, a 21-day PHI and 4 applications; minimum 0.84 mg/kg, maximum 2.6 mg/kg; estimated maximum residue level 5 mg/kg) and 18 trials using 0.22 kg ai/ha, with a PHI of 21 days and also 4 applications. It is considered that residue levels from applications based on accepted use recommendations would normally fall below 5 mg/kg.

Gherkin. Residues in samples from 4 supervised German trials using an application rate of 0.08 kg ai/ha , a 3-day PHI and 3 applications did not exceed 0.1 mg/kg (minimum <0.01 mg/kg, maximum 0.1 mg/kg; estimated maximum residue level 0.2 mg/kg).

Peppers, Sweet. Residues from outdoors supervised trials based on 3 applications of 0.01% and a 0-1-day PHI in Japan and Spain ranged from 0.2 mg/kg to 1.2 mg/kg (estimated maximum residue level 1 mg/kg). Spanish residues (indoors, 7-day PHI) ranged from 0.04 to 0.38 mg/kg and for a 2-day PHI from 0.34 to 0.52 mg/kg.

Tomato. The highest levels were seen in four Japanese studies, because GAP in Japan allows an application rate of 0.25 kg ai/ha and a one-day PHI. One figure exceeded 1 mg/kg. Residue results of 5 outdoor and 8 indoor supervised trials in Germany with a lower application rate of 0.08 kg ai/ha show that 3 days after the last application residues were all below 0.6 mg/kg. (Outdoors: minimum <0.01 mg/kg, maximum 0.37 mg/kg. Indoors: minimum <0.01 mg/kg, maximum 0.46 mg/kg; estimated maximum residue level 1 mg/kg).

Egg plant. Residues from 4 Japanese trials based on 3 - 5 applications of 0.01% and a 1-day PHI were low (minimum 0.12 mg/kg, maximum 0.19 mg/kg; estimated maximum residue level 0.2 mg/kg).

Cotton seed. A well-known factor that can influence the level of residues in cotton seed is whether an appreciable number of bolls have opened at the time of the last application. If not, residues in the seed are usually very low but if there is direct contact between the insecticide spray and the seed, residues can reach measurable levels. In considering the MRL needed it is important that it should be high enough to include cases where the last application was to plants with a comparatively high proportion of open bolls. It was possible to use 26 trials with an application rate of 0.22 kg ai/ha, 8 - 11 applications and a PHI of 18 - 22 days (minimum residues <0.01 mg/kg, maximum 1 mg/kg; estimated maximum residue level 1 mg/kg).

Residues in food of animal origin

Cattle. Residues in whole milk when a plateau level had been reached were approximately

0.15% of the level in the feed. If cows were fed on a diet consisting entirely of dried apple pomace at the postulated maximum residue level of 45 mg/kg (see processing of apples, below), it could be argued that the maximum level in milk would be 0.07 mg/l. Assuming that these residues would all be present in the fat and that the fat content of the milk would be 4%, such a level would be equivalent to 1.8 mg/kg in the milk fat. An animal transfer study showed levels in body fat to be approximately 1.4% of the level in the feed. Using the apple pomace figure of 45 mg/kg, it is reasonable to conclude that residues in meat fat would not exceed 0.6 mg/kg. Based on similar arguments and the data from the same studies, residues in meat (muscle) were about 0.08% of the feed level so that animals fed on apple pomace at 45 mg/kg would not be expected to have more than 0.05 mg/kg in muscle, kidney or liver.

Poultry. Poultry are unlikely to receive dietary items containing appreciable residues of fenpropathrin with the possible exception of cotton seed meal. With a maximum level of 1 mg/kg in raw cotton seed, it is unlikely that residues in meal would exceed 0.1 mg/kg. With a total feed level of 2.5 mg/kg, the level in fat reached only 0.02 mg/kg so that measurable residues would not be expected in the eggs, meat or edible offal of poultry fed on cotton seed meal.

In processing

In fruits the residues are essentially surface residues. As would be expected juice extraction leaves the great majority of the residues in the solids. In the case of dry apple pomace the data suggest a maximum concentration factor of 9, so that residues in dry apple pomace would not be expected to exceed 45 mg/kg on the basis of a maximum residue level in whole apples of 5 mg/kg.

As would be expected, raisins have higher residues than the raw grapes. The highest concentration factor in the trials is about 3. Using this factor and assuming that residues in raw grapes will not exceed 5 mg/kg, it would seem reasonable to estimate that residues in raisins would not exceed 15 mg/kg.

Dry grape pomace contained between 2 and 7 times the residue level in the original grapes. If the highest level in raw grapes is 5 mg/kg, the highest level to be expected in dry grape pomace would be 35 mg/kg.

Processing grape juice into wine appears to reduce residue levels still further and although strictly comparable data are only rarely available, residues of fenpropathrin have not been found above the limit of determination in wine, whereas in juice the highest level found was 0.06 mg/kg, which disappeared during vinification. In this particular case residues in the raw grapes were up to 5.6 mg/kg, so that even at this high level measurable residues did not survive in the wine.

As would be expected from the lipophilic nature of fenpropathrin, residues in oil obtained from cotton seed are higher than in the raw seed by roughly the inverse proportion of oil weight to seed weight. The residues in the meal ranged from 0.01 to 0.09 mg/kg. Residues in soapstock were about twice the level in the raw seed and residues in the refined oil were in the region of three times the seed level. Assuming a maximum level in raw cotton seed of 1 mg/kg, it can reasonably be concluded that residues in soapstock will not exceed 2 mg/kg and in oil 3 mg/kg.

Stability of stored analytical samples

In stability studies carried out on apples, pears, grapes, oranges, cotton seed, eggs and kidney of cattle over periods from 3-12 months there was no evidence of a decline in residue levels of fenpropathrin during storage at -20^{o}C.

Methods of residue analysis

Methods of analysis used GLC with an EC detector after solvent extraction of the substrate and clean-up by either silica gel or Florisil column chromatography. The limit of determination in most crop samples is between 0.005 and 0.01 mg/kg.

4.22 FENTIN (040)

RESIDUE AND ANALYTICAL ASPECTS

Fentin was last evaluated in 1991. The proposed limit of 1 mg/kg for dry hops was discussed at the 1993 CCPR (ALINORM 93/24A) and the delegation of France promised to send written comments which were considered by the Meeting.

The Meeting's attention was drawn to some printing errors in Table 7 (1991 Evaluations p.346) where the residue in dry hops was shown as <1.01 mg/kg instead of <0.01 mg/kg for reports A23619 and A23616, and <1.0 mg/kg instead of 10 mg/kg for the two trials from 1989 (Report No. A44068).

As the compound is applied before flowering according to GAP no residue would be expected in green hops at harvest. The Meeting therefore considered that the levels of 10 mg/kg of report A44068 were inconsistent with the other data. On the basis of the corrected residues in dry hops the Meeting concluded that a level of 0.5 mg/kg should not be exceeded when the compound is used in accordance with GAP.

4.23 FLUCYTHRINATE (152)

RESIDUE AND ANALYTICAL ASPECTS

Flucythrinate was reviewed for residues by the 1985, 1987, 1988, 1989 and 1990 Meetings. The 1985 JMPR listed additional data for a number of crops and information regarding likely residues in animal products as desirable. MRLs for these commodities have been retained at step 7, as have limits for maize forage and fodder. The 1990 JMPR required full documentation of data which were submitted only in summary form for citrus, cucumber, green beans and peppers. That Meeting did not evaluate summary information submitted on residues in animal products in the absence of the full reports. The present Meeting reviewed submissions made in response to the 1990 requirements, additional data for crops with and without current or proposed MRLs, and a re-submission of summary information on animal residues.

Green beans. Submission, as requested, of detailed reports on green bean trials provided only in summary to the 1990 JMPR reveals that the data are the same as the Egyptian data reviewed by the 1985 JMPR. The two values of 0.14 and 0.22 mg/kg after 3 days are consistent with reported Spanish GAP, but the Meeting concluded (as apparently did the 1985 JMPR) that the data were insufficient to support an MRL for green beans.

Brassica vegetables. There are no outstanding issues concerning the CXLs of 0.5 mg/kg for head cabbages and 0.2 mg/kg for flowerhead brassicas (broccoli, cauliflower). The Meeting received data on broccoli and Brussels sprouts which were reviewed by the 1985 JMPR and which did not need further review. New data were also received for red, white and Savoy cabbages. With maximum residues of 0.37 mg/kg from applications within Spanish GAP, no change in the current limits was required.

Citrus. Data reviewed by the 1985 JMPR from trials in Egypt (not GAP) and Japan were re-

submitted, together with additional detail and GAP information for Japan and Spain. While the data suggest that residues are unlikely to exceed 2 mg/kg from GAP, the Meeting concluded (as implicitly did the 1985 JMPR) that data reflecting GAP in additional countries are needed to support an MRL for a major crop such as citrus. Processing studies would also be needed.

Cotton seed. Summary data reported from supervised trials in the United States and apparently not previously reviewed by the JMPR indicate residues well below the 0.1 mg/kg CXL. The Meeting concluded that there was no need to request the complete studies nor to revise previous estimates.

Cucumber. Submission of detailed reports as requested by the 1990 Meeting reveals that the data are the same as the Egyptian data reviewed by the 1985 JMPR. While the detailed reports indicate that the maximum residues of <0.05 mg/kg are consistent with reported Spanish GAP (3-day PHI) for cucurbits, the manufacturer could not confirm that the use is GAP on cucumbers anywhere. The Meeting concluded (as apparently did the 1985 JMPR) that the data were too limited to support an MRL.

Peppers. Submission, as requested by the 1990 JMPR, of detailed reports indicate that the summary data provided to that Meeting are the same as the Italian data reviewed by the 1985 JMPR. While the detailed reports indicate that the 0.14 and 0.13 mg/kg values after 4 days should be within Spanish GAP, the Meeting concluded (as apparently did the 1985 JMPR) that the data were too limited for estimating MRLs.

Tomatoes. There were no questions on the 0.2 mg/kg CXL recommended by the 1985 JMPR. The re-submitted studies had been previously reviewed.

Cattle meat and milk; goat meat. The current 0.5 mg/kg limits for the meat of cattle and goats and the 0.1 mg/kg limit for cattle milk have been retained at Step 7 by the CCPR pending submission of adequate animal feeding studies representative of feeding levels likely to occur in practice. Government comments provided to the Meeting proposed deletion of these limits with the view that available information was based on feeding levels irrelevant to actual animal intakes. The manufacturers again submitted discussion points and summary information previously provided to the 1990 JMPR, which required submission of the detailed reports from which the summary information was taken. This information was not available to the Meeting.

The Meeting considered likely levels in commodities which could be used as animal feed items and agreed that the maximum dietary intake for cattle was unlikely to exceed 5 mg/kg, and in practice would probably be lower. Assuming this level, using the available feeding data summary, and assuming that residues in the animal products vary linearly with their levels in the feed from the 13 to 100 ppm levels fed experimentally down to the postulated 5 ppm, maximum residues of the order of 0.08 mg/kg in the fat of meat of cattle and goats and 0.1 mg/kg in milk could be estimated. This would suggest that the previously estimated levels of 0.5 mg/kg in the fat of meat and 0.1 mg/kg in milk would be adequate.

The summary information and comments provided to the Meeting provide greater insight into likely residues in animal products. However in the absence of the detailed reports from which the transfer data were summarized, and in view of the fact that the fat-solubility of flucythrinate leaves the potential for residues in animal tissues and milk, the Meeting recommended that the temporary limits for the meat and milk of cattle and goat meat should be withdrawn.

4.24 FLUSILAZOLE (165)

RESIDUE AND ANALYTICAL ASPECTS

Flusilazole was previously reviewed for residues by the 1989, 1990 and 1991 Meetings. The present Meeting reviewed information provided in response to the 1991 JMPR requirement for additional GAP and residue data to confirm the 0.1 mg/kg temporary estimate for peaches and nectarines, and information listed as desirable on grapes, details of wheat grain freezer storage studies, stability of metabolites in freezer-stored grain samples, hen metabolism, metabolites in grain processed fractions and soil studies. Additional residue data on pome fruit, grapes and cereals (although there were no outstanding residue data requirements on these commodities) and new data on sugar cane were also provided.

Fate of residues in animals. Several reports on hen metabolism were provided. Some had been submitted before and some, including the requested study by Smyser, were new. Only the Smyser report included data in need of review by the Meeting.

The report basically combines and summarizes information in two previously reviewed reports and provides further clarification of the residues of metabolites, especially in terms of the percentage of the total radioactivity in poultry tissues and eggs, for both the phenyl and triazole labels. It confirms previous JMPR conclusions that bis(4-fluorophenyl)(methyl)silanol (IN-F7321, the methyl silanol) and 4-fluorophenyl(methyl)silanediol are the predominant residues in poultry tissues and eggs arising from the phenyllabelled compound and that triazole is the main residue from the triazole label, except in fat where flusilazole is the primary residue from the triazole label.

The report does not effectively answer questions raised by the 1989 and 1991 Meetings concerning differences in residues found between ruminant and poultry metabolism and feeding studies. The Meeting noted and agreed with the 1991 JMPR conclusion that these differences probably result largely from the more detailed residue characterization and identification in the poultry studies than in the ruminant studies. The Meeting also agreed with the 1991 JMPR that although all questions have not been completely answered, the nature of the residue in animal products can be considered to be reasonably well understood in view of the low residues expected (especially for flusilazole) in animal products.

Soil dissipation. The Meeting reviewed the final report of a 3-year soil dissipation study (4 applications per year) for which an interim report was reviewed by the 1989 JMPR. It confirms the 1989 observations that over 92% of the radioactivity is confined to the top 8 cm of soil over the test period, and that the predominant residues in this segment are flusilazole and its silanol metabolite IN-F7321. The author cites statistical evaluation of the data to support the view that residues will reach a steady level at 57% of yearly application levels after repeated application levels under worst-case conditions.

The report cites the steady-state conclusion, the strong adsorption to the top layers of soil, the lack of residues exceeding 0.01 mg/kg in the 24-36 cm soil depths and the weak leaching potential indicated in other studies as evidence that residues in ground water were unlikely. While the data indicate that over 92% of the radioactivity remains in the top 8 cm of the silt loam soil investigated, and indeed that residue levels are extremely low in the 24-36 cm depths, it also shows an increasing penetration by low levels of radioactivity over the test period in this soil type. The identity of these residues in the deeper soil segments was not indicated.

While the adsorption of this persistent pesticide to soil is strong, the 1989 JMPR had noted that uptake of low residue levels can occur in rotational crops and that the leaching potential would be less for silt loams (as in this study) than for more sandy soils. Because the silt loam study was under worst-case conditions (bare ground, repeated applications) and was consistent with reassuring findings of a number of other relevant studies, the Meeting accepted that ground water residues from silt loam soils were unlikely.

Freezer storage stability. Instead of details of a previous 36.5-month study for the parent

compound only, the Meeting was provided with a new 11-month freezer storage study of flusilazole and its metabolites in wheat grain and straw. While the results suggest that about 30% of 0.3 mg/kg residues of the parent compound and its phenyl metabolites in grain and straw are lost after various storage intervals up to 11 months, the variability in the recoveries of freshly fortified samples indicates that the apparent losses are probably as much the result of analytical variability as actual storage losses. The Meeting concluded that the data demonstrated adequate stability of flusilazole and the metabolites IN-7321, 1,1,3,3-tetrakis(4-fluorophenyl)-1,3-dimethyldisiloxane (IN-G7072), 2-fluoro-5-[(4-fluorophenyl)(methyl)(1-*H*-1,2,4-triazol-1-ylmethyl)silyl]phenol (IN-37722) and 2-fluoro-5-[(4-fluorophenyl)(hydroxy)(methyl)silyl]phenol (IN-37738) (presumably unconjugated) over 11 months under the conditions of the study.

The 11-month storage interval compares with sampling-to-laboratory-receipt intervals ranging from 2 to 15 months in cereal grain trials from which data were reviewed by the 1989 JMPR. The Meeting did not know the actual sampling-to-analysis intervals for the data reviewed in 1989, although according to the 1989 monograph all samples were generally stored at -20°C.

Cereals. The original 1989 JMPR estimates of maximum residue levels of 0.1 and 2 mg/kg respectively for cereal grains and straws or fodders (dry) were based on maximum residues of 0.07 mg/kg in grain and 1.7 mg/kg in straw. Although there were no outstanding requirements for additional supervised trials data, the Meeting received extensive additional cereal grain, plant, forage and straw data from Europe and North America. Because no need for MRL revisions was indicated, the Meeting only briefly summarized the submitted data on grain and straw. It concluded that there was no need to revise the recently adopted limits of 0.1 mg/kg in the grains and 2 mg/kg in the straws and fodders (dry) of barley, rye and wheat at present. This conclusion may need to be reconsidered at a future Meeting in the light of future GAP information.

Cereal grain processing. The 1991 JMPR reviewed a wheat processing study submitted in response to a 1989 requirement. While no concentration in milled fractions was observed, samples were not analysed for metabolites (especially IN-F7321) and such analysis had been recorded as desirable. A barley grain processing study provided to the Meeting confirmed that no concentration of flusilazole or the major metabolite IN-F7321 occurred in milling fractions.

Grapes. Limited additional information on GAP in Europe and Australia and additional grape data submitted in response to the 1991 requests showed maximum residues reflecting GAP of 0.22 mg/kg compared to the recently adopted CXL of 0.5 mg/kg. A delegation to the CCPR had suggested that a 0.2 mg/kg limit was sufficient. The Meeting confirmed the 1989 JMPR conclusion that residues were unlikely to exceed 0.3 mg/kg.

Pome fruit. Additional GAP information and residue data did not require a revision of the current 0.2 mg/kg limit.

Stone fruit. The 0.1 mg/kg limit for peaches and nectarines recommended by the 1991 JMPR was temporary pending the submission of additional GAP and residue data. It had been based on data from New Zealand and France and GAP from New Zealand and Spain. The Meeting received information on current GAP from Spain, France, Greece (pending) and Italy, and residue data on nectarines from France and on peaches from Australia, Italy, Greece, and the United States. French apricot data were also provided as supporting information. No GAP information was available for Australia or the United States. One to 4 applications at 3-4 g ai/hl and a PHI of 7 to 10 days appears to be usual for countries with established GAP, although in two cases the maximum number of permitted applications was not indicated.

At a 7-day PHI, the new French data or those summarized by the 1991 JMPR which reflect GAP rates showed maximum residues of flusilazole *per se* in peaches of 0.09 mg/kg (1991) or

0.08 mg/kg (1993), except in one trial in the 1993 submission where a residue of 0.55 mg/kg after 8 days was reported from 9 applications at GAP rates. Maximum apricot residues reflecting GAP rates were 0.08 mg/kg after 7 days. Maximum residues in the US trials were 0.09 mg/kg at a 2.4 g ai/hl spray concentration after 7 or 14 days (0.2 mg/kg after 5 days) and 0.3 mg/kg at a 4.8 g ai/hl rate after 12 days. At a pending GAP rate, maximum residues after 7 days in the Greek trials were 0.09 mg/kg. Residues were not detected in the Australian or Italian trials (<0.05 mg/kg and <0.01 mg/kg respectively), but that is not unexpected in view of the long PHIs and the type of application. The Meeting concluded that a 0.5 mg/kg limit was supported for peaches. Observing that GAP for apricots and nectarines is similar to that for peaches, the Meeting concluded that the available data could also mutually support 0.5 mg/kg limits for apricots and nectarines at a 7-day PHI.

Limited data for plums and cherries were insufficient to recommend MRLs.

Sugar cane. No residues (<0.02 mg/kg) were detected in the juice from plants grown after dip treatments of sugar cane sets at fivefold application rates. No stalks were analysed. The Meeting concluded that the data were inadequate to support a limit for sugar cane.

FURTHER WORK OR INFORMATION

Desirable

1. Submission of analytical method AMR-115-85 cited in Du Pont, 1993, Vol. 1, exhibit 6. Submission of validation information to permit estimation of limits of determination is desirable.

2. On completion, submission of the soil dissipation report AMR-791-87 (Fujinari, 1988). The interim report was reviewed by the 1989 JMPR.

4.25 FOLPET (041)

TOXICOLOGY

Folpet was evaluated for acceptable daily intake by the Joint Meeting in 1969, 1973, 1982, 1984, 1986 and 1990. A temporary ADI of 0-0.01 mg/kg bw was established in 1986, which was extended in 1990 pending submission of the following studies for review in 1993:

- Results of further investigation of the relevance of metabolic data in animals for humans.

- Further studies to elucidate the mechanism for the induction of gastrointestinal tract tumours in mice.

- Studies designed to establish an NOAEL in mice.

The data that were reviewed at the present Meeting included a DNA-binding study in mice with the analogue, captan; an acute inhalation toxicity study in rats; a delayed cutaneous hypersensitivity study in guinea-pigs; and carcinogenicity studies in both $B_6C_3F_1$ mice (study designed to focus on the mechanism of tumour induction) and CD-1 mice (interim report: 0-42/60 weeks).

These data did not satisfy the above request but since relevant studies are known to be in progress, the Meeting extended the temporary ADI of 0-0.01 mg/kg bw to 1995, with submission of data to WHO by 1994.

A toxicological monograph was not prepared.

RESIDUE AND ANALYTICAL ASPECTS

Folpet was evaluated first in 1969, and several times since. The 1987 JMPR recommended that a detailed review of all aspects of the use of folpet be carried out at the 1989 Meeting or as soon as possible. At the 23rd (1991) Session of the CCPR it was decided (ALINORM 91/24A, para 95) to maintain CXLs for apple, cherries, cucumbers, grapes, bulb onions and strawberries, regarding them as temporary until 1992 when the results of current and planned supervised trials could be reviewed.

The 25th (1993) Session of the CCPR was informed that the manufacturer had provided information for all commodities with temporary MRLs except cherries and onions (ALINORM 93/24A, para 66).

The Meeting received information on registered uses of folpet and data from supervised trials on fruit and vegetables. MRLs for bulb onions and cherries will not be supported by new supervised field trials. Residue data from supervised trials on the following crops were reviewed:

mandarins (*Spain*), oranges (*Israel, Spain*), apples (*Chile, France, Israel, Portugal*), grapes (*Argentina, Chile, France, Israel, Italy, Spain*), strawberries (*Brazil, Hungary, Israel, Spain, Uruguay*).

melons (*France, Israel*), squash (*Greece*), lettuce (*Brazil, France, Israel*), potatoes (*Denmark, Israel, Netherlands, UK, Uruguay*), tomatoes (*France, Hungary, Israel*).

The Meeting was informed that the proposed GAP for folpet in Israel would probably become official in the near future. Only current official GAP is used in the evaluation of residue data.

There are no current registered uses for folpet on citrus, so the Meeting could not estimate a maximum residue level for citrus fruits. If the proposed use in Israel becomes registered, supervised trials data from Israel and Spain would suggest an MRL of 2 mg/kg.

The registered use of folpet on apples in Portugal requires a spray concentration of 0.13 kg ai/hl and a PHI of 21 days. Trials in Portugal and Israel conformed with this use pattern; the highest residues of folpet were 1.4 and 1.8 mg/kg. The Meeting was also aware of supervised trials on apples in France currently awaiting a final report. Because of the limited number of trials currently available within GAP the Meeting recommended withdrawal of the temporary MRL for apples.

Folpet is registered for use on grapes in Argentina, France, Italy and Spain. Supervised trials data were available from these countries. Trials were also available from Israel and were evaluated against GAP for grapes in Portugal and Spain. Residues arising from use according to GAP commonly fall in the 0.5-1 mg/kg range but residues of 1.3 and 2.0 mg/kg were recorded in a Spanish trial. The Meeting estimated a maximum residue level of 2 mg/kg for folpet in grapes.

Residue trial data on strawberries were provided from Brazil, Hungary, Israel, Spain and Uruguay, but there was no GAP for Hungary, Israel or Uruguay. Most of the residues in the Brazilian trials within GAP were in the 1-2 mg/kg range. The highest folpet residue in the Spanish trial within GAP was 1.1 mg/kg.

A folpet trial on strawberries in Israel (where registration is proposed) was evaluated against Portuguese GAP. Residues were consistently in the 2-5 mg/kg range, and were quite persistent. The highest residues were 4.7 and 4.8 mg/kg. The Hungarian trial was evaluated against Netherlands GAP; the highest folpet residue was 0.78 mg/kg. The Meeting estimated a maximum residue level of 5 mg/kg for folpet in strawberries.

No folpet was detected (<0.01 mg/kg) in melons from a French trial where an exaggerated spray concentration, approximately threefold, had been used. The laboratory had reported some problems with folpet recoveries when the sample was spiked before chopping. The fact that no residues were detected on samples taken the same day as the final application also throws doubt on the validity of the trial results.

No residues were detected (<0.02 mg/kg) in the pulp of melons in the Spanish trial. Data are required on a whole fruit-basis for MRL purposes. Trial data from the melon trials in Israel could not be evaluated because there is, as yet, no registered use in Israel.

The Meeting was unable to estimate a maximum residue level for folpet residues in winter squash because the data were too limited. The Meeting was informed that cucumber trial data would become available in the future from Turkey, Israel and Cyprus.

Folpet residues in lettuce treated according to Brazilian GAP ranged up to 1.4 mg/kg. Trial data on lettuce from Israel could not be evaluated because there is no registered use of folpet on lettuce in Israel, although it is proposed. The Meeting was also aware of supervised trials on lettuce in France currently awaiting a final report. Because of the limited number of trials available at present within GAP the Meeting recommended withdrawal of the temporary MRL for lettuce.

Supervised trials data for folpet on potatoes were available from Denmark, Israel, The Netherlands, the UK and Uruguay. The only country in this list which has registered uses for folpet on potatoes is Uruguay. Folpet residues in the trials from these countries were mostly not detectable (<0.01, <0.02 mg/kg). The pattern of residues expected for potatoes from the foliar use of a non-systemic pesticide is the occasional detection where a tuber has been directly exposed, but with no residues in most tubers. This pattern would not be much affected by the rate of application. The highest residue detected was 0.49 mg/kg in one plot in the Uruguay trial.

The Meeting noted that residues were generally undetectable in potatoes from application rates of 1.3 to 4.0 kg ai/ha in a number of different countries. The Meeting estimated a maximum residue level of 0.02* mg/kg for folpet in potatoes.

The maximum application rate for folpet on tomatoes in France is 1 kg ai/ha, but the rates used in the supervised trials were 1.5 and 3.0 kg ai/ha, so the data could not be used to estimate maximum residue levels. Folpet is not registered for use on tomatoes in Israel (although there is a proposed registration) so the data from trials in Israel could not be used. Folpet residues in tomatoes in Hungary treated according to GAP were not detectable (<0.02 mg/kg) 14 days after the final application. The Meeting considered the data were insufficient to estimate a maximum residue level for tomatoes.

Phthalimide residue data were also provided for most of the supervised trials. Phthalimide is the major primary metabolite of folpet. In many cases phthalimide residues were not detected in the trials, but in some cases they were of the same order as those of folpet, or even exceeded them. Phthalimide levels were generally not well related to the use of folpet and should not be included in the residue definition as an indicator of compliance with GAP.

The stability of folpet and phthalimide residues in stored analytical samples (lettuce, potato,

tomato, melon), separately fortified with each compound at 1 mg/kg, was tested at -18°C. About 10-20% of the residues were lost during 6 months freezer storage.

In the analytical methods used for many of the trials, samples were chopped and extracted with ethyl acetate, then cleaned up on a Florisil column for folpet, or by solvent partition (hexane, phosphate buffer) for phthalimide. Gas-liquid chromatography on a megabore column with a ^{63}Ni electron-capture detector for folpet and with a thermionic nitrogen-specific detector for phthalimide was used for the final determination. No interference was caused by 25 common pesticides which might occur in crop samples. The limits of determination were 0.05 mg/kg for folpet and 0.2 mg/kg for phthalimide. Limits of detection were lower by factors of 2-2.5.

The Meeting received information on national MRLs for folpet from Canada, the EEC, Hungary, The Netherlands and the USA.

FURTHER WORK OR INFORMATION

Desirable

1. Full details and results of the French trials on apples and lettuce now awaiting final reports, together with full details of the relevant French GAP.

4.26 HEPTACHLOR (043)

RESIDUE AND ANALYSIS ASPECTS

Information on use patterns of heptachlor was supplied to the 1991 JMPR, but only in a summarized form and no registered or recommended uses on vegetables were available. Residue data from monitoring heptachlor and heptachlor epoxide in fruits and vegetables and animal products were also reported. The 1991 Meeting recommended that the existing Extraneous Residue Limits for heptachlor in carrots, tomatoes and other vegetables should be converted to temporary limits until more information was available on the possible occurrence of residues in food in commerce or at consumption.

Monitoring data were received by the present Meeting from The Netherlands, Sweden and the USA.

Residues in fruit and vegetables occurred only to a very limited extent. In 15,300 samples of fruit, vegetables and cereal grains examined in The Netherlands residues were present in only 13 samples. In Sweden residues occurred in only 3 of about 9000 samples analysed, including many samples of carrots and tomatoes. In the USA residues of heptachlor were present in 18 of 14,800 samples of fruit and vegetables and 5 of 573 samples of cereal grains. The monitoring in the USA as well as in Sweden included the analysis of carrots and tomatoes. No residues were present in any of about 700 samples of carrots and 2200 samples of tomatoes analysed. The limit of determination in the three countries was 0.01-0.02 mg/kg.

In animals heptachlor is metabolised to heptachlor epoxide. This compound was not present in the animal products examined, with a few exceptions. No residues were detected in about 800 samples of domestic animal products examined in The Netherlands. In the USA no residues were found in milk, eggs or imported meat. Residues occurred at a level of 0.03-0.13 mg/kg in only three samples of domestic pigs and at 0.04 and 0.06 mg/kg in two samples of geese.

With this very low incidence of heptachlor in carrots, sugar beets, tomatoes and other

vegetables the Meeting was of the opinion that there is no further need for ERLs for heptachlor in vegetables.

4.27 HEXACONAZOLE (170)

RESIDUE AND ANALYTICAL ASPECTS

The 1990 JMPR recommended that MRLs for wheat and wheat straw and fodder be temporary pending receipt of data on residues in animal products resulting from feeding grain and/or straw, together with a method of analysis for hexaconazole in products of animal origin suitable for regulatory purposes, by 1993. Data on the fate of residues during the processing of grain were also required.

The Panel considered these requirements in view of the guidance for animal transfer studies discussed elsewhere in this report (Section 2.7). On the basis of the goat metabolism study with radio-labelled hexaconazole (1990 JMPR) and estimates of dietary intake, no detectable residues of hexaconazole would be expected in animal tissues or milk; total radioactive residues would be expected to be low (of the order of 0.01 mg/kg). It was agreed that in view of this a livestock feeding study and an analytical method for animal tissues would no longer be required. It was noted that while there may be some concentration in wheat processed fractions, in view of the low residues in the wheat grain and the character of the active ingredient, this information was no longer required but was desirable.

The Meeting recommended MRLs for wheat (0.1 mg/kg) and wheat straw and fodder (0.5 mg/kg) to replace previous temporary limits.

FURTHER WORK OR INFORMATION

Desirable

Data on the fate of residues during the processing of grain.

4.28 MANCOZEB (050)

TOXICOLOGY

Mancozeb was evaluated by the Joint Meeting in 1967, 1970, 1974, 1977 and 1980. An ADI of 0-0.05 mg/kg bw was established at the 1980 Meeting for mancozeb or the sum of maneb, mancozeb and zineb, of which not more than 0.002 mg/kg bw may be present as ethylenethiourea (ETU).

In pharmacokinetic studies conducted in male and female mice, orally administered ^{14}C-labelled mancozeb was rapidly absorbed, peaking in whole blood between 1 and 2 hours, extensively metabolized, and rapidly excreted (90%) within 24 hours. ETU was the major metabolite.

Rats given single oral doses of ^{14}C-labelled mancozeb absorbed about 50% of the dose within 3-6 hours. Most of the dose was excreted in 24 hours with half eliminated in the urine and half in the faeces. Less than 4% was found in the tissues, with the thyroid containing the highest residue level. Most of the ^{14}C dose in the faeces was unabsorbed, since only 2-8% of the dose was found in bile. ETU was the major metabolite. The half-life of ETU elimination was 4-5 hours. The estimated bioavailability of ETU in rats was about 6.8% on a weight/weight

basis and 20% on a mole/mole basis.

The acute oral, dermal and inhalation toxicity of mancozeb technical is low. WHO has classified mancozeb as unlikely to present acute hazard in normal use.

In a 13-week study in rats, mancozeb was administered in dietary concentrations of 0, 30, 60, 125, 250 or 1000 ppm. The NOAEL was 125 ppm (equal to 7.4 mg/kg bw/day), based on increased serum TSH and decreased T_4 values at the next higher dose.

Dogs administered 0, 10, 100, 1000 or 5000 ppm of mancozeb in the diet for three months demonstrated an NOAEL of 100 ppm, equal to 3.0 mg/kg bw/day. At the next higher dose, decreased body-weight gains and decreased erythrocyte count, packed cell volume and haemoglobin were observed.

In a 52-week study in dogs, mancozeb was administered in the diet at concentrations of 0, 50, 200, 800 or 1600 ppm. The NOAEL was 200 ppm, equal to 7.0 mg/kg bw/day, based on decreases in body-weight gain, increased cholesterol and decreased haemoglobin and packed cell volume at 800 ppm.

The NOAEL for dogs given mancozeb technical for 52 weeks, 7 days a week, by gelatin capsule was 2.3 mg/kg bw/day, based on decreased body weight, food consumption and thyroxine levels at 23 mg/kg bw/day.

In a 78-week carcinogenicity study in mice at dietary concentrations of 0, 25, 100 or 1000 ppm, there was no evidence of carcinogenicity. The NOAEL was 100 ppm, equal to 17 mg/kg bw/day, based on decreased body-weight gain at 1000 ppm.

In a second 78-week carcinogenicity study in mice at dietary concentrations of 0, 30, 100 or 1000 ppm in the diet, there was no evidence of carcinogenicity. The NOAEL was 100 ppm, equal to 13 mg/kg bw/day, based on decreased body weight and decreased T_3 and T_4 values at 1000 ppm.

The overall NOAEL in the two 78-week studies in mice was 17 mg/kg bw/day.

In a two-year toxicity/carcinogenicity feeding study in rats at dietary concentrations of 0, 20, 60, 125 or 750 ppm, the NOAEL was 125 ppm (equal to 4.8 mg/kg bw/day), based on decreased body-weight gain, decreased T_3 and T_4 values, increased TSH values, increased absolute and relative thyroid weight, thyroid follicular cell hypertrophy, hyperplasia, and nodular hyperplasia at 750 ppm. Tumorigenic effects were noted in both sexes in the form of thyroid follicular cell adenomas and/or carcinomas at the highest dose level.

Mancozeb technical when administered in the diet to rats for two years at dose levels of 0, 28, 113 or 454 ppm was not tumorigenic. The NOAEL was 113 ppm (equal to 4.0 mg/kg bw/day), based on decreased body-weight gain, decreased thyroxine levels, an increase in the height of the thyroid follicular epithelium and an increase in prominent microfollicles at 450 ppm.

The overall NOAEL in the two 2-year studies in rats was 4.8 mg/kg bw/day.

In a two-generation reproduction study in rats at dietary concentrations of 0, 25, 150 or 1100 ppm, the NOAEL was 25 ppm (equal to 1.7 mg/kg bw/day), based on decreased body weight at 150 ppm.

In a second two-generation reproduction study in rats at dietary concentrations of 0, 30, 120 or 1200 ppm, the NOAEL was 120 ppm, equal to 7.0 mg/kg bw/day, based on microscopic changes in the thyroid, kidney and pituitary, increased relative weights of the liver, kidney and thyroid, increased absolute thyroid weight, decreased body weight and feed consumption of

females during gestation and lactation, and decreased pre-mating body weight and feed consumption, at 1200 ppm.

The overall NOAEL in both reproduction studies was 7.0 mg/kg bw/day.

In a 90-day (neuropathology) study conducted in rats at dietary concentrations of 0, 20, 125, 750 or 5000 ppm, the NOAEL was 125 ppm, equal to 8.2 mg/kg bw/day, based on decreased food consumption and neurohistopathological changes at 750 ppm.

An oral teratogenicity study in rats at dose levels of 0, 2, 8, 32, 128 or 512 mg/kg bw/day produced no maternal effects at 32 mg/kg bw/day (NOAEL) and no teratogenic effects at 128 mg/kg bw/day (NOAEL). Maternal effects in the form of decreased body-weight gain and decreased food consumption were seen at 128 mg/kg bw/day. Teratogenic effects seen at 512 mg/kg bw/day included agnathia, cleft palate, meningoencephalocele and dilated brain ventricles.

A second oral teratogenicity study in rats at dose levels of 0, 10, 60 or 360 mg/kg bw/day showed no maternal or embryo/fetotoxic effects at 60 mg/kg bw/day (NOAEL). Maternal toxicity at 360 mg/kg bw/day was seen as "reeling gait", hind limb paralysis, and decreased body-weight gain and food consumption. Embryo/fetotoxicity at the highest dose was seen as reduction in the degree of ossification of the intraparietal bone, a marginal increase in the size of the anterior fontanelle and incomplete ossification of the thoracic vertebrae centra.

The NOEL in an oral teratogenicity study in rabbits given 0, 5, 30, 55, or 100 mg/kg bw/day was 55 mg/kg bw/day for maternal effects and greater than 100 mg/kg bw/day for embryo/fetotoxic effects. An increase in abortions, body-weight loss, and decreased food consumption were observed at 100 mg/kg bw/day.

The NOEL in an oral teratogenicity study in rabbits given 0, 10, 30 or 80 mg/kg bw/day was 30 mg/kg bw/day for maternal toxicity. The NOAEL for embryo/fetotoxic effects was greater than 80 mg/kg bw/day. Maternal toxicity at 80 mg/kg bw/day was based on an increase in aborted fetuses, decreased number of litters produced, decreased body-weight gain and food consumption, an increase in clinical signs and death.

Mancozeb has been tested in a series of *in vitro* and *in vivo* genotoxicity assays. Chromosomal aberrations were induced *in vitro*, whereas conflicting data were obtained with *in vivo* assays. There was no evidence for the induction of gene mutations or cell transformations. The Meeting concluded that the data on mancozeb were equivocal for genotoxicity. A number of available studies were not considered either because DMSO was used as a solvent in which mancozeb is very unstable or because of important omissions from the reports.

The data on mancozeb would support an ADI of 0-0.05 mg/kg bw, based on the NOAEL of 4.8 mg/kg bw/day for thyroid effects in rats, using a 100-fold safety factor. However, the Meeting established a group ADI of 0-0.03 mg/kg bw for mancozeb, alone or in combination with maneb, metiram and/or zineb, because of the similarity of the chemical structures of the EBDCs, the comparable toxicological profiles of the EBDCs based on the toxic effects of ETU, and the fact that parent EBDC residues cannot be differentiated using presently-available regulatory analytical procedures (see Section 4.15 - dithiocarbamates).

A toxicological monograph was prepared, summarizing the data received since the previous evaluation and containing relevant summaries from previous monographs and monograph addenda on mancozeb.

TOXICOLOGICAL EVALUATION

Level causing no toxicological effects

Mouse: 100 ppm in the diet, equal to 17 mg/kg bw/day (78-week studies)

Rat: 125 ppm in the diet, equal to 4.8 mg/kg bw/day (two-year studies)
120 ppm in the diet, equal to 7.0 mg/kg bw/day (reproduction studies)
125 ppm in the diet, equal to 8.2 mg/kg bw/day (90-day neuropathology study)

Dog: 200 ppm in the diet, equal to 7.0 mg/kg bw/day (52-week study)

Estimate of acceptable daily intake for humans

0-0.03 mg/kg bw (group ADI with maneb, metiram, and zineb)

Studies which will provide valuable information in the continued evaluation of the compound

1. Clarification of genotoxicity potential.

2. Observations in humans.

RESIDUE AND ANALYTICAL ASPECTS

Mancozeb, evaluated in 1967 and several times since, was scheduled for review in 1993 in the CCPR periodic review programme (ALINORM 93/24A, para 71).

The Meeting received extensive information on GAP, supervised residue trials, animal transfer studies, metabolic fate in farm animals and crops, fate during processing and storage, residues in food in commerce and at consumption, and methods of residue analysis.

When lactating goats were dosed with [^{14}C]mancozeb ([^{14}C]ethylenediamine) in the feed, most of the ^{14}C was excreted in the faeces and urine. Excretion levels reached a plateau by day 2. The concentration of ^{14}C in milk reached a plateau by day 3 at all dosing levels. Concentrations of ^{14}C were higher in liver and kidney than in the other tissues or organs, most of it being incorporated into natural products. The main metabolites identified in the kidney were glycine, *N*-formylglycine, ethylenediamine, *N*-acetylethyl-enediamine, ethyleneurea, ethylenethiourea (ETU) and ethylenebisisothiocyanate sulphide.

When laying hens were dosed with [^{14}C]mancozeb in the feed, most of the ^{14}C was excreted in the faeces. ^{14}C levels in whole eggs were still increasing at the end of the 7-day dosing period, but declined rapidly in eggs from a group of hens in which dosing was discontinued. Ethyleneurea was the identified metabolite present at highest levels in eggs and tissues. ^{14}C was present at higher levels in liver and kidney than in other organs or tissues. In the highest dosed group (equivalent to 36 ppm mancozeb in the feed) dithiocarbamate levels (as CS_2) by direct chemical analysis were: muscle 0.02-0.04 mg/kg, liver 0.09 mg/kg, and eggs 0.007-0.02 mg/kg. ETU levels in the tissues of this group were either at or below the level of detection (0.007 mg/kg), and in eggs were 0.06 mg/kg. ETU levels in eggs were not detectable (<0.007 mg/kg) in the group dosed at the equivalent of 14 ppm.

Most of the ^{14}C was incorporated into the carbon pool, appearing in a range of natural products, when a tomato crop was treated with [^{14}C]mancozeb. Ethyleneurea was the major primary metabolite identified.

When a soya bean crop was treated with [^{14}C]mancozeb the primary metabolites identified in soya bean pods were 1-(2-imidazolin-2-yl)-2-imidazolidinethione, ethyleneurea, hydantoin and ethylenebisisothiocyanate sulphide. Much of the ^{14}C was incorporated into protein, lignin

and oil.

In a sugar beet crop treated with [^{14}C]mancozeb, 1-(2-imidazolin-2-yl)-2-imidazolidinethione was the major primary metabolite to be identified. The total ^{14}C label was distributed 77% in the leaf and stem, and 23% in the root.

The primary metabolites identified in wheat which had received foliar applications of [^{14}C]mancozeb were ethyleneurea, ethylenediamine, ethylenebisisothiocyanate sulphide, 2-imidazoline and 1-(2-imidazolin-2-yl)-2-imidazolidinethione. Much of the ^{14}C was incorporated into carbohydrates.

Mancozeb is registered as a protective fungicide for use on citrus fruits, pome fruits, stone fruits, berries and other small fruits, tropical and subtropical fruits, bulb vegetables, root and tuber vegetables, Brassica vegetables, leafy vegetables, stalk and stem vegetables, fruiting vegetables, legume vegetables, cereals, tree nuts, oilseeds and miscellaneous crops in very many countries.

Typical spray concentrations for high-volume application of mancozeb were 0.15-0.20 kg ai/hl to a wide variety of crops in many countries, but higher concentrations were recommended in some cases. The application rate for high-volume application depended on the volume of spray per hectare required for the particular crop and the typical spray concentration.

The Meeting received extensive residue data from supervised trials on the following crops and commodities:

grapefruit (*USA*), lemons (*Spain, USA*), limes (*USA*), mandarins (*Japan, Spain*), oranges (*Australia, Brazil, Spain, USA*);

apples (*Australia, Austria, Belgium, Brazil, France, Germany, Hungary, Italy, Japan, Netherlands, UK, USA*), pears (*Australia, Brazil, France, Germany, Italy, Japan, USA*);

apricots (*Australia*), peaches (*Australia, Brazil*), plums (*Brazil, France*);

black currants (*UK*), cranberries (*USA*), grapes (*Australia, Brazil, France, Hungary, Italy, Japan, Portugal*), strawberries (*Japan, Spain*);

avocados (*Brazil*), bananas (*Australia, Brazil, Honduras, USA*), figs (*Brazil*), mangoes (*Australia, Brazil*), papayas (*USA*), passion fruit (*Australia*), persimmons (*Japan*);

garlic (*Brazil, France, Japan*), leeks (*France, Japan*), onions (*Australia, Brazil, Finland, Japan, Netherlands, USA*);

broccoli (*Brazil*), cabbage (*Brazil, Germany, Japan*), cauliflower (*Brazil, Spain*), Chinese cabbage (*Japan, Spain*);

cantaloupes (*USA*); cucumbers (*Australia, Brazil, France, Germany, Japan, Spain, USA*), gherkins (*Germany*), melons (*France, Germany, Japan*), pumpkins (*Australia, Brazil*), squash (*France, Japan*), summer squash (*Australia, France, USA*), watermelons (*Australia, Japan, USA*), winter squash (*USA*);

egg plants (*Brazil*), peppers (*Brazil, Spain*), sweet corn (*USA*), tomatoes (*Brazil, France, Germany, Italy, Japan, Netherlands, Portugal, Spain, USA*);

kale (*Brazil*), lettuce (*Spain*);

azduki beans (*Japan*), beans (*Australia, Brazil, France, Netherlands, Spain*), French beans (*Brazil*), kidney beans (*Japan*), peas (*Brazil, France*);

beet (*Brazil*), carrots (*Australia, Brazil, France, Germany, USA*), lotus (*Japan*), potatoes (*Australia, Brazil, Finland, France, Germany, Italy, Japan, Netherlands, UK, USA*), sugar beet (*France, Italy, Japan*), yams (*Japan*);

asparagus (*France, USA*), celery (*USA*), chard (*Australia*), witloof (*France, Netherlands*);

barley (*Brazil, Netherlands, USA*), maize (*USA*), rice (*Brazil*), summer wheat (*Germany*), wheat (*Brazil, Canada, France, Spain, USA*), winter wheat (*Germany, Netherlands, UK*);

hops (*Germany*);

peanuts (*Australia, USA*), rape seed (*France, Netherlands*);

almonds (*USA*), cocoa (*Brazil*), coffee (*Brazil*),

barley straw (*Netherlands, USA*), maize fodder (*USA*), wheat straw (*Canada, France, Germany, Netherlands, UK, USA*);

almond hulls (*USA*), bean pods and foliage (*Australia*), bean straw (*Australia*), peanut foliage (*Australia*), peanut hay (*USA*), sugar beet leaves (*Italy, Japan, USA*).

Dithiocarbamate residues are expressed as mg CS_2 /kg throughout.

Mancozeb is used as a cover fungicide, often with the same spray concentrations for high-volume application, on a wide range of crops. Because the residue is on the surface and there is no translocation from foliage to fruits, residue levels are often similar on fruits of a similar size.

Mancozeb use patterns are common across the citrus fruits in each country. Spanish trials on mandarins (GAP spray concentration 0.32 kg ai/hl, PHI 15 days) produced dithiocarbamate residues up to 4.7 and 6.6 mg/kg at 14 days. For a similar use pattern on oranges, residues of dithiocarbamates were mostly less than 1 mg/kg (highest 1.3 mg/kg). Japanese trials showed that most of the residues are in the peel while the Spanish trials confirmed that washing the fruit generally removes 90% or more of the residue. The Meeting estimated maximum residue levels of 10 mg/kg and 2 mg/kg for mandarins and oranges respectively, based on mancozeb uses.

US trials on lemons, limes and oranges demonstrated that most residues of both dithiocarbamates and ETU were on the peel with little in the pulp. US data on citrus could not be evaluated because there was no US GAP.

Residue data and mancozeb GAP for apples were available from many countries. The mancozeb spray concentrations used in high-volume applications were quite similar in most countries (0.15-0.2 kg ai/hl). GAP information from France did not include a PHI so French data were evaluated according to the German GAP for pome fruit. Residues in apples above 1 mg/kg were recorded in trials in Australia, Austria, Brazil, Germany, Italy and the UK when mancozeb was used within GAP. The highest recorded residue exceeded 4 mg/kg. The Meeting estimated a maximum residue level of 5 mg/kg for apples.

Use patterns on pears were the same as on apples, with the highest recorded residue being 2.2 mg/kg. The Meeting recommended an MRL for pome fruit of 5 mg/kg for dithiocarbamates, based on mancozeb uses.

The number of trials on apricots, peaches and plums was inadequate to recommend MRLs. No data were available for cherries. The Meeting agreed to withdraw the MRL recommendations for cherries, peaches and plums.

Grape residue data were supplied from many countries. The highest residues from the main population of data were in the 2.1-2.8 mg/kg range (Italy) suggesting an MRL of 5 mg/kg. Australian trials produced residues higher than 20 mg/kg when mancozeb was used according to GAP, and residues seemed somewhat anomalous when compared with similar uses elsewhere. The Australian use pattern is currently under review; Australian residue data were not included in the current evaluation.

The number of trials on strawberries was inadequate to permit the estimation of a maximum residue level. The Meeting recommended the withdrawal of the strawberry MRL.

A consistent series of mancozeb trials on cranberries in the USA in 1985 and 1988 suggested an MRL of 5 mg/kg.

The highest residues in black currants from UK mancozeb trials exceeded 5 mg/kg (5.1, 5.4 mg/kg). The Meeting estimated an MRL of 10 mg/kg for currants.

Residue data on bananas and mangoes are mutually supportive with similar uses leading to a similar range of residues. The Meeting estimated a maximum residue level of 2 mg/kg for banana and mango. Data on papayas, where the use pattern permits harvest on the same day as application, suggested an MRL of 5 mg/kg. The number of trials for avocados, figs and passion fruit was too limited for recommendations.

Residue data on garlic were made available from trials in Brazil, France and Japan. Generally, residues were not detectable (<0.05 mg/kg and lower) as would be expected from a foliar-applied non-systemic fungicide. However, residues were detected in a control sample at 0.1 mg/kg, and the possibility should not be excluded that some varieties of garlic or some conditions of production and storage could generate endogenous CS_2 as in onions. Mancozeb trials on leeks in France and Japan were made available for evaluation. The highest residue of 0.30 mg/kg and the possibility of endogenous CS_2 (a control sample registered 0.21 mg/kg of CS_2) suggested a maximum residue level of 0.5 mg/kg for garlic and leeks.

Onion trials in Brazil, Japan, The Netherlands and the USA showed residues up to 0.17 mg/kg, with control samples in Japan at 0.12 mg/kg. The highest residues in onions were in an Australian trial at 1.7 mg/kg but appeared to be an order of magnitude higher than others and difficult to explain for an immobile residue such as mancozeb. The Meeting agreed to evaluate bulb onions, garlic and leeks as a group, and estimated a maximum residue level of 0.5 mg/kg for onions resulting from mancozeb use.

Residue data from trials on broccoli and cauliflower in Brazil in 1989 according to GAP were mutually supportive, and suggested a maximum residue of 0.2 mg/kg. Broccoli has, however, been shown to contain endogenous CS_2. In a US study 8 samples of broccoli (6 varieties, 6 sites in the USA) certified to be untreated with dithiocarbamates, on analysis contained CS_2 residues ranging from undetectable (<0.01 mg/kg) to 0.79 mg/kg, median 0.32 mg/kg. The Meeting had no information on endogenous CS_2 levels in cauliflower. It did not estimate a maximum residue level for broccoli or cauliflower because of the limited number of trials. The Meeting drew attention to the endogenous CS_2 levels in broccoli and possible endogenous CS_2 in related crops.

The highest residue in cabbages from trials according to GAP in Brazil and Japan was 0.22 mg/kg. Chinese cabbage from trials in Japan contained residues of 0.1 mg/kg in the untreated control, again suggesting endogenous CS_2 in the various Brassica vegetables. The Meeting was unable to recommend MRLs for cabbage or Chinese cabbage because of the limited data.

Cucumber residue data from trials according to GAP were supplied from Australia, Brazil, France, Japan and the USA, with residues up to 0.3 mg/kg in US trials. The Meeting estimated a maximum residue level of 0.5 mg/kg for cucumbers, based on mancozeb uses.

Residues in melons from the same use patterns were generally in the same range as in cucumbers. The Meeting recommended an MRL of 0.5 mg/kg for melons except watermelon.

There were only two trials on pumpkins according to GAP, one from Australia and one from Brazil, but residues were generally consistent with those in other cucurbits. The Meeting estimated a maximum residue level of 0.2 mg/kg for pumpkins.

Summer squash in trials in Australia, France and the USA showed residues from undetectable levels to 0.83 mg/kg, the last in a US trial where the harvest took place on day 4 after the last application. Residues would have been higher than on day 5 (the recommended PHI), but the level on day 10 was still 0.65 mg/kg. The Meeting estimated an MRL of 1 mg/kg for summer squash.

US data on winter squash could not be evaluated because no US GAP was available. Residues in squash in trials in France and Japan were quite similar, even though there was quite a difference in the use patterns, with PHIs of 3 and 30 days in France and Japan respectively. The Meeting estimated an MRL of 0.1 mg/kg for winter squash.

A US watermelon trial with mancozeb used 12 applications, but this would probably have little influence on the residues since US GAP allows a maximum of 8. The residue level on day 5 after the final treatment was 0.38 mg/kg. In the Australian trials residues were not detected (<0.1 mg/kg), and in the Japanese trials residues were measured on the watermelon pulp rather than the whole fruit. Residues in the pulp were at quite low levels, 0.01-0.02 mg/kg. The Meeting estimated a maximum residue level of 0.5 mg/kg, based on mancozeb uses on watermelon.

When mancozeb was used according to GAP on peppers in Brazil and Spain the highest residues were in the 0.5-0.6 mg/kg range. The Meeting recommended an MRL of 1 mg/kg for sweet peppers.

Sixty-eight trials with mancozeb on tomatoes were available from many countries. Many of the measured residue levels were in the 0.1-1 mg/kg range, but residues up to 4.1 mg/kg were recorded in the US trials. The Meeting recommended an MRL of 5 mg/kg for tomatoes.

US trials on sweet corn showed that dithiocarbamate residues were on the husk rather than in the kernels. Residues were not detected (<0.03 mg/kg) in the cob + kernels. Additional data were available from US processing studies where application of mancozeb at the recommended US rate produced undetectable residues (<0.03 mg/kg) in cob + kernels. The residue level was 0.03 mg/kg when mancozeb was used at 5 times the recommended rate. Mancozeb, an immobile residue, would not be expected in the cob and kernels, which are protected by the husk from direct application. The Meeting recommended an MRL for sweet corn of 0.1* mg/kg as being a practical limit of quantification.

In supervised mancozeb residue trials on kale in Brazil dithiocarbamate residues 14 days after the last application were 0.95 and 1.0 mg/kg for label rate and double label rate of application, but the number of trials was too limited to allow the estimation of a maximum residue level.

When lettuce was sprayed with mancozeb at 0.16 kg ai/hl in trials in Spain and harvested 14 days after the final application residues in the 3-10 mg/kg range were found. The Meeting estimated a maximum residue level of 10 mg/kg for mancozeb use on head lettuce.

Trials in Japan on adzuki beans and kidney beans, and in Brazil on beans and French beans generally demonstrated undetectable or low residues on bean seeds, but the LOD for some of the older results was too high to be useful. The Meeting was unable to recommend an MRL for dry beans because of the limited data. It was not completely clear whether the commodity analysed in the Brazilian trials on peas included peas + pods, or peas only. The Meeting did not recommend an MRL for beans or peas.

Information on mancozeb residues in beetroot from trials in Brazil was made available, but the number of trials was insufficient to recommend an MRL.

Most residue levels in carrots arising from approved uses of mancozeb were less than 0.2 mg/kg, but a number of values were found in the 0.5-1 mg/kg range in the Brazilian trials. The Meeting estimated a maximum residue level of 1 mg/kg for carrots.

Dithiocarbamate residues were not detected (<0.02 mg/kg) in East Indian lotus in two trials from Japan, but the data were insufficient to estimate a maximum residue level.

One hundred and seventeen mancozeb potato trials, but many not within GAP, were available from 9 countries for review. Residues were mostly undetectable even when mancozeb had been used at exaggerated application rates. Residues were sometimes detected, and the residues are more likely to depend on the inadvertent spraying of exposed potatoes than on the application rates or pre-harvest intervals. The highest residues were found in a French trial at 0.32 mg/kg and a German trial at 0.26 mg/kg, but they appeared exceptional when compared with all the other results. The Meeting estimated a maximum residue level of 0.2 mg/kg for uses of mancozeb on potatoes.

Dithiocarbamate residues from sugar beet trials in France, Italy and the USA were mostly around 0.1 mg/kg or lower, but residues in the 0.2-0.4 mg/kg range were recorded in US trials. The Meeting recommended a maximum residue level of 0.5 mg/kg for mancozeb use on sugar beet.

The US use pattern for mancozeb on asparagus requires a long PHI, 120 days in some states and 180 days in others. As expected, residues were low after this interval in the US trials. The French trials on asparagus could not be evaluated because no information on the French PHI was available. The Meeting recommended a maximum residue level of 0.1 mg/kg for asparagus.

No US GAP for mancozeb uses on celery was available to permit evaluation of US trials. Only one trial on chard according to GAP was available, from Australia, and this was insufficient in the absence of data from other similar vegetables which could have provided mutual support. Witloof trial data from France and The Netherlands could not be evaluated in the absence of GAP information.

Results of barley trials in Brazil, The Netherlands and the USA were made available to the Meeting. Dithiocarbamate residues up to 0.55 mg/kg were recorded in the US trials, and an MRL of 1 mg/kg for barley is recommended.

Results of a large number of mancozeb trials on wheat were supplied from 8 countries. The highest dithiocarbamate residues were recorded from trials in France (0.26 mg/kg), Germany (0.4 mg/kg), The Netherlands (0.82, 0.75 and 0.49 mg/kg) and the UK (0.42, 0.5 mg/kg), but in many of the trials residues were not detected. The Meeting estimated a maximum residue level of 1 mg/kg for mancozeb uses on wheat.

The PHI for the use of mancozeb on maize in the USA is 40 days; most of the residue data in the supervised trials were from shorter treatment-to-harvest intervals, and so could not be

evaluated. In two trials where the longer interval was observed the commodity analysed was the "ear". Presumably this is the cob + grain. The appropriate commodity for a maize MRL is the grain.

Data from two supervised trials on rice according to the conditions of Brazilian GAP were made available to the Meeting. The data suggest a maximum residue level of 2 mg/kg, but trials covering a wider range of conditions are desirable for such an important crop. Also, if dithiocarbamate residues in this range or higher are likely, information on their fate during milling and cooking is desirable.

Two German trials with mancozeb on hops led to dithiocarbamate levels in dry hops of 2.2 and <1 mg/kg, but the information was too limited to permit the estimation of a maximum residue level.

Dithiocarbamate residues were not detected (<0.1, <0.03 mg/kg) in peanuts in Australian and US trials even when exaggerated application rates were employed. An MRL of 0.1* mg/kg was recommended.

Residues were detected in almonds in an Australian trial at the recommended application rate, but not at twice this rate. Because mancozeb is a surface residue only it is likely that any residues detected in the kernel were physically transferred during the cracking process. In the US trials dithiocarbamate residues were present in the almond hulls at 3 mg/kg, but no residues were detected (<0.03 mg/kg) in the almonds. The Meeting estimated a maximum residue level of 0.1* mg/kg for the use of mancozeb on almonds.

Mancozeb trials on cocoa and coffee in Brazil were insufficient for the Meeting to estimate maximum residue levels for cacao beans or coffee beans.

Residue data were available for wheat straw and fodder harvested at the same time as the wheat in the previously mentioned trials. Data on barley straw from trials in The Netherlands were also included for evaluation. Many of the residues were in the 2-5 mg/kg range but residues ranged up to 18 mg/kg. Two additional trials on barley with an identical use pattern were available from the USA, with residues of 24 mg/kg on barley straw from one of them. Wheat straw and barley straw should be assessed together for the same use pattern. The Meeting estimated maximum residue levels of 25 mg/kg for both. This level is compatible with animal commodity MRLs recommended on the basis of animal transfer studies.

Dithiocarbamate residues of 1.2 and 1.4 mg/kg were found in maize plants in two US trials 39 and 40 days after the final application of mancozeb. The Meeting estimated a maximum residue level of 2 mg/kg for maize fodder.

Dithiocarbamate residues up to 3.3 mg/kg on peanut foliage from previously mentioned Australian trials permitted the Meeting to estimate a maximum residue level of 5 mg/kg for peanut fodder. Data on almond hulls and peanut hay from US trials could not be evaluated because no US GAP was available for almonds and application rates on the peanuts were in excess of recommended rates.

When mancozeb was used on sugar beet crops according to US GAP, dithiocarbamate residues up to 17 mg/kg were found on sugar beet leaves. The Meeting estimated a maximum residue level of 20 mg/kg for sugar beet leaves or tops from mancozeb use.

Animal transfer studies with lactating dairy cows and laying hens were made available to the Meeting.

When dairy cows were fed a diet containing aged mancozeb residues equivalent to 5, 15 and 45 ppm mancozeb for 28 days dithiocarbamate residues were not detected (<0.04 mg/kg

as CS_2) in the milk from any group. In the highest feeding group residues were not detected (<0.02 mg/kg,as CS_2) in muscle, while residues in the kidney and liver were 0.04 and 0.1 mg/kg respectively. The Meeting estimated maximum residue levels of 0.05*, 0.02* and 0.1 mg/kg for milks, meat and edible mammalian offal, respectively. These levels should accommodate animals eating 45 ppm mancozeb (25 ppm as CS_2) in the diet.

ETU residues were not detected (<0.01 mg/kg) in milk from the highest feeding group, but were detected in the thyroids of all the animals, with the highest doses causing the highest levels. ETU was detectable in muscle, liver and kidney of the highest feeding group, but had disappeared from the tissues of an animal returned to a residue-free diet for 7 days.

When laying hens were fed aged mancozeb residues (5, 15 and 45 ppm as mancozeb) for 28 days, dithiocarbamate residues were not detected (<0.04 mg/kg as CS_2) in the eggs from any feeding group. In the middle and highest feeding groups residues were 0.08 and 0.09 mg/kg (as CS_2) in muscle, while residues in the liver were 0.03 mg/kg. Measured residues in control samples were also around 0.03 mg/kg. The Meeting estimated maximum residue levels of 0.05*, 0.1 and 0.1 mg/kg for eggs, poultry meat and poultry edible offal, respectively.

ETU residues were detected in some eggs from the highest feeding group (0.01-0.02 mg/kg), but were not detected in tissues.

Processing studies were made available to the Meeting on apples, grapes, sweet corn, tomatoes, potatoes, sugar beet, barley, wheat, maize and peanuts.

In general, mancozeb residues (which are on the surface) can be substantially diminished by vigorous washing. The remaining residues tend to remain with the insoluble fractions, so that clear juices are unlikely to contain them. The remaining mancozeb residues may, however, be converted to ETU if processing includes a heating step.

In the commercial processing of apples, washing removed 30-50% of the residue, the remainder being carried through the process into the pomace. Neither mancozeb nor ETU residues were detectable in clarified apple juice.

De-stemming and cleaning removed about 70% of the mancozeb residues from bunches of grapes. Dithiocarbamate residues were not detectable in clear grape juice, but were present in the thick juice. ETU was generated in the production of the grape juices and jelly.

Less than 1% of the dithiocarbamate residues in mancozeb-treated grapes entered red and white wines produced from them. Approximately 7% conversion to ETU occurred during the wine production.

In one study mancozeb residue levels in dried raisins were on average 3 times as high as in the raw grapes, while in another study levels in the raisins were 20-50% of the levels in the grapes. No ETU was generated in raisin production.

Mancozeb residues in frozen corn and canned corn were less than 10% of the levels in the raw sweet corn whole ears; ETU was not generated in the process.

The commercial washing of tomatoes removed more than 90% of the mancozeb residues. Dithiocarbamate residues in the tomato juice and pomace produced from the washed tomatoes were undetectable. ETU residue levels in the juice were of the same order as the dithiocarbamate levels in the washed tomatoes.

Dithiocarbamate residues were essentially undetectable (<0.1 mg/kg) in potatoes field-treated with mancozeb at an exaggerated rate, and in the processed potato products. ETU was present in potato granules (0.08 mg/kg) and potato flakes (0.23 mg/kg).

Dithiocarbamate and ETU residues were not detected (<0.03 and <0.01 mg/kg respectively) in white sugar produced from mancozeb-treated sugar beet containing dithiocarbamate residues of 0.15 mg/kg.

The cleaning of barley grain prior to milling reduced residue levels by 70%. Mancozeb residues were not detectable in bran or flour.

Milling and baking trials on wheat harvested after foliar mancozeb applications showed that dithiocarbamate residues in the bread were either undetectable or, on average, 30% of the levels in the grain. ETU was not detectable (<0.01 mg/kg) in the bread.

Maize was field-treated with mancozeb and harvested for processing into meal, flour, germ, grits, crude oil, refined oil and soapstock. Neither dithiocarbamates nor ETU were detected in the maize kernels or any of the products (<0.03 and <0.01 mg/kg respectively).

A peanut crop was field-treated with mancozeb and harvested for processing into meal, crude oil, refined oil and soapstock. Neither dithiocarbamates nor ETU were detected in the raw peanuts or any of the products (same limits as above).

The ETU level was 0.04 mg/kg in beer produced from mancozeb-treated hops (dithiocarbamates 2.2 mg/kg as CS_2).

Typical consumer practices were shown to reduce mancozeb residue levels in potatoes, tomatoes, apples and onions. Residues in potatoes subjected to washing, brushing, drying and peeling were reduced by 97%. Residues in tomatoes and apples subjected to washing and drying were reduced by 80% and 65% respectively. Residues in onions were reduced by 95% on peeling.

Mancozeb residues were stable (>70% remaining) in homogenised samples of apples, tomatoes and wheat stored for 2 years at -20°C. ETU residues were more labile; more than 70% of the ETU remained in tomato and wheat matrices after 12 months storage, but not after two years. ETU residues in an apple matrix had declined to less than 70% after 6 months storage and to less than 50% after 12 months.

Mancozeb residues were shown to be stable at -20 ± 5°C in stored analytical samples of dry beans, corn, lettuce, meat, milk, raw potato (marginal stability), and tomato. ETU residues were shown to be stable at -20 ± 5°C in stored analytical samples of dry beans, corn, lettuce (marginal stability), meat, milk, raw potato (marginal stability), and tomato.

Under a US Food and Drug Administration monitoring programme a variety of baby foods (864 samples) were monitored for pesticide residues. ETU residues were detected in 65 samples; the highest levels detected were 0.06 mg/kg.

In 1989-90 in the USA a large survey of food items (approximately 300 samples each of 19 different raw and processed commodities) was conducted for dithiocarbamate and ETU residues. Most of the samples (91% of 5241 samples) did not contain measurable dithiocarbamate residues (<0.003 mg/kg as CS_2); broccoli and onions were excluded because of endogenous CS_2 generation. No measurable residues of ETU (LOD 0.001 mg/kg) were found in 82% of the samples.

Grape juice samples (100), from major grape juice producers in the USA using grapes from districts where dithiocarbamates had been used on the 1990 crop, contained no detectable ETU residues (LOD 0.005 mg/kg). Dithiocarbamates were detected in 92 of the samples (median value approximately 0.022 mg/kg as CS_2). If the dithiocarbamates were ethylenebis(dithiocarbamate)s, ETU should also have been detected because the production

of grape juice involves several heating steps. There was a suggestion that ferbam, a dithiocarbamate fungicide but not an ethylene-bis(dithiocarbamate), may have been the source of some of the dithiocarbamate residues.

In an Australian study in 1991, ETU residues were not detected (<0.1 mg/kg) in tomatoes, commercially produced tomato paste or thin pulp (41 samples).

Analytical methods for dithiocarbamates rely on the generation of CS_2, which can be measured by GLC or by colorimetry.

Reaction with hydrochloric acid + stannous chloride at 100°C is needed for quantitative conversion to CS_2, which can be analysed by head-space GLC. Alternatively, the evolved CS_2 can be swept by a current of air into an ethanol trap maintained at dry ice/acetone temperature, and the ethanol solution then analysed by GLC. In the colorimetric approach the evolved CS_2 is swept into a trap of cupric acetate/diethanolamine reagent. Some types of sample can give a false response by generating a false colour in the reagent.

A UK Panel on the Determination of Dithiocarbamate Residues (1981) drew attention to the loss of dithiocarbamate residues which can occur between commencement of cutting of the sample and insertion into the reaction bottle. Vegetables and fruits must be analysed for residues as soon as possible after cutting or picking, and any further cutting or dicing of the whole commodity should be carried out immediately before placing in the reaction flask, and should be kept to a minimum. Foodstuffs should be frozen whole, when this becomes necessary, and chopped and mixed in the frozen state immediately before taking the analytical samples.

ETU methods rely on HPLC or GLC for final analysis. Samples are typically extracted with aqueous ammonia (pH 11-12) + methanol or ethanol and the extract cleaned up on an alumina column. ETU is easily oxidised or lost during the analysis; precautions are needed, such as the use of silanized glassware. Precautions must also be taken to prevent ethylenebisdithiocarbamate residues from being converted to ETU during the analysis.

The Meeting was aware of national MRLs established in Australia, Canada, Germany, Mexico, Spain and the USA.

FURTHER WORK OR INFORMATION

Desirable

1. Supervised trials on rice covering a wider range of conditions.

2. Fate of mancozeb residues during the milling and cooking of rice.

4.29 MANEB (dithiocarbamates, 105)

TOXICOLOGY

Maneb was evaluated by the Joint Meeting in 1963, 1965, 1967, 1970, 1974, 1977 and 1980. An ADI of 0-0.05 mg/kg bw was established at the 1980 Meeting for maneb or the sum of any combination of maneb, mancozeb, and zineb, of which not more than 0.002 mg/kg bw may be present as ethylenethiourea (ETU).

Male and female rats given 25 mg/kg bw/day of ^{14}C labelled maneb orally showed no differences between sexes with regard to excretion patterns. More than 90% of the absorbed

^{14}C was eliminated in urine by 24 hours. Less than 1% was eliminated as carbon dioxide. The average ^{14}C concentration as a percentage of the dose per g tissue was greatest for the thyroid, followed by the kidney and liver. The percentage of ^{14}C present in urine at 12 hours on a mole/mole basis was 21-30% as ETU and less than 0.4% as maneb.

The acute oral, dermal and inhalation toxicity of maneb technical and maneb 75% dust is low. WHO has classified maneb as unlikely to present acute hazard in normal use.

Rats were fed dietary concentrations of 0, 80, 400 or 1300 ppm maneb technical for 13 weeks. The NOAEL was 80 ppm (equal to 5.0 mg/kg bw/day), based on an increase in absolute thyroid weight and thyroid follicular cell hyperplasia at 400 ppm.

In dogs fed dietary concentrations of maneb technical at 0, 100, 400 or 1600 ppm for 13 weeks, the NOAEL was 100 ppm (equal to 3.7 mg/kg bw/day), based on thyroid follicular cell hyperplasia at 400 ppm.

In a 52-week study in dogs, maneb was administered at dietary concentrations of 0, 50, 200, 1000 or 2200 ppm. The NOAEL was 200 ppm (equal to 6.4 mg/kg bw/day), based on thyroid enlargement and thickening and thyroid follicular cell hyperplasia at 1000 ppm.

The overall NOAEL in dogs, based on the evaluation of all of the data on this species, was 6.4 mg/kg bw/day.

In a six-month study in monkeys, maneb was administered at dietary concentrations of 0, 100, 300 or 3000 ppm. The NOAEL was 100 ppm (equal to 7.3 mg/kg bw/day), based on an increase in thyroid weight at 300 ppm.

In a 79-week carcinogenicity study in mice at dietary concentrations of 0, 60, 240 or 2400 ppm, the NOAEL was 60 ppm (equal to 11 mg/kg bw/day), based on decreased body weight and decreased thyroxine levels at 240 ppm. Hepatocellular adenomas were observed at 2400 ppm in both sexes.

In a 31-month toxicity/carcinogenicity study in rats at dietary concentrations of 0, 30, 100, 300 or 1000 ppm, the NOAEL was 300 ppm (equal to 20 mg/kg bw/day), based on decreased body weight, an increase in the half-life retention time of ^{131}I in the thyroid, decreased T_4 values and an increased absolute thyroid weight at 1000 ppm. There was no evidence of carcinogenicity.

In a two-generation reproduction study in rats at dietary concentrations of 0, 75, 300 or 1200 ppm, the NOAEL was 75 ppm (equal to 5.6 mg/kg bw/day), based on increased organ-to-body-weight ratios for liver and kidney, and thyroid follicular cell hyperplasia at 300 ppm.

An oral teratogenicity study in rats was conducted at dose levels of 0, 20, 100 or 500 mg/kg bw/day. An NOAEL of 20 mg/kg bw/day for maternal toxicity and embryo/fetotoxicity was established. Maternal toxicity was seen at 100 mg/kg bw/day as decreased body weight and decreased food consumption. Embryo/fetotoxicity was observed as increased (early) resorptions, increased post-implantation losses and a decrease in viable fetuses at 100 mg/kg bw/day. No teratogenicity was observed.

In a second oral teratogenicity study in rats conducted at dose levels of 0, 20, 100 or 500 mg/kg bw/day the NOAEL for maternal toxicity and embryo/fetotoxic and teratogenic effects was 100 mg/kg bw/day. Maternal toxicity was seen at the highest dose as decreased body weight and clinical signs. Embryo/fetotoxicity and teratogenicity were seen at the highest dose as decreased fetal body weight and body length, and an increase in the number of anomalous litters and fetuses for all malformations combined and for all variations and retardations combined.

An oral teratogenicity study was conducted in rabbits at dose levels of 0, 5, 20 or 80 mg/kg bw/day. Owing to study deficiencies, an NOAEL could not be determined.

Maneb has been adequately tested in a series of *in vitro* and *in vivo* genotoxicity assays. The Meeting concluded that maneb is not genotoxic. A number of available studies were not considered, either because DMSO was used as a solvent in which maneb is very unstable or because of important omissions from the reports.

The data on maneb would support an ADI of 0-0.05 mg/kg bw, based on the NOAEL of 5.0 mg/kg bw/day for thyroid effects in rats, using a 100-fold safety factor. However, the Meeting established a group ADI of 0-0.03 mg/kg bw for maneb, alone or in combination with mancozeb, metiram and/or zineb, because of the similarity of the chemical structures of the EBDCs, the comparable toxicological profiles of the EBDCs based on the toxic effects of ETU, and the fact that parent EBDC residues cannot be differentiated using presently-available regulatory analytical procedures (see Section 4.15 - dithiocarbamates).

A toxicological monograph summarizing the data received since the previous evaluation and containing relevant summaries from previous monographs and monograph addenda on maneb was prepared.

TOXICOLOGICAL EVALUATION

Level causing no toxicological effect

Mouse: 60 ppm in the diet, equal to 11 mg/kg bw/day (79-week study)

Rat: 80 ppm in the diet, equal to 5.0 mg/kg bw/day (13-week study)
300 ppm in the diet, equal to 20 mg/kg bw/day (31-month study)
75 ppm in the diet, equal to 5.6 mg/kg bw/day (reproduction study)

Dog: 200 ppm in the diet, equal to 6.4 mg/kg bw/day (52-week study)

Monkey: 100 ppm in the diet, equal to 7.3 mg/kg bw/day (six-month study)

Estimate of acceptable daily intake for humans

0-0.03 mg/kg bw (group ADI with mancozeb, metiram, and zineb)

Studies which will provide valuable information in the continued evaluation of the compound

Observations in humans.

RESIDUE AND ANALYTICAL ASPECTS

Maneb, first evaluated in 1967, was scheduled for periodic re-evaluation at the 1993 JMPR (ALINORM 93/24A, para 133).

The Meeting received extensive information on GAP, supervised residue trials, metabolic fate in farm animals and crops, fate during processing, residues in food in commerce and at consumption, and methods of residue analysis.

When [^{14}C]maneb ([^{14}C]ethylenediamine) was fed to lactating goats for 5 days at the equivalent of 50 ppm maneb in the feed, the total ^{14}C residues in milk were close to a steady-state concentration by days 3 and 4. The levels in the morning milk samples, collected just

before the daily dose, were always considerably lower than in the evening samples, which suggested that levels in milk would decrease rapidly when dosing ceased. Total ^{14}C residues were distributed among the tissues and milk, with the highest levels in the liver and kidney. Ethyleneurea, 1-(2-imidazolin-2-yl)-2-imidazolidinethione and ethylenethiourea (ETU) were identified in all the tissues and milk. The main primary metabolite was Jaffe's base (1-(2-imidazolin-2-yl)-2-imidazolidinethione). Much of the ^{14}C had been incorporated into natural products.

ETU was identified in the goat tissues and milk. The levels of ETU by direct chemical analysis were: liver 0.075 mg/kg, kidney 0.050 mg/kg, muscle 0.035 mg/kg, fat <0.01 mg/kg, milk 0.037 mg/kg.

When [^{14}C]maneb ([^{14}C]ethylenediamine) was fed to laying hens for 7 days at the equivalent of 51 ppm maneb in the feed, the total ^{14}C residues in egg whites had reached a plateau by days 5-6 while the total ^{14}C in egg yolk was still increasing at the end of the study. Total ^{14}C was distributed among the tissues, but the highest levels were in the liver and kidney. Ethyleneurea was the main metabolite. Ethyleneurea, 1-(2-imidazolin-2-yl)-2-imidazolidinethione and ethylenediamine were identified in all of the tissues, egg white and egg yolk. Much of the ^{14}C had been incorporated into natural products.

ETU was identified in all of the tissues (except skin) and eggs. ETU levels in tissues and eggs by direct chemical analysis were: liver 0.14 mg/kg, breast muscle 0.044 mg/kg, egg white 0.098 mg/kg, and egg yolk 0.039 mg/kg.

Lettuce plants treated with foliar sprays of [^{14}C]maneb were harvested and surface-rinsed with an EDTA solution to identify the components of the dislodgable residue. Surface residues included mainly maneb and the primary metabolites ethylenebisisothiocyanate sulphide, ethyleneurea and ethylenethiourea. The identified metabolites in the lettuce tissue included ethylenebisisothiocyanate sulphide, ethyleneurea, ethylenethiourea, ethylenediamine and *N*-acetylethylenediamine. Amino acids and protein were found to contain ^{14}C, which demonstrated that metabolites had been incorporated into the natural carbon pool. ETU accounted for 7% of the total ^{14}C residues in lettuce + rinsings, or 2.8% of the total ^{14}C in the rinsed lettuce.

Most of the ^{14}C residues were in the foliage, with less than 0.3% in the tubers, of potatoes harvested 17 days after the final foliar application of [^{14}C]maneb. The primary metabolites constituted only a minor part, less than 9%, of the residues in the tuber. ETU identified in the potato peel, but not in the body of the tuber, (0.02 mg/kg tuber, by direct chemical analysis) was thought to be the result of contamination rather than of metabolism. The metabolism may be interpreted in terms of a relatively rapid conversion of the primary metabolites to a common plant metabolite such as glycine, which provides the mechanism for the ^{14}C to be incorporated widely into natural products.

In a metabolism study tomato plants were treated with foliar sprays of [^{14}C]maneb and harvested 24 days after the final application for metabolite identification and analysis. Much of the ^{14}C residue (49-63%) was dislodgable and was removed from harvested tomatoes when they were washed with a 1% EDTA solution. Maneb and EBIS (ethylenebisisothiocyanate sulphide) constituted the major part of the dislodgable residue; most of the ETU residue in the whole tomatoes was dislodgable. EBIS was the major metabolite identified in the whole tomato. The processes in tomato metabolism are similar to those in the other crops studied. The ^{14}C enters the metabolic carbon pool probably via glycine, from which it is incorporated into natural products.

Maneb is registered as a protective fungicide for use on pome fruits, stone fruits, berries and other small fruits, tropical and subtropical fruits, bulb vegetables, root and tuber vegetables, Brassica vegetables, leafy vegetables, stalk and stem vegetables, fruiting vegetables, legume vegetables, cereals, and tree nuts in many countries.

Typical spray concentrations for high-volume application are 0.15-0.3 kg ai/hl, and typical application rates for a wide range of crops are 1.3-3 kg ai/ha.

The Meeting received residue data from supervised trials on the following crops and commodities:

apples (*Netherlands, USA*), peaches (*USA*), grapes (*USA*);

onions (*USA, Netherlands*), broccoli (*USA*), cabbage (*USA*), cucumbers (*USA*), watermelons (*USA*), endive (*Canada*), kale *(USA*), lettuce (*Canada, USA*), beans (*USA*), peppers (*USA*), sweet corn (*USA*), tomatoes (*Netherlands, USA*), potatoes (*Netherlands, UK, USA*), sugar beet (*USA*), celery (*USA*);

barley (*Netherlands*), wheat (*Netherlands, UK, USA*), almonds (*USA*);

barley straw (*Netherlands*), maize forage (*USA*), wheat straw (*Netherlands, UK*) bean vines (*USA*), almond hulls (*USA*), sugar beet tops (*USA*).

Dithiocarbamate residues are expressed as mg CS_2/kg throughout.

The residue data on apples from The Netherlands could not be evaluated because the recommended use pattern was expressed in terms of spray concentration while the trial use pattern was expressed in terms of application rate. The US apple trials did not meet GAP conditions because the longest treatment-to-sampling interval in the trials was 30 days, but the US recommended use pattern requires a 77-day PHI.

Peach trials in the USA could not be evaluated because GAP information was not available.

The highest residues in grapes were 1.8 and 1.9 mg/kg in US trials where maneb was used within GAP conditions. The Meeting estimated a maximum residue level of 2 mg/kg for maneb uses on grapes.

Maneb use on green onions according to US GAP resulted in residues up to 7.4 mg/kg. The Meeting noted that analysis of a control sample of green onions produced 0.5 mg/kg as CS_2, probably resulting from endogenous CS_2. The Meeting estimated a maximum residue level of 10 mg/kg for spring onions.

The maneb application rate to bulb onions in Netherlands trials was 2.4 kg ai/ha, which is higher than the Netherlands recommended rate of 1.6 kg ai/ha. Residues in bulb onions are likely to arise from inadvertent spraying of exposed onions; the application rate will not be so influential on the residues. Dithiocarbamate residues in onions from the Netherlands trials were low (0.1 mg/kg and lower).

The recommended PHI in the USA for bulb onions is 7 days, but in the trials onions were harvested on the same day as the final spray and the results could not be evaluated against the recommended use pattern. The Meeting noted the repeated detection of CS_2 in control samples at levels up to 0.13 mg/kg.

Broccoli in US trials was sampled 3 and 4 days after the final maneb application, but US GAP specifies a PHI of 7 days. The Meeting noted the detection of CS_2 in control samples at levels up to 0.55 mg/kg, which was consistent with other analyses on control broccoli (up to 0.79 mg/kg as CS_2). The Meeting did not estimate a maximum residue level for broccoli because of the limited number of trials. It drew attention to the endogenous CS_2 levels in broccoli and possible endogenous CS_2 in related crops.

The highest residue in untrimmed cabbage from US maneb trials in 1987 was 10 mg/kg, but residues in that trial seemed much higher than the others. These trials and four others in 1989 and 1990 also included analyses of trimmed cabbages; removal of the wrapper leaves reduced maneb residue levels by an average of 30%. The highest residue in a control sample was 0.59 mg/kg, suggesting that endogenous CS_2 levels could be similar to those reported for broccoli. The Meeting estimated a maximum residue level of 5 mg/kg for maneb uses on cabbage and noted that the correct portion of the sample for analysis included the wrapper leaves unless obviously withered or decayed.

Dithiocarbamate residues in cucumbers exceeded 1 mg/kg in one trial when maneb was used according to GAP in a series of trials in the USA in 1987 and 1989. The Meeting estimated a maximum residue level of 2 mg/kg for maneb uses on cucumber.

The highest dithiocarbamate residue in watermelons was 0.57 mg/kg when maneb was used according to US GAP. An experiment in one trial demonstrated that residues existed exclusively on the peel and not in the pulp. The Meeting estimated a maximum residue level of

1 mg/kg for maneb uses on watermelon.

Residues in kale from a series of supervised trials in the USA during 1987 were typically in the 4-8 mg/kg range, but the highest were 14 and 28 mg/kg. The questionable aspect of this trial was that residues on day 10 after the final application were somewhat higher than on day 7. The distribution of the results suggested that residues up to 15 mg/kg would be possible. The Meeting estimated a maximum residue level of 15 mg/kg for maneb uses on kale.

Supervised trials data from Canada and the USA were made available for uses of maneb on lettuce, leaf lettuce and cos lettuce. The commodity described as lettuce was taken to be head lettuce. The highest residues in the US trials on lettuce were in the 5-7 mg/kg range. The highest residues in cos lettuce from the Canadian trials were in the 6-9 mg/kg range. Only one trial was specified as leaf lettuce and residues were just under 1 mg/kg. The Meeting estimated maximum residue levels of 10 mg/kg for uses of maneb on cos lettuce and head lettuce.

Samples from the US trials on lettuce were analysed with and without wrapper leaves. Removal of the wrapper leaves reduced residue levels by an average of 87%.

US maneb trials on spinach could not be evaluated because no US GAP was available. The trials demonstrated that maneb residue levels in washed spinach were about 25% lower than in unwashed spinach.

The official PHI for maneb use on beans in the USA is 30 days; the PHI in the trials was 4 days so no MRL could be recommended. Washing the beans did not significantly affect the dithiocarbamate residue levels.

The use of maneb on sweet peppers in US trials in 1987-89 typically produced residues in the 0.2-1 mg/kg range. The Meeting estimated a maximum residue level of 1 mg/kg for maneb uses on sweet peppers.

For sweet corn in the USA the registered application rate and PHI are 1.3 kg ai/ha and 7 days. The use pattern in the trials was 1.8 kg ai/ha with 4- and 5-day PHIs; consequently, a maximum residue level could not be estimated.

The highest dithiocarbamate residue in tomatoes arising from maneb use within US GAP was 2.0 mg/kg. Most commonly, residues were in the 0.1-0.5 mg/kg range. The Meeting estimated a maximum residue level of 2 mg/kg for the use of maneb on tomatoes.

Potato trials from The Netherlands could not be evaluated because application rates were double the official rate, 1.6 kg ai/ha, and because Netherlands GAP did not specify a PHI. Residues in potatoes in UK trials were undetectable (<0.01 mg/kg) with application at recommended rates and double recommended rates.

In 8 of the 9 US maneb trials on potatoes residues were not detected (<0.03 mg/kg), and in the other trial residues of 0.23 mg/kg were recorded for one plot. Maneb residues are generally immobile in the plant and residues on the tuber are only likely to arise if tubers are exposed above the soil during spraying. The Meeting estimated a maximum residue level of 0.2 mg/kg for maneb uses on potatoes.

In one sugar beet trial in the USA residues were much higher than in the remaining trials. Residues in the sugar beet tops up to 76 and 88 mg/kg seemed excessive for an application rate of 1.8 kg ai/ha. The Meeting was unable to estimate maximum residue levels for sugar beet or sugar beet leaves and tops because the number of trials was too small.

Supervised maneb trials on celery in the USA and barley in The Netherlands could not be evaluated because no relevant GAP was available.

When maneb was used within GAP on wheat in The Netherlands and the UK dithiocarbamate residues were mostly undetectable or in the 0.01-0.05 mg/kg range. The highest residue (0.65 mg/kg) from one plot of a trial in The Netherlands appeared to be anomalous; residues in wheat from the other plot in the same trial were undetectable (<0.01 mg/kg). The Meeting estimated a maximum residue level of 0.2 mg/kg for the use of maneb on wheat.

Residues in almonds from maneb trials in the USA were mostly undetected (<0.03 mg/kg). The Meeting estimated a maximum residue level of 0.05 mg/kg for almonds from the use of maneb.

Residues in wheat straw from The Netherlands and the UK ranged up to 2.1 mg/kg for registered uses of maneb. The Meeting estimated a maximum residue level of 5 mg/kg for wheat straw and fodder, resulting from maneb uses.

GAP information was not available for maize forage or bean vines, so trials data could not be evaluated for MRL purposes. Barley straw data from Netherlands trials evaluated against registered wheat uses supported the estimated maximum residue level in wheat straw and fodder resulting from maneb uses.

Many of the residues in almond hulls were in the 3-10 mg/kg range in US maneb trials on almonds, but the distribution of results suggested that residues in the 10 to 20 mg/kg would be likely from use according to GAP. The Meeting estimated a maximum residue level of 20 mg/kg for almond hulls.

Processing studies were made available to the Meeting on apples, beans, grapes, sugar beet, sweet corn and tomatoes.

Maneb residues in apple juice were approximately 20-50% of the levels in apples when no washing step was included in the process. Maneb residues were retained in the pomace fraction. ETU residue levels in the juice were lower than in the raw commodity.

Beans field-treated with maneb were passed through a simulated commercial process to produce canned beans, frozen beans and pureed beans (baby food). Dithiocarbamate residue levels were much reduced in frozen beans and were at very low levels in canned beans and not detectable in baby food. Heat was used in the production of these commodities; consequently ETU was produced in all of them.

In the processing of maneb-treated grapes dithiocarbamate residue levels in wet pomace and thick grape juice were approximately 60% and 7% respectively of the level in the raw grapes. Juice was heated at 82-85°C before being separated into thick juice and clear juice. The heating caused substantial conversion to ETU, the level in the thick juice being 5 mg/kg.

Dithiocarbamate and ETU residues were not detectable (<0.03, <0.01 mg/kg respectively) in white sugar produced from sugar beet field treated with exaggerated application rates (tenfold) of maneb in the USA.

Dithiocarbamate and ETU residues were not detectable (<0.03, <0.01 mg/kg respectively) in sweet corn (cut, washed and blanched) produced in a commercial process from sweet corn field-treated with a fivefold application rate of maneb in the USA.

Dithiocarbamate and ETU residues were at or about limits of quantification (0.03 and 0.01 mg/kg respectively) in canned whole tomatoes, tomato puree, tomato ketchup and tomato juice commercially produced from tomatoes field-sprayed with maneb at twice the recommended application rate in the USA. It is likely that the first step, commercial washing, reduced residue

levels substantially.

No freezer storage stability studies for maneb were available. Because of the nature of the residue the Meeting agreed that the results of the storage stability studies for mancozeb would also apply to maneb.

Information on dithiocarbamate surveys of food items is included in Section 4.28 - mancozeb.

Analytical methods for maneb residues rely on conversion by acid hydrolysis to CS_2, which is then measured colorimetrically or by GLC. Information on methods for dithiocarbamates and ETU is included under mancozeb (4.28).

4.30 METIRAM (186)

TOXICOLOGY

zinc ammoniate ethylenebis(dithiocarbamate) - poly(ethylenethiuram disulphide)
Note: metiram appears to be a mixture rather than a complex.

Metiram was considered for the first time by the present Joint Meeting.

Metiram was incompletely absorbed when administered orally to rats. Elimination was primarily via the faeces, with minimal biliary excretion. Comparatively higher urinary excretion at low doses suggested that metiram may be more poorly absorbed at higher doses. The highest residual tissue levels were found in the thyroid and kidney, with slightly higher concentrations present in females than in males. Comparison of tissue residues after single or multiple doses suggested slight accumulation in the body with multiple dosing.

The metabolism of metiram has not been completely elucidated. In the rat, the predominant urinary components were polar and were identified as ethylenediamine, *N*-acetylethylenediamine, ethanolamine, oxalic acid and glycine. Major, less polar components were ethyleneurea, ethylenethiourea (ETU) and ethylenebisisothiocyanato sulphide.

Metiram was practically non-toxic upon acute oral, dermal and inhalation administration to rats. The WHO has classified metiram as unlikely to present acute hazard in normal use.

The principal target organ upon repeated dietary exposure to metiram was the thyroid.

Mice treated with metiram for three months at 0, 300, 1000, 3000 or 7500 ppm in the diet revealed minimal to slight hypertrophy and vacuolation of the thyroid follicular epithelium in both sexes at levels of 3000 ppm and above. An NOAEL of 300 ppm (equal to 84 mg/kg bw/day) was based on decreased serum T_4 levels in both sexes at levels of 1000 ppm and above.

Thirteen-week dietary administration of metiram to SD CFY rats at 0, 50, 100, 300 or 900 ppm revealed an NOAEL of 100 ppm (equal to 6 mg/kg bw/day), based on decreased serum T_4 levels and increased thyroid weights at dietary levels of 300 and 900 ppm. Slight to minimal hyperplasia of the thyroid was observed at 900 ppm. Although reduced iodine uptake by the thyroid was observed at all dietary levels, these changes were shown to be reversible following the cessation of treatment. At the lowest dietary levels of 50 and 100 ppm, the effects on iodine uptake were not correlated with changes in thyroid hormone levels or any overt morphological alterations of the thyroid gland, thus rendering the toxicological significance of this finding doubtful.

In a recently conducted three-month study with Wistar rats receiving metiram at 0, 5, 80, 320 or 960 ppm in the diet, decreased serum T_4 levels and increased thyroid weights were observed at 960 ppm. Slight evidence of anaemia was observed at 320 ppm, indicating an NOAEL of 80 ppm (equal to 5.8 mg/kg bw/day).

Other effects of treatment in the diet with metiram in the rat were manifest as hind limb paralysis with corresponding atrophy of muscle fibres. In the 13-week study with SD CFY rats, microscopic changes in muscle fibres at levels of 300 ppm (equal to 20 mg/kg bw/day) and above were still prevalent in previously treated rats after the 6-week recovery period. Muscular atrophy was observed in a long-term study in SD CD rats treated at the highest level of 320 ppm (see below). General muscle weakness/ataxia and reduced grip strength of the limbs with no histopathological consequences were observed in the 3-month study with Wistar rats fed metiram at the highest level of 960 ppm.

A 52-week study in dogs at dietary levels of 0, 30, 80, 1000 or 3000 ppm yielded an NOAEL of 80 ppm (equal to 2.5 mg/kg bw/day), based on thyroid follicular hyperplasia with increased size, thickening and weight of this organ, in conjunction with decreased serum T_4 levels at dietary levels of 1000 ppm and above. Other effects recorded at 1000 ppm and above were a dose-related increased incidence of focal hepatic lipofuscin pigment deposition, slight evidence of anaemia, diarrhoea and changes in blood biochemical parameters. A preliminary 4-week study in dogs uncovered an increased frequency of microfollicles in the thyroid, in association with colloid depletion and minimal hyperplasia in both sexes treated at the highest level of 900 ppm (equal to 41 mg/kg bw/day).

Metiram given by gavage to rhesus monkeys at dose levels of 0, 5, 15 or 75 mg/kg bw/day for a period of 26 weeks indicated an NOAEL of 5 mg/kg bw/day, based on significantly decreased serum T_3 and T_4 levels, increased thyroid weights and minimal thyroid follicular hyperplasia at 15 and 75 mg/kg bw/day. Morphological changes of the thyroid were still apparent after a 15-week recovery period. In the absence of any correlation between thyroid hormone levels and morphological alterations, no significance was attributed to fluctuations in iodine uptake by the thyroid recorded at 5 mg/kg bw/day.

Long-term dietary treatment of mice with metiram at 0, 100, 300 or 1000 ppm resulted in an NOAEL of 300 ppm (equal to 24 mg/kg bw/day), based on decreased body weights recorded at 1000 ppm. Chronic dietary administration of metiram to SD CD rats at 0, 5, 20, 80 or 320 ppm revealed muscular atrophy at 320 ppm (equal to 12 mg/kg bw/day), with an NOAEL of 80 ppm (equal to 3.1 mg/kg bw/day).

Metiram was not carcinogenic when fed to mice or rats at dietary levels of up to 1000 and 320 ppm, respectively.

A three-generation, two litter per generation reproduction study in rats treated at 0, 5, 40 or 320 ppm in the diet failed to reveal any adverse effects on reproductive parameters. The NOAEL was 40 ppm (equal to 1.8 mg/kg bw/day), based on decreased parental body weight and food consumption recorded in the F_0 and F_1 generations treated at 320 ppm.

Metiram when administered to pregnant rats at 0, 40, 80 or 160 mg/kg bw/day or rabbits at 0, 10, 40 or 120 mg/kg bw/day during critical periods of organogenesis was not teratogenic at any dose. The NOAEL for maternal toxicity in the rat was 80 mg/kg bw/day, based on decreased body-weight gain and in the rabbit the NOAEL was 10 mg/kg bw/day, based on increased abortions, decreased body weights and decreased food consumption. The NOAELs for embryo/fetotoxicity were 80 mg/kg bw/day in the rat, based on slight decreases in litter size and weight, and 40 mg/kg bw/day in the rabbit, based on decreases in mean fetal weights.

Metiram has been tested in a series of *in vitro* and *in vivo* genotoxicity assays. The Meeting concluded that metiram is not genotoxic.

The Meeting allocated an ADI of 0-0.03 mg/kg bw, based on an NOAEL of 2.5 mg/kg bw/day in the 52-week study in dogs, using a 100-fold safety factor. This ADI is supported by the NOAEL of 3.1 mg/kg bw/day observed in the long-term study in rats. This ADI served as the basis for a group ADI that was established for metiram, alone or in combination with mancozeb, maneb, and/or zineb (see Section 4.15 - dithiocarbamates).

A toxicological monograph summarizing the data that were reviewed at the present Meeting was prepared.

TOXICOLOGICAL EVALUATION

Level(s) causing no toxicological effect

Mouse: 300 ppm, equal to 24 mg/kg bw/day (88-week study)

Rat: 80 ppm, equal to 3.1 mg/kg bw/day (111-week study)
40 ppm, equal to 1.8 mg/kg bw/day (reproduction study)

Rabbit: 10 mg/kg bw/day (teratogenicity study)

Dog: 80 ppm, equal to 2.5 mg/kg bw/day (52-week study)

Monkey: 5 mg/kg bw/day (26-week study)

Estimate of acceptable daily intake for humans

0 - 0.03 mg/kg bw (group ADI with mancozeb, maneb, and zineb)

Studies which will provide information valuable in the continued evaluation of the compound

Observations in humans.

4.31 MONOCROTOPHOS (054)

TOXICOLOGY

Monocrotophos was evaluated by the Joint Meeting in 1972, 1975, and 1991. In 1991 the ADI was changed to 0-0.00005 mg/kg bw, based on an NOAEL of 0.005 mg/kg bw/day in a two-year study in rats. The Meeting identified (1) genotoxicity studies, known to exist, with commercial and purified monocrotophos and (2) historical control data on the incidence of brain malformations in rats at the laboratory that performed a recent teratogenicity study in rats, as being studies which would provide information valuable in the continued evaluation of the compound. Information relevant to these issues (new teratogenicity studies in rats and rabbits) was considered at the present Meeting. In addition, a human volunteer study that was reviewed at the 1975 Joint Meeting was re-evaluated together with all other available human data.

Monocrotophos is rapidly excreted without evidence of significant accumulation in the body.

In a new teratogenicity study in rats, no evidence of teratogenicity or embryo/fetotoxicity was observed at any doses tested (up to 2 mg/kg bw/day by gavage). Malformations of the brain were not observed. The NOAEL for maternal toxicity was 0.3 mg/kg bw/day.

Upon re-evaluation of the teratogenicity study in rats reviewed by the 1991 Joint Meeting

and of additional information provided, the Meeting concluded that the previously described brain malformations were artifacts due to incorrect tissue sampling and handling. This conclusion is also supported by the lack of a clear dose-response relationship and by the new negative teratogenicity study.

In a teratogenicity study in rabbits, monocrotophos was not teratogenic at doses up to 6 mg/kg bw/day, which was lethal to the mothers. Embryo/fetotoxicity was observed at this dose. The NOAEL for maternal toxicity was found to be 1 mg/kg bw/day.

Commercial formulations containing monocrotophos are genotoxic *in vitro*. In addition, *in vivo* results suggest that these formulations may cause chromosomal damage and sperm abnormalities in rodents. High-purity monocrotophos has not been adequately tested for genotoxicity.

Carcinogenicity studies in mice and rats evaluated by the 1991 Joint Meeting were negative.

In a human volunteer study (6 males), an oral dose of 0.0059 mg/kg bw/day for 30 days caused up to 28% plasma cholinesterase depression without erythrocyte cholinesterase depression.

The Meeting allocated an ADI of 0-0.0006 mg/kg bw on the basis of the 30-day human volunteer study with an NOAEL of 0.006 mg/kg bw/day based on the absence of erythrocyte cholinesterase inhibition, using a 10-fold safety factor.

An addendum to the toxicological monograph was prepared.

TOXICOLOGICAL EVALUATION

Level causing no toxicological effect

Mouse: <1 ppm in the diet, equivalent to <0.15 mg/kg bw/day (two-year study) (1991 JMPR)

Rat: 0.1 ppm in the diet, equivalent to 0.005 mg/kg bw/day (two-year study) (1991 JMPR)

Human: 0.006 mg/kg bw/day (30-day study)

Estimate of acceptable daily intake for humans

0-0.0006 mg/kg bw

Studies which will provide information valuable in the continued evaluation of the compound

1. Further observations in humans.

2. Genotoxicity studies with high-purity monocrotophos.

4.32 PHORATE (112)

RESIDUE AND ANALYTICAL ASPECTS

New information on use patterns and data on residues in carrots resulting from supervised trials were evaluated. The MRL proposed for carrots (0.5 mg/kg) by the 1977 Joint Meeting has been the subject of much discussion at the CCPR as it would appear that the use is limited to

the United Kingdom and Australia. GAP in the United Kingdom will result in residues that are unlikely to exceed 0.2 mg/kg. The residue data available were inadequate to permit an effective assessment of the residues likely to result from Australian GAP.

4.33 PHOSALONE (060)

TOXICOLOGY

Phosalone was previously evaluated by the Joint Meeting in 1972, when an ADI of 0-0.006 mg/kg bw/day was allocated.

After oral administration, phosalone was moderately well absorbed, 15-25% appearing in the faeces. It was extensively metabolized to phosphorothioates, phosphorodithioates and 3-methylthiomethyl-6-chlorobenzoxazolone, the last of which is subsequently metabolized ultimately to 3-methylsulphonylmethyl-6-chlorobenzoxazolone.

Pure phosalone is almost certainly not a cholinesterase inhibitor, but acquires inhibitory activity after conversion to phosalone oxon *in vivo*.

The acute oral toxicity varies with species, but is in the region of 100-200 mg/kg bw in rodents. Phosalone has been classified as moderately hazardous by WHO.

There were two short-term studies in rats which could be used to give NOAELs. In a five-week oral gavage study at doses of 0, 7.5 or 15 mg/kg bw/day, the NOAEL was 7.5 mg/kg bw/day, based on brain cholinesterase inhibition. In an eight-week study in rats using dietary concentrations of 0, 10, 100, 300, 600 or 1200 ppm, the NOAEL was 10 ppm (equal to 0.87 mg/kg bw/day), based on brain cholinesterase inhibition. It is possible that NOAELs could have been established at 100 or 300 ppm, but the dose rates for those groups were increased to 2,400 and 4,800 ppm, respectively, after 5 weeks to establish a maximum tolerated dose.

Five studies were carried out in dogs. In a one-month oral dosing study, an NOAEL could not be determined as plasma and erythrocyte cholinesterase depression were seen at the lowest dose (7.5 mg/kg bw/day). In another one-month study using dietary concentrations of 0, 12.5, 25 or 37.5 ppm, the NOAEL was at the highest level, which was equal to 0.81 mg/kg bw/day; although plasma cholinesterase depression was seen in the study, neither erythrocyte nor brain cholinesterase activity was depressed. In a 6-month study using dietary concentrations of 0, 10 or 25 ppm, although plasma and erythrocyte cholinesterase were depressed the brain enzyme was not, so the NOAEL was the highest dose (equivalent to 0.63 mg/kg bw/day). In a two-year study in beagle dogs, males and females were fed phosalone in the diet at concentrations of 0, 100, 200 or 1000 ppm. The NOAEL was 200 ppm, equivalent to 5 mg/kg bw/day, based on brain cholinesterase depression, body-weight loss and elevated alanine aminotransferase at the highest dose. In a more recent one-year study in dogs using dietary concentrations of 0, 5, 25 or 300 ppm phosalone, the NOAEL was 25 ppm (equal to 0.89 mg/kg bw/day), based on brain cholinesterase depression at 300 ppm. The overall NOAEL for dogs was considered to be 200 ppm, in view of the spacing of the doses in the most recent study.

In a lifetime carcinogenicity study in mice, phosalone was given at dietary concentrations of 0, 15, 50 or 150 ppm. The NOAEL was 150 ppm, equal to 23 mg/kg bw/day, based on the lack of depression of brain cholinesterase activity, although plasma and red blood cell cholinesterase depression were seen at this level. There was no evidence of carcinogenicity.

In a two-year study in rats using concentrations of 0, 25, 50 or 250 ppm phosalone in the diet, the NOAEL was 50 ppm, equivalent to 2.5 mg/kg bw/day, based on brain cholinesterase depression at the highest dose. In a second 2-year study in rats, dietary concentrations of 0,

5, 50 or 1000 ppm were used, the highest dose being reduced to 500 ppm later in the study. There was a statistically significant increase in the prevalence of testicular atrophy and reduction in testicular weight in both the high and mid-dose groups and a dose-response relation across all groups for both effects. The Meeting concluded that the NOAEL was ≤5 ppm, ≤0.2 mg/kg bw/day.

In a teratogenicity study in rats at doses of 0, 2, 10 or 20 mg/kg bw/day, the NOAEL was 10 mg/kg bw/day for both maternal toxicity and fetotoxicity. In a study in rabbits, using doses of 0, 2, 6 or 18 mg/kg bw/day, the NOAEL was the highest dose. In another study in rabbits, using doses of 0, 1, 10 or 20 mg/kg bw/day, the NOAEL was 10 mg/kg bw/day, based on maternal toxicity. Phosalone was not teratogenic in either the rat or rabbit.

Two multigeneration reproduction studies in rats were reviewed. In the first study, using dietary concentrations of phosalone of 25 or 50 ppm, no adverse effects were observed. The NOAEL was ≥ 50 ppm, equivalent to 2.5 mg/kg bw/day. In the second study, in which phosalone was administered at dietary concentrations of 0, 10, 50 or 400 ppm, the NOAEL was 50 ppm (equivalent to 2.5 mg/kg bw/day), based on retarded pup growth and plasma and erythrocyte cholinesterase depression.

Phosalone has been adequately tested in a series of *in vitro* and *in vivo* genotoxicity assays. The Meeting concluded that phosalone was not genotoxic.

There is no evidence that phosalone has the potential to cause delayed neuropathy.

Pralidoxime salts and obidoxime are both effective in experimental phosalone poisoning.

No human study was available from which an NOAEL could be derived.

An ADI of 0-0.001 mg/kg bw was established, based on the lowest dose (0.2 mg/kg bw/day) in the recent two-year study in rats. A 200-fold safety factor was used because of concerns that the trend for the occurrence of testicular atrophy and reduction in testis weight existed across all groups.

TOXICOLOGICAL EVALUATION

Level causing no toxicological effect

Mouse: 150 ppm, equal to 23 mg/kg bw/day (two-year study)

Rat: <5 ppm, equal to 0.2 mg/kg bw/day (two-year study)
50 ppm, equivalent to 2.5 mg/kg bw/day (multigeneration reproduction study)

Rabbit: 10 mg/kg bw/day (teratology study)

Dog: 200 ppm, equivalent to 5 mg/kg bw/day (several studies)

Estimate of acceptable daily intake for humans

0-0.001 mg/kg bw

Studies which will provide information valuable in the continued evaluation of the compound

1. Explanation of the testicular atrophy seen in the recent study in rats.

2. Observations in humans.

4.34 PROCYMIDONE (136)

RESIDUE AND ANALYTICAL ASPECTS

Procymidone was reviewed by the Joint Meeting in 1981, 1989 and 1990. The 1992 CCPR retained all MRLs at step 7B in view of the need to ensure that the residue data which were reviewed in 1981 reflected current GAP.

Residue data for common beans, cucumbers, grapes, lettuce, bulb onions and tomatoes were required together with information on current GAP.

In response to the request of the CCPR extensive information was provided by the manufacturer and some member countries on use patterns, together with some residue data from supervised field trials and monitoring.

In order to estimate maximum residue levels, the residues resulting from supervised trials published in the previous evaluations which accorded with current use patterns (GAP) were also taken into consideration.

Apples. GAP was reported from Japan and the Lebanon. Two trials reported from Japan in 1981 do not reflect GAP in either country. The previous recommendation (5 mg/kg) is withdrawn.

Cherries. GAP was reported from 6 countries. The maximum dosage rate is 0.75 kg ai/ha with a PHI of 14-28 days. A single trial in Hungary corresponds to current GAP. However the initial residues (0.8 mg/kg) are much lower than in the Australian trials (2.1-6.2 mg/kg) reflecting GAP which were reported in 1990. The previous recommendation (5 mg/kg) is replaced by 10 mg/kg.

Beans. GAP was reported from 6 countries with PHIs ranging between 2 and 21 days. Residues, reported in 1981, 1989 and 1993, deriving from corresponding national GAP (7-14 days, maximum 0.75 kg ai/ha) range from 0.1 to 0.8 mg/kg. The current GAP leads to lower residues, consequently the recommended limit is 1 mg/kg.

Cucumbers and gherkins. GAP was reported from 12 countries with PHIs ranging between 1 and 15 days. Trials reported in 1981 from Japan which reflect current GAP showed residues of 0.33-1.2 mg/kg at day 1. The previous recommendation (2 mg/kg) is maintained.

Currants. GAP was reported from one country where procymidone is used for stock treatment. Two trials reported in 1981 involved foliar applications to black currants. The residue limit established previously is not supported by current GAP, so the recommendation (10 mg/kg) is withdrawn.

Egg plants. GAP was reported from 6 countries with PHIs of 1-3 days. Residues from a trial in Poland ranged from 0.6 to 0.93 mg/kg at days 1 and 3. A trial reported from France in 1981 showed a residue level of 1.5 mg/kg at 14 days. The data are insufficient to estimate a maximum residue level, so the recommendation (2 mg/kg) is withdrawn.

Grapes. GAP was reported from 27 countries with PHIs of 1-28 days, 1-4 applications at 0.25-1.0 kg ai/ha. An extensive trial programme was conducted in seven wine-growing regions of Europe. Dosage and pre-harvest intervals were selected according to the relevant national GAP which cover the world-wide uses. Residues deriving from recommended uses ranged from 0.34 mg/kg to 4.6 mg/kg. They are in the same range as those obtained in earlier trials. The present limit (5 mg/kg) is reaffirmed and it should no longer be temporary.

Kiwifruit. GAP was reported from Italy. The trial conditions reported from New Zealand in 1981 reflect the current Italian GAP, but there was no information on the comparability of climatic conditions and cultural practices. Consequently the previous recommendation (7 mg/kg) is withdrawn.

Lettuce. GAP was reported from 16 countries with PHIs of 2-35 days. The number of applications is from 1 to 10, and the rates are between 0.28 and 2 kg ai/ha. Residues reported from France (indoor and outdoor) and Spain were in the range of 0.07 to 3.4 mg/kg 21-22 days after the last application. The residues derived from glass-house applications were about 2 to 4 times those from trials conducted outdoors. The previous recommendation (5 mg/kg) is reaffirmed.

Melons. GAP was reported from 4 countries. Trials reported in 1981 reflect the current use patterns, but no residues were reported in the whole commodity. The data base is considered inadequate for estimating a maximum residue level. The previous recommendation (1 mg/kg) is withdrawn.

Onions. GAP was reported from 14 countries with PHIs of 1-28 days. Trial conditions reported in 1981 are within the current recommended uses and lead to residues in the range of 0.01-0.14 mg/kg which support the present limit (0.2 mg/kg).

Peaches and nectarines. GAP for foliar and post-harvest applications was reported from 15 countries with PHIs of 1-14 days and maximum rates of 0.37-1.0 kg ai/ha applied 2-5 times. The trial conditions reported from Australia and New Zealand in 1981 and 1990 are in line with present use recommendations. The previous recommendation (10 mg/kg) is reaffirmed.

Peppers. GAP was reported from 11 countries on sweet, green and chilli peppers with PHIs of 1-7 days. The trials reported from Japan in 1981 reflect the current use and lead to residues up to 3.8 mg/kg one day after the last application. The present limit (5 mg/kg) is reaffirmed.

Potatoes. GAP was reported from 5 countries with PHIs of 3-35 days. In two Japanese trials, carried out in 1977 but reported in 1981 and again in 1993, the residues were 0.02, 0.03, 0.05 and 0.08 mg/kg in potatoes 19-28 days after the last application. The data base was considered inadequate to estimate a maximum residue level. The previous recommendation (0.1 mg/kg) is withdrawn.

Raspberries. GAP was reported from 3 countries with PHIs of 7-14 days. In German trials reported in 1989 residues ranged from 0.59 to 6.9 mg/kg at 14 days after applications according to current GAP. Trials in France in 1989 and in Hungary and Poland in 1992 resulted in lower residues, but the combined data support the present limit (10 mg/kg).

Rice. GAP was reported from Thailand where the application is repeated every 7-10 days. Results reported in 1981 were from samples taken 19-22 days after the last application with about 2.5 times the rate registered in Thailand. The trial conditions cannot be related to GAP, so the recommendations (rice, husked: 3 mg/kg and rice, polished: 1 mg/kg) are withdrawn.

Strawberries. GAP was reported from 27 countries with PHIs of 2-21 days and application rates of 0.23-1.0 kg ai/ha. Residues from field trials in France, Germany, The Netherlands and Poland in 1981 ranged from 0.4 to 5.1 mg/kg. Following glasshouse application in Japan, the residues were between 0.9 and 8.0 mg/kg. Residues reported from Spain in 1993 were in the range of 1.3-4.24 mg/kg. The previous recommendation (10 mg/kg) is maintained.

Sunflower seed. GAP was reported from 6 countries with PHIs of 14-42 days. Residues deriving from treatments with recommended and double rates were in the range of 0.02 to 0.12 mg/kg 14-28 days after the last application. The Meeting considered the results of a single trial leading to high residues atypical and estimated a maximum residue level of 0.2 mg/kg which

replaces the previous recommendation (2 mg/kg).

Tomatoes. GAP was reported from 25 countries with PHIs of 1-21 days and maximum rates of 0.5-1.8 kg ai/ha. Residues reported from France, Japan and New Zealand in 1981, from New Zealand in 1990 and from Italy in 1993 ranged from 0.1 to 2.1 mg/kg with a residue of 2.5 mg/kg at day 1 from a glasshouse trial in Japan. The previous recommendation (5 mg/kg) is maintained.

The fate of residues in wine processing was extensively studied. Grapes were harvested from 42 separately treated test plots and fermented into wine. The vinification procedure used for grapes from a given site was chosen to match the procedure used locally in the country or region of origin. The use of different procedures, each typical of the locale in which the grapes originated, allows a realistic estimation of residues expected in commercially-produced wine.

In addition to procymidone, 3,5-dichloroaniline (DCA), which may be formed during or just after vinification, was also determined in the wine.

When grapes were treated according to GAP, the wine contained procymidone residues between 0.04 and 0.59 mg/kg. The level of DCA ranged from <0.01 to 0.07 mg/kg in the same samples.

The results indicate that procymidone residues remaining in or on grapes after treatment show no tendency to concentrate in the wine. The average wine/grape ratio for procymidone ranged between 0.07 and 0.27, with an overall average of 0.16. DCA amounted to a maximum of 20% of the procymidone concentration in wine.

Sunflower seeds, containing residues of 0.04-0.12 mg/kg, were processed to oil. The crude and refined oil samples contained residues of 0.1-0.34 mg/kg and 0.08-0.14 mg/kg respectively. The concentration factors were between 2 and 3 for seed to crude oil, and between 1 and 2 for seed to refined oil.

A survey of procymidone residues in fresh fruits and vegetables imported by Finland gave positive results in 16 commodities. The maximum values were below the recommended limits in all cases. The commodities in which the positive results exceeded 10% were the following: broccoli 28%, cucumber 47%, pear 65%, sweet pepper 26%, strawberry 32%, tomato 15%. It is to be noted that maximum residue levels have not been estimated by previous Meetings for broccoli or pears. Furthermore, information on current GAP indicates that the compound is registered for pears only in Italy and not at all for broccoli.

4.35 PROPINEB

TOXICOLOGY

Propineb was first evaluated by the Joint Meeting in 1977, when a temporary ADI of 0-0.005 mg/kg bw/day was established. The temporary ADI was extended in 1980 and 1983. At the 1985 Joint Meeting, the temporary ADI was not extended in view of the carcinogenic response in the liver of mice to propylenethiourea (PTU) and the lack of NOAELs for thyroid effects of propineb and PTU.

Orally administered propineb in rats is rapidly absorbed and excreted largely via urine and faeces. Although there was some evidence of excretion by exhalation, the available metabolic studies (which detected PTU and propyleneurea as the main urinary metabolites together with propylenediamine and a small amount of 4-methylimidazoline) found no metabolites which could be considered as potential intermediates for degradation to CO_2. The study results indicated that a proportion of the administered dose accumulates temporarily in the thyroid.

Although most of the elimination occurred within 4 days of dosing, the half-life of elimination for the proportion remaining was relatively long. This could be due to incorporation of portions of the molecule into endogenous substances following metabolism, which would also account for the radioactivity eliminated with exhaled air.

Propineb has moderate to low acute toxicity in mice, rats, hamsters, cats and sheep. WHO has classified propineb as unlikely to present acute hazard in normal use.

The results of toxicity studies clearly indicate that propineb has a goitrogenic effect in rats, although no similar finding was noted in rabbits or dogs. In a 62-day study in male rats using dietary levels of 0, 2, 10, 50 or 250 ppm, the NOAEL was 10 ppm (equal to 0.74 mg/kg bw/day), based on changes in thyroxine concentration and increased thyroid weight at higher doses. Although in a later 63-day study using dietary levels of 0, 0.2, 0.6, 2 or 10 ppm slight hyperplasia was seen in the thyroid in 2 rats of 10 treated with 10 ppm, this finding was considered not to be a permanent adverse effect in rats.

In a comparative study of the effects of propineb, PTU, ETU, zineb and methylthiouracil on thyroid weight, propineb had only a moderate effect compared to methylthiouracil, whereas PTU had an equivalent effect to that of methylthiouracil and was somewhat stronger than ETU. These results suggest that the effects of propineb on the thyroid in rats may be caused primarily by the metabolite PTU. Dogs tolerated much higher doses of propineb, dietary administration of 3000 ppm, equivalent to 75 mg/kg bw/day, causing no adverse effects over 2 years.

In long-term studies, treatment-related alterations in tumour incidence were seen in rats and mice. In mice treated with 0, 50, 200 or 800 ppm, an increase in hepatocellular adenomas was seen only in males at the highest dose tested, but there was no increase in the incidence of hepatic carcinomas and no similar effect in females. The NOAEL was 200 ppm, equal to 26 mg/kg bw/day, based on this change in hepatic tumour incidence. In rats there were two studies, using dietary levels of 0, 1, 10, 100, 1000, 2000 or 8000 ppm in the first and 0, 5, 10, 25, 50 or 100 ppm in the second. The overall NOAEL was 50 ppm, equivalent to 2.5 mg/kg bw/day, based on increased kidney and liver weight (without histological correlation) and increased thyroid weight at 100 ppm and above. An increase in TSH-related thyroid tumours and skeletal muscle degeneration was seen in rats at dietary levels of 1000 ppm and above, but these doses were accompanied by increased mortality.

In a three-generation reproduction study in rats using dietary levels of 0, 20, 60, 200 or 600 ppm the NOAEL was 60 ppm, equivalent to 3 mg/kg bw/day, with adverse effects on maternal health and impaired reproductive performance seen at higher doses.

Teratogenicity studies indicated that propineb has teratogenic potential in rats, but no evidence of teratogenicity was seen in rabbits even in the presence of maternal toxicity. In rats, using dose levels of 0, 3, 10, 30 or 100 mg/kg bw/day, the NOAELs were 10 and 30 mg/kg bw/day for maternal and embryo/fetotoxicity, respectively, with evidence of teratogenicity at 100 mg/kg bw/day. In rabbits, using dose levels of 0, 10, 30 or 100 mg/kg bw/day, the NOAEL for maternal toxicity was 10 mg/kg bw/day, while there was no evidence of teratogenicity or embryo/fetotoxicity at 100 mg/kg bw/day, the highest dose used.

Propineb has been adequately tested in a series of genotoxicity assays from which the Meeting concluded that it is not genotoxic.

An ADI for propineb was based on the NOAEL from the short-term thyroid function study in rats (10 ppm, equal to 0.74 mg/kg bw/day) using a safety factor of 100 (see Section 4.15 - Dithiocarbamates).

A toxicological monograph summarizing the data received since the previous evaluation

and incorporating relevant studies from the previous monograph on propineb was prepared.

TOXICOLOGICAL EVALUATION

Level causing no toxicological effect

Mouse: 200 ppm in the diet, equal to 26 mg/kg bw/day (long-term study)

Rat: 10 ppm in the diet, equal to 0.74 mg/kg bw/day (62-day thyroid function study)
50 ppm, equivalent to 2.5 mg/kg bw/day (long-term study)

Dog: 3000 ppm in the diet, equivalent to 75 mg/kg bw/day (2-year study)

Estimate of acceptable daily intake for humans

0-0.007 mg/kg bw

Studies which will provide information valuable in the continued evaluation of the compound

Further observations in humans.

RESIDUE AND ANALYTICAL ASPECTS (propineb and PTU)

Propineb was evaluated in 1977, 1984 and 1985. The temporary ADI was withdrawn by the 1985 JMPR, but the CCPR maintained the Guideline Levels for propylenethiourea (PTU). The compounds are included in the CCPR periodic review programme.

Propineb is currently registered on a large number of crops in several countries around the world, but the Meeting was informed that its actual use is restricted to a few crops. The results of numerous supervised field trials and processing studies were provided by the principal manufacturer only for grapes, tomatoes, potatoes, pome fruits, onions and melons. The use of the compound will still be recommended on these crops and on bell peppers, but the use recommendations for other crops are due to be withdrawn.

The metabolism in plants has been sufficiently presented in the 1984 Evaluations. The laboratory animal metabolism studies are discussed in the Toxicological Evaluations. The metabolic pathways in plants and animals are essentially the same. Propylenediamine and 4-methylimidazoline identified in animals were present in the form of *N*-formylpropylendiamine and 2-methoxy-4-methylimidazoline, respectively. No information was available on metabolism by farm animals or on animal transfer studies.

In the supervised trials, propineb residues were determined and expressed as mg/kg CS_2, and propylenethiourea (PTU) residues were determined and expressed as mg/kg PTU throughout.

In apples and pears, the residues of propineb ranged from <0.05 mg/kg to 0.96 mg/kg 14-21 days after the last treatment. For the main metabolite, PTU, the results were in the range of <0.02 mg/kg to 0.08 mg/kg. If propineb were used alone the estimated maximum residue levels on apples and pears would be 2 mg/kg propineb and 0.1 mg/kg PTU.

In grapes, at pre-harvest intervals ranging from 49 to 69 days, residues of propineb and PTU were between <0.05 and 1.2 mg/kg, and <0.01 and 0.08 mg/kg respectively, except in

one trial where 2.1 mg/kg propineb and 0.15 mg/kg PTU were measured. Following a different application schedule, the residues of propineb were more or less in the same range. The variety of grape did not influence the residue levels. If propineb were used alone the estimated maximum residue levels on grapes would be 2 mg/kg propineb and 0.1 mg/kg PTU.

In onions, 14 days after the last treatment, no residues of propineb were found above the limit of determination (0.2 mg/kg) in Australia. PTU was not determined. In Japanese trials seven days after the last treatment the residues of propylenediamine (PDA) were <0.05 mg/kg in four samples and 0.05 and 0.08 mg/kg in two samples, while the residues of PTU were below the limit of determination (<0.01 mg/kg). Propineb was not determined. The data are not sufficient to estimate a maximum residue level for the use of propineb on onions.

In melons, 7 to 21 days after the last treatment the residues of propineb and PTU were below the limits of determination (0.01-0.2 and 0.01 mg/kg respectively) in all samples, while PDA ranged between 0.06 and 0.72 mg/kg. If propineb were used alone the estimated maximum residue levels in melons would be 0.2 mg/kg propineb and 0.05 mg/kg PTU, both levels being at or about the limit of determination.

In tomatoes treated according to German GAP, seven days after the last application residues of propineb ranged from 0.08 to 0.55 mg/kg and residues of PTU were at or below the limit of determination of 0.02 mg/kg. If propineb were used alone the estimated maximum residue levels in tomatoes would be 1 mg/kg for propineb and 0.05 mg/kg for PTU.

In potatoes no residues of propineb or PTU were found above the limits of determination (0.2 mg/kg and 0.01 mg/kg respectively) within 8 to 69 days after the last treatment. These residue trials do not completely correspond to the current registered uses but they cover present good agricultural practice as the application rate was higher. All trials showed that in spite of the great variations in pre-harvest interval no residues of propineb or the major metabolite PTU could be detected in potatoes. If propineb were used alone the estimated maximum residue levels in potatoes would be 0.2 mg/kg propineb and 0.05 mg/kg PTU (the limits of determination).

The effects of processing on the residues were extensively studied on apples, cherries, grapes, hops and tomatoes. These studies showed that the concentration of propineb residues was reduced to non-detectable (<0.02 mg/kg) in the case of apple juice and puree, wine, beer, and tomato juice and ketchup, while in cherry juice and jam the average propineb residue was 40% of that in the fruits. The residue level of PTU in processed products is primarily influenced by the level of propineb and the mode of processing. The ratio of PTU in the processed product to propineb in the raw commodity was 0.04 for apple puree, 0.003 for beer, 0.2 for cherry juice, 0.1 for cherry jam, 0.2 for must and wine, 0.1 for tomato juice and 0.2 for ketchup. The residue levels of PTU were higher in products where the processing involves extensive contact with the peel of the harvested crop as in red wine and tomato ketchup.

The freezer storage stability of the residues in samples has not been studied systematically. However the Meeting was informed that the repeated analyses of samples analysed when taken and after prolonged freezer storage did not show any difference in the residue levels. Samples were always frozen whole before storage and homogenized deep-frozen before analysis in order to eliminate decomposition of the residues. The Meeting noted that this information provided on propineb residues was consistent with the results of frozen storage stability studies on mancozeb reported under that heading. It was also considered likely that the results of frozen storage stability studies on ETU would apply to PTU.

No information was reported on PTU levels in food moving in commerce or at

consumption.

Residue analytical methods are available to determine propineb residues as CS_2, using colorimetric or GLC detection, and PTU residues by HPLC. These methods are suitable for regulatory purposes with limits of determination of 0.1-0.2 mg/kg for CS_2 and 0.05 mg/kg for PTU. The propineb residues can be qualitatively distinguished from the other dithiocarbamates by converting them to propylenediamine which can be determined by gas chromatography after derivatization.

FURTHER WORK OR INFORMATION

Desirable

1. Residue data from supervised trials on bell peppers.

2. Freezer storage stability studies on propineb and PTU residues in representative commodities.

3. Metabolism study on farm animals.

4. Residue transfer study on farm animals.

5. Monitoring data on PTU in food in commerce and consumption.

4.36 PROPYLENETHIOUREA (PTU) (150)

TOXICOLOGY

Propylenethiourea (PTU) is a plant and animal metabolite and a degradation product of propineb. Results of the toxicological evaluation of propineb indicate that the effects of propineb on the rat thyroid may be caused primarily by PTU. PTU is also of interest because it forms part of the terminal residue to which consumers of produce treated with propineb are exposed and because the levels of PTU in treated produce generally increase during food processing as the levels of propineb decrease.

Following oral administration to rats, PTU was rapidly absorbed and eliminated in the urine and faeces. Less than 0.2% of the administered dose was detected in the exhaled air and after 10 days only 1-2% remained in the body. Biliary excretion was also observed, as was evidence for enterohepatic recirculation. Distribution in body tissues was essentially uniform, with the exception of the thyroid which had about 12 times the level found in other tissues.

The results of toxicity studies clearly indicate that PTU has a goitrogenic effect in rats. In a 21-day comparative study of the effects of propineb, PTU, ETU, zineb and methylthiouracil on thyroid weight, PTU had an equivalent effect to that of methylthiouracil, and was somewhat more potent than ETU. The thyroid enlargement was partially reversible during a 28-day withdrawal period and the results suggested that the effects of propineb on the thyroid in rats may be caused primarily by the metabolite PTU. In a 63-day study in male rats, in which PTU was administered in the drinking water at levels of 0, 0.1, 0.3, 1 or 10 ppm, no consistent effects on thyroid function were seen at doses up to 10 ppm, the highest dose tested, which was equal to 1 mg/kg bw/day. In a 24-month study on thyroid function, using dietary levels of 0, 1, 10, 100 or 1000 ppm, the NOAEL (based on increased thyroid weight at higher doses) was 10 ppm in the diet, equivalent to 0.5 mg/kg bw/day.

In long-term studies, treatment-related alterations in tumour incidences were seen in rats

and mice. In a long-term study in mice, using dietary levels of 0, 1, 10, 100 or 1000 ppm, it was considered not possible to establish an NOAEL, since the incidence of liver tumours in all treated groups was higher than in the controls. However, there was evidence of a dose-response relationship and the lowest dose level (equal to 0.2 mg/kg bw/day) was considered to be a marginal effect level. Thyroid tumour incidence was not affected by treatment of mice with PTU. In a long-term study in rats, also using dietary levels of 0, 1, 10, 100 or 1000 ppm, the NOAEL was 10 ppm in the diet, equal to 0.6 mg/kg bw/day. Treatment-related thyroid tumours were seen at 1000 ppm. This dietary level was accompanied by increased mortality, while non-neoplastic thyroid changes and reduced body-weight gain were seen at 100 ppm.

A published article indicated that PTU showed teratogenic effects in rats at 45 and 90 mg/kg bw/day, doses which showed slight maternal toxicity. Results were not, however, reported in sufficient detail to fully evaluate this study. No information was available to the Meeting regarding special studies with PTU on embryo/fetotoxicity in species other than rats.

PTU is not mutagenic in bacteria and does not cause damage to mouse DNA *in vivo*. The Meeting could not reach any conclusion regarding the genotoxicity of PTU because of the limited data.

A temporary ADI for PTU was based on the marginal effect level in the long-term study in mice (1 ppm in the diet, equal to 0.2 mg/kg bw/day). The Meeting felt reassured that, in view of the metabolic conversion of propineb to PTU, the long-term study in mice with PTU identified the same target organ as the long-term study in mice with propineb. However, in view of the overall inadequacy of the toxicological data for PTU, the Meeting concluded that a 1000-fold safety factor was necessary (see also Section 4.15 - dithiocar-bamates.

A toxicological monograph summarizing the data received since the previous evaluation and incorporating relevant studies on PTU from the previous monograph on propineb was prepared.

TOXICOLOGICAL EVALUATION

Level causing no toxicological effect

Mouse: 1 ppm in the diet, equal to 0.2 mg/kg bw/day (marginal effect level in long-term study)

Rat: 10 ppm in the diet, equivalent to 0.5 mg/kg bw/day (long-term thyroid function study)
10 ppm in the diet, equal to 0.6 mg/kg bw/day (long-term study)

Estimate of temporary acceptable daily intake for humans

0-0.0002 mg/kg bw

Studies without which the determination of a full ADI is impracticable

Results to be submitted to WHO by 1998:

1. Long-term carcinogenicity study in mice, identifying an NOAEL.

2. Clarification of the genotoxic potential of PTU.

3. Clarification of the embryo/fetotoxic and teratogenic potential of PTU in rodents.

RESIDUE AND ANALYTICAL ASPECTS

See 4.35 - propineb.

4.37 PYRAZOPHOS (153)

RESIDUE AND ANALYTICAL ASPECTS

Owing to the late submission of GAP information, evaluation of the residue data for pyrazophos was postponed from the 1992 Meeting, at which an ADI was estimated and the existing Guideline Levels became MRLs. Full GAP information has now been recorded and some previously unreported data on residues resulting from supervised trials on fruits, vegetables and cereals have been evaluated, together with those included in the 1985 and 1987 reviews. In general, these additional data served only to reinforce the conclusions reached earlier. There were insufficient new data to support any recommendations on new crops, other than on barley and wheat straw.

For apples, the additional data were deemed adequate to support a recommendation to increase the MRL from 0.5 to 1 mg/kg. Data for strawberries, Brussels sprouts, cucumbers, barley and wheat supported the recommendations previously made for those crops. MRLs could also now be recommended for barley and wheat straw, based mainly on the ample trials data on wheat treatments. No additional data were received for carrots and hops,but a review of the data previously reported led to confirmation of the existing recommendations.

Data on residues in a few crops for which MRLs had not previously been recommended, namely nectarine, peach, summer squash, watermelon, egg plant, peppers, tomato, beetroot and Witloof chicory, were mostly only in summary form and, in any event, they were deemed insufficient as a basis for any MRL recommendation.

Some limited data on residue changes during the processing of barley and wheat were made available in response to previous requests. These showed that residues of pyrazophos were unlikely to be found in beer or bread produced from crops treated according to GAP. Residues in pressed apple juice and in a cooked mash from treated apples were below 0.01 mg/kg. Data on residues in animal products and the identities of plant metabolites were still not available.

FURTHER WORK OR INFORMATION

Desirable

1. Information on residues in meat and milk from cattle, meat from pigs, and meat and eggs from poultry fed on a diet containing pyrazophos.

2. Additional information on the identities and quantities of metabolites in plants after treatment with pyrazophos.

3. Full reports of the residue trials supplied in summary form by Spain and The Netherlands.

4.38 TRIAZOPHOS (143)

TOXICOLOGY

In 1991 the temporary ADI of 0-0.0002 mg/kg bw was extended in view of the uncertainty

regarding the potential for triazophos to cause delayed neurotoxicity. The 1991 Meeting also noted that previous investigations of the effects of antidotes to acute triazophos intoxication were inadequate.

Re-examination of sections of spinal cord and peripheral nerve from a 90-day study in hens (which the 1991 JMPR meeting had found difficult to interpret) revealed that lesions previously considered to be due to treatment with triazophos were in fact probably variations in background pathology and were not consistent with delayed neurotoxicity. In a new acute delayed neurotoxicity study in hens, there was no indication that treatment with triazophos was associated with any induction of delayed neurotoxicity. However the Meeting criticized the study design, in that higher doses could have been used in order to maximize the potential exposure of the nervous system to triazophos.

In an investigation of antidote treatment to triazophos intoxication in rats the expected results were obtained, a combination of atropine and oxime proving to be efficient antidotes.

The Meeting concluded that, despite shortcomings in the design of the neurotoxicity studies, the total available data indicated that triazophos does not have the potential to cause delayed neurotoxicity following dietary exposure. An ADI was allocated using the NOAEL from the human volunteer study reviewed in 1982 and 1991, using a 10-fold safety factor. This ADI was supported by the NOAEL from a 52-week study in dogs, using a 100-fold safety factor.

An addendum to the toxicological monograph was prepared.

TOXICOLOGICAL EVALUATION

Level causing no toxicological effect

Mouse: 30 ppm in the diet, equal to 4.5 mg/kg bw/day (2-year study) (1991 JMPR)

Rat: 3 ppm in the diet, equal to 0.17 mg/kg bw/day (2-year study) (1991 JMPR)
27 ppm in the diet, equal to 2-3 mg/kg bw/day (multigeneration reproduction study) (1991 JMPR)

Dog: 4 ppm in the diet, equal to 0.12 mg/kg bw/day (1-year study) (1991 JMPR)

Human: 0.0125 mg/kg bw/day (3-week study) (1982 and 1991 JMPR)

Estimate of acceptable daily intake for humans

0-0.001 mg/kg bw

Studies which will provide information valuable in the continued evaluation of the compound

Further observations in humans.

RESIDUE AND ANALYTICAL ASPECTS

Triazophos was evaluated in 1982 and several times since then, most recently in 1991. Maximum residue levels were estimated for a number of commodities and recommended as TMRLs because the ADI was temporary. At the 23rd and 24th Sessions of the CCPR (1991-92) the proposed TMRLs for citrus fruits, bananas, Brussels sprouts, head cabbages, common beans and cauliflower were held at step 7b and referred back to the JMPR. Written comments were received from France, Germany and The Netherlands on citrus fruits, bananas, Brussels sprouts and head cabbages. The manufacturer submitted new residue data from supervised trials on carrots, strawberries and soya beans.

At the same Sessions of the CCPR it was proposed to lower the limit of determination from 0.05 mg/kg to 0.01 mg/kg for residues of triazophos in cereal grains, potatoes, bulb onions and sugar beet.

The Meeting took note of the observations made by France, Germany and The Netherlands, who stated that the data base was insufficient to set residue limits for citrus fruits and bananas. The Meeting examined the data and proposed to withdraw the TMRL of 1 mg/kg for bananas, as the limit is based on residue data from only two trials. It was also difficult to link the summarized data for citrus fruits to the information on GAP in the 1983 Evaluation. The Meeting was informed that new information on GAP for triazophos and residue data from a number of trials on citrus fruits would be available in the near future, and therefore recommended that the limit for triazophos in citrus fruits should be made temporary, irrespective of the status of the ADI, until the new data were evaluated.

In 1986 the JMPR had already re-evaluated and confirmed the proposed limits for residues in Brussels sprouts and head cabbages. No new data were available. The Meeting reaffirmed the recommendation.

The Meeting considered the limit of determination of 0.05 mg/kg for triazophos in cereal grains, potatoes, bulb onions and sugar beets. The residues in these crops reported to the 1983 and 1990 Meetings were below the limit of determination, which ranged from 0.001 to 0.07 mg/kg. Although most residues were below 0.02 mg/kg, the Meeting proposed to maintain the limit of determination of 0.05 mg/kg as a realistic limit of determination for the purpose of enforcement.

Supervised trials on carrots were carried out in the UK, all except two in accordance with recommended use. The highest residue in samples from two trials 28 days after the last treatment was 0.23 mg/kg. In addition to the trials a survey was carried out in the UK in which many samples of carrots with a known pesticide treatment history were analysed, all applications being in accordance with label recommendations. Residues were from <0.02 mg/kg to 0.94 mg/kg with a mean value of 0.17 mg/kg, and the 90th percentile was approximately 0.4 mg/kg. The Meeting recommended a maximum residue limit of 0.5 mg/kg for carrots.

Supervised trials were also carried out on strawberries in Germany. Residues were very low and except for the PHI of 11 days all residues were below 0.05 mg/kg. No information on GAP on in Germany was available, but GAP in The Netherlands would give rise to even lower residues. The Meeting estimated a maximum residue level at the limit of determination for strawberries (0.05 mg/kg).

Residues in seeds from trials on soya beans in Brazil with application in accordance with Brazilian GAP were also below the limit of determination, and a maximum residue limit was proposed at the limit of determination (0.05 mg/kg).

FURTHER WORK OR INFORMATION

Required (by 1994).

Information on GAP for triazophos on citrus fruits and residue data from trials in accordance with GAP.

4.39 ZINEB (dithiocarbamates, 105)

TOXICOLOGY

Zineb was previously evaluated by the Joint Meeting in 1963, 1965, 1967, 1970, 1974, 1977 and 1980. An ADI of 0-0.05 mg/kg bw, of which not more than 0.002 mg/kg bw may be present as ETU, was allocated at the 1980 Meeting for zineb or the sum of maneb, mancozeb, and zineb. Little new information on zineb has become available since the previous evaluation.

Zineb was poorly absorbed when administered orally to mice. The extent to which enterohepatic circulation may have been involved in the species studied, namely the mouse, rat and marmoset, has not been investigated. Absorption, as measured by urinary excretion in the rat, was highly variable and the reasons for this variability are not known. In the marmoset the majority of the administered dose was excreted in the urine, with lesser amounts in the faeces. The principal routes of excretion were via the faeces and urine, with negligible amounts in expired CO_2.

The metabolic pathway of zineb has not been clearly delineated. Characterization of urinary components in the mouse, rat and marmoset have revealed the presence of ethylenethiourea (ETU), ethyleneurea and polar components.

Zineb was practically non-toxic upon acute oral administration to rats and guinea pigs, when given subcutaneously to rats, or when given by intraperitoneal injection to mice. WHO has classified zineb as unlikely to present acute hazard in normal use.

Dietary administration of zineb to rats for six weeks at 0, 500 or 5000 ppm indicated an NOAEL of 500 ppm (equivalent to 25 mg/kg bw/day), based on morphological changes of the thyroid gland and reduced uptake of ^{124}iodine at 5000 ppm. Rats treated with zineb orally by gavage for four weeks (5 days/week) at 0, 15, 60, 250 or 1000 mg/kg bw/day exhibited slight hyperplasia of the thyroid at a dose level of 1000 mg/kg bw/day, resulting in an NOAEL of 250 mg/kg bw/day. There were no significant changes in the thyroid noted in rats previously treated at 1000 mg/kg bw/day following a two week recovery period. Rats administered zineb at doses of 490 or 2450 mg/kg bw, twice weekly for four months, developed paresis of the hind limbs, which progressed to complete paralysis. Similar treatment-related effects on the hind limbs were not confirmed in a two-year study in rats (see below) at the highest dietary level of 10,000 ppm, equivalent to 500 mg/kg bw/day.

In a limited one-year study, dogs treated with zineb at dietary levels of 20, 2000 or 10,000 ppm revealed thyroid hyperplasia at 10,000 ppm, resulting in an NOAEL of 2000 ppm (equivalent to 50 mg/kg bw/day).

Zineb was not carcinogenic when given to mice at 460 mg/kg bw/day from postnatal day 7 until weaning followed thereafter by dietary administration of 1300 ppm until 18 months of age. A two-year study in which rats (10/sex/group) were fed zineb at dietary levels of 500, 1000, 2500, 5000 or 10,000 ppm revealed goitrogenic effects at all doses. Treatment-related effects were manifest at or above 1000 ppm as renal congestion, nephritis and nephrosis, increased mortality and diminished growth rate. There was no evidence of carcinogenic potential. It

should be recognized, however, that neither of these long-term studies was judged to have adequately studied the carcinogenic potential of zineb.

Treatment of rats with zineb at doses of 50 to 960 mg/kg bw/day suggested adverse effects on reproduction, depicted as sterility, decreased fertility and resorption of fetuses. From the limited data available, a dose level of 50 mg/kg bw/day appeared to be without significant adverse reproductive effect.

Treatment of mice with zineb at dose levels of 0, 200, 630 or 2000 mg/kg bw/day during critical periods of organogenesis did not induce any maternal toxicity, embryo/fetotoxicity or teratogenicity at any of the dose levels studied.

An oral teratogenicity study in rats at doses of 0, 200, 630 or 2000 mg/kg bw/day revealed that zineb was teratogenic at the maternally toxic dose level of 2000 mg/kg bw/day. Treatment with zineb resulted in a significant increase in hydrocephalus, skeletal anomalies (enlarged frontal and occipital fontanelles, split centra and incomplete ossification of the supraocciput), and a higher incidence of abnormalities of the tail.

Zineb has been adequately tested in a series of *in vivo* genotoxicity assays. Positive responses were obtained in a *Drosophila* study and in a study for chromosomal aberrations in cultured mammalian cells. Other assays were negative. The Meeting concluded that zineb was not likely to be a significant genotoxic hazard.

The Meeting concluded that the toxicological data specifically generated for zineb were inadequate to estimate an ADI. However, because of the similarity of the chemical structure of zineb to that of the other ethylenebis(dithiocarbamate)s (EBDCs), the comparable toxicological profile of the EBDCs based on the toxic effects of ETU, and the fact that parent EBDC residues cannot be differentiated using presently-available regulatory analytical methods, zineb was included in the group ADI of 0-0.03 mg/kg bw for the EBDC group evaluated at this Meeting (mancozeb, maneb, metiram). (See also Section 4.15 - dithiocarbamates).

A toxicological monograph was prepared summarizing the data that were reviewed at the present Meeting, including relevant data that were summarized in the previous monograph and monograph addenda on zineb.

TOXICOLOGICAL EVALUATION

Level causing no toxicological effect

Mouse: 460 mg/kg bw/day (18-month study: did not adequately study long-term toxicity or carcinogenic potential)
2000 mg/kg bw/day (teratogenicity study)

Rat: 500 ppm, equivalent to 25 mg/kg bw/day (six-week study)
<500 ppm, equivalent to <25 mg/kg bw/day (two-year study: did not adequately study long-term toxicity or carcinogenic potential)
50 mg/kg bw/day (reproduction study: did not adequately study potential for adverse effects on reproduction)
630 mg/kg bw/day (teratogenicity study)

Dog: 2000 ppm, equivalent to 50 mg/kg bw/day (one-year study: did not adequately study potential for toxicity in a non-rodent species)

Estimate of acceptable daily intake for humans

0-0.03 mg/kg bw (Group ADI with mancozeb, maneb and metiram)

Studies which will provide information valuable in the continued evaluation of the compound.

1. Further elucidation of absorption/distribution/excretion patterns and metabolic pathways.
2. Reproduction study.
3. Long-term toxicity/carcinogenicity studies in two appropriate species.
4. Short-term repeated exposure studies in a rodent and non-rodent species to determine NOAELs with respect to effects on the thyroid and potential for neurotoxicity.
5. Observations in humans.

5. RECOMMENDATIONS

5.1 In the interests of public health and agriculture and in view of the needs of the Codex Committee on Pesticide Residues, the Meeting recommended that Joint Meetings on Pesticide Residues should continue to be held annually.

5.2 The Meeting recommended (Section 2.2) that:

(1) the Codex working paper, "Risk Assessment Procedures Used by the Codex Alimentarius Commission and its Subsidiary and Advisory bodies" (ALINORM 93/37), should not be published or distributed further in its present form,

(2) a revised paper be prepared that would take into account new developments in the dynamic field of risk assessment and recent Codex developments, and

(3) the revised paper should be circulated to national and international organisations to give them the opportunity to comment before publication or wider distribution.

5.3 The Meeting recommended (Section 2.3) that governments conduct appropriate dietary surveys, especially for population groups of special concern such as infants and children.

5.4 Regarding concomitant pesticide and veterinary uses of chemicals, the Meeting recommended (Section 2.5) that:

(1) the Codex Alimentarius Commission and the FAO/WHO Secretariats of the expert committees develop procedures to ensure consistency and appropriate exchange of information among the involved committees,

(2) the Joint Secretaries of the JMPR and JECFA obtain and make available to their respective meetings, and in advance to data reviewers, copies of pertinent evaluations,

(3) the Codex Secretariat arrange distribution of the pertinent evaluations to country Codex contact points, and

(4) for periodic review compounds the submission to the FAO Panel should include full information on approved veterinary uses, data from trials in accordance with those uses, and metabolism data for plants and animals.

5.5 Regarding the mode of action, mechanism of toxicity and toxicological evaluation of pesticides, the Meeting recommended (Section 2.10) that, where known, information on the mode of pesticidal action as well as on the mechanism of toxicity in non-target species should be made available to the Joint Meeting.

5.6 The Meetng recommended (Section 3.2) that the development of additional regulatory analytical methods to differentiate propineb residues from those arising from the use of ethylenebis(dithiocarbamate)s (EBDCs) should be aggressively pursued.

5.7 With the expectation that residues of DDT as an environmental contaminant will decrease as production ceases, the Meeting recommended (Section 4.11) that monitoring data should again be evaluated in 1998 with a view to the possible lowering of ERLs.

6. FUTURE WORK

The following items should be considered at the 1994 or 1995 Meeting. Compounds recommended for priority attention by the 25th or earlier Sessions of the CCPR which have not yet been evaluated are marked with an asterisk (*), and compounds scheduled for re-evaluation in the CCPR periodic review programme with a double asterisk.

6.1 1994 Meeting (tentative)

Toxicological Evaluation

Abamectin
Azocyclotin
Carbofuran
Chlorfenvinphos **
Chlormequat **
Clethodim *
Cyhexatin
Fenpropimorph *
Parathion **
Parathion-methyl **
Phorate
Phosmet **
Tebuconazole *
Tecnazene **
Teflubenzuron *
Tolclofos-methyl *

Residue Evaluation

Abamectin
Acephate
Aldicarb **
Bentazone
Captan
Chlorfenvinphos **
Chlormequat **
Clethodim *
Diazinon
Disulfoton
Fenpropimorph *
Glufosinate-ammonium
Hexythiazox
Imazalil
Methamidophos
Metiram *
Monocrotophos
Parathion-methyl **
2-Phenylphenol **
Phosalone **
Phosmet **
Pirimiphos-methyl
Profenofos
Pyrazophos
Tebuconazole *
Tecnazene **
Teflubenzuron *
Thiram **
Tolclofos-methyl *
Triazophos

6.2 1995 Meeting (tentative)

Toxicological Evaluation

Benomyl **
Captan
Carbendazim **
Cartap **
Chlorpropham *
Fenarimol *
Fenpyroximate *
Fenthion **
Folpet
Haloxyfop *
Malathion **
Piperonyl butoxide **
Quintozene **
Thiometon **
Thiophanate-methyl **
Trichlorfon **
Vinclozolin

Residue Evaluation

Buprofezin
Cartap **
Chlorpropham *
Fenarimol *
Fenpyroximate *
Fenthion **
Folpet
Haloxyfop *
Malathion **
Parathion
Quintozene **
Thiometon **
Trichlorfon **

7. REFERENCES

PREVIOUS FAO AND WHO DOCUMENTS

1. FAO/WHO. 1962 Principles governing consumer safety in relation to pesticide residues. Report of a meeting of a WHO Expert Committee on Pesticide Residues held jointly with the FAO Panel of Experts on the Use of Pesticides in Agriculture. FAO Plant Production and Protection Division Report, No. PL/1961/11; WHO Technical Report Series, No. 240.

2. FAO/WHO. 1964 Evaluations of the toxicity of pesticide residues in food. Report of a Joint Meeting of the FAO Committee on Pesticides in Agriculture and the WHO Expert Committee on Pesticide Residues. FAO Meeting Report, No. PL/1963/13; WHO/Food Add./23.

3. FAO/WHO. 1965a Evaluations of the toxicity of pesticide residues in food. Report of the Second Joint Meeting of the FAO Committee on Pesticides in Agriculture and the WHO Expert Committee on Pesticide Residues. FAO Meeting Report, No. PL/1965/10; WHO/Food Add./26.65.

4. FAO/WHO. 1965b Evaluations of the toxicity of pesticide residues in food. FAO Meeting Report, No. PL/1965/10/1; WHO/Food Add./27.65.

5. FAO/WHO. 1965c Evaluation of the hazards to consumers resulting from the use of fumigants in the protection of food. FAO Meeting Report, No. PL/1965/10/2; WHO/Food Add./28.65.

6. FAO/WHO. 1967a Pesticide residues in food. Joint report of the FAO Working Party on Pesticide Residues and the WHO Expert Committee on Pesticide Residues. FAO Agricultural Studies, No. 73; WHO Technical Report Series, No. 370.

7. FAO/WHO. 1967b Evaluation of some pesticide residues in food. FAO/PL:CP/15; WHO/Food Add./67.32.

8. FAO/WHO. 1968a Pesticide residues. Report of the 1967 Joint Meeting of the FAO Working Party and the WHO Expert Committee. FAO Meeting Report, No. PL:1967/M/11; WHO Technical Report Series, No. 391.

9. FAO/WHO. 1968b 1967 Evaluations of some pesticide residues in food. FAO/PL:1967/M/11/1; WHO/Food Add./68.30.

10. FAO/WHO. 1969a Pesticide residues in food. Report of the 1968 Joint Meeting of the FAO Working Party of experts on Pesticide Residues and the WHO Expert Committee on Pesticide Residues. FAO Agricul-tural Studies, No. 78; WHO Technical Report Series, No. 417.

11. FAO/WHO. 1969b 1968 Evaluation of some pesticide residues in food. FAO/PL:1968/M/9/1; WHO/Food Add./69.35.

12. FAO/WHO. 1970a Pesticide residues in food. Report of the 1969 Joint Meeting of the FAO Working Party of experts on Pesticide Residues and the WHO Expert Committee on Pesticide Residues. FAO Agricultural Studies, No. 84; WHO Technical Report Series, No. 458.

13. FAO/WHO. 1970b 1969 Evaluation of some pesticide residues in food. FAO/PL:1969/M/17/1; WHO/Food Add./70.38

14. FAO/WHO. 1971a Pesticide residues in food. Report of the 1970 Joint Meeting of the FAO Working Party of experts on Pesticide Residues and the WHO Expert Committee on Pesticide Residues. FAO Agricultural Studies, No. 87; WHO Technical Report Series, No. 474.

15. FAO/WHO. 1971b 1970 Evaluation of some pesticide residues in food. AGP:1970/M/12/1; WHO/Food Add./71.42.

16. FAO/WHO. 1972a Pesticide residues in food. Report of the 1971 Joint Meeting of the FAO Working Party of experts on Pesticide Residues and the WHO Expert Committee on Pesticide Residues. FAO Agricultural Studies, No. 88; WHO Technical Report Series, No. 502.

17. FAO/WHO. 1972b 1971 Evaluation of some pesticide residues in food. AGP:1971/M/9/1; WHO Pesticide Residues Series, No. 1.

18. FAO/WHO. 1973a Pesticide residues in food. Report of the 1972 Joint Meeting of the FAO Working Party of experts on Pesticide Residues and the WHO Expert Committee on Pesticide Residues. FAO Agricultural Studies, No. 90; WHO Technical Report Series, No. 525.

19. FAO/WHO. 1973b 1972 Evaluation of some pesticide residues in food. AGP:1972/M/9/1; WHO Pesticide Residues Series, No. 2.

20. FAO/WHO. 1974a Pesticide residues in food. Report of the 1973 Joint Meeting of the FAO Working Party of experts on Pesticide Residues and the WHO Expert Committee on Pesticide Residues. FAO Agricultural Studies, No. 92; WHO Technical Report Series, No. 545.

21. FAO/WHO. 1974b 1973 Evaluation of some pesticide residues in food. FAO/AGP/1973/M/9/1; WHO Pesticide Residues Series, No.3.

22. FAO/WHO. 1975a Pesticide residues in food. Report of the 1974 Joint Meeting of the FAO Working Party of experts on Pesticide Residues and the WHO Expert Committee on Pesticide Residues. FAO Agricultural Studies, No. 97; WHO Technical Report Series, No. 574.

23. FAO/WHO. 1975b 1974 Evaluation of some pesticide residues in food. FAO/AGP/1974/M/9/11; WHO Pesticide Residues Series, No.4.

24. FAO/WHO. 1976a Pesticide residues in food. Report of the 1975 Joint Meeting of the FAO Working Party of experts on Pesticide Residues and the WHO Expert Committee on Pesticide Residues. FAO Plant Production and Protection Series, No.1; WHO Technical Report Series, No. 592.

25. FAO/WHO. 1976b 1975 Evaluation of some pesticide residues in food. AGP:1975/M/13; WHO Pesticide Residues Series, No. 5.

26. FAO/WHO. 1977a Pesticide residues in food. Report of the 1976 Joint Meeting of the FAO Panel of Experts on Pesticide Residues and the Environment and the WHO Expert Group on Pesticide Residues.
FAO Food and Nutrition Series, No. 9; FAO Plant Production and Protection Series, No. 8; WHO Technical Report Series, No. 612.

27. FAO/WHO. 1977b 1976 Evaluation of some pesticide residues in food.
AGP:1976/M/14.

28. FAO/WHO. 1978a Pesticide residues in food - 1977. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues and the Environment and the WHO Expert Group on Pesticide Residues.
FAO Plant Production and Protection Paper 10 Rev.

29. FAO/WHO. 1978b Pesticide residues in food: 1977 evaluations.
FAO Plant Production and Protection Paper 10 Sup.

30. FAO/WHO. 1979a Pesticide residues in food - 1978. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues and the Environment and the WHO Expert Group on Pesticide Residues.
FAO Plant Production and Protection Paper 15.

31. FAO/WHO. 1979b Pesticide residues in food: 1978 evaluations.
FAO Plant Production and Protection Paper 15 Sup.

32. FAO/WHO. 1980a Pesticide residues in food - 1979. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 20.

33. FAO/WHO. 1980b Pesticide residues in food: 1979 evaluations.
FAO Plant Production and Protection Paper 20 Sup.

34. FAO/WHO. 1981a Pesticide residues in food - 1980. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 26.

35. FAO/WHO. 1981b Pesticide residues in food: 1980 evaluations.
FAO Plant Production and Protection Paper 26 Sup.

36. FAO/WHO. 1982a Pesticide residues in food - 1981. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 37.

37. FAO/WHO. 1982b Pesticide residues in food: 1981 evaluations.
FAO Plant Production and Protection Paper 42.

38. FAO/WHO. 1983a Pesticide residues in food - 1982. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 46.

39. FAO/WHO. Pesticide residues in food: 1982 evaluations.

1983b FAO Plant Production and Protection Paper 49.

40. FAO/WHO. 1984 Pesticide residues in food - 1983. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 56.

41. FAO/WHO. 1985a Pesticide residues in food: 1983 evaluations. FAO Plant Production and Protection Paper 61.

42. FAO/WHO. 1985b Pesticide residues in food - 1984. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 62.

43. FAO/WHO. 1985c Pesticide residues in food: 1984 evaluations. FAO Plant Production and Protection Paper 67.

44. FAO/WHO. 1986a Pesticide residues in food - 1985. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 68.

45. FAO/WHO. 1986b Pesticide residues in food: 1985 evaluations. Part I - Residues. FAO Plant Production and Protection Paper 72/1.

46. FAO/WHO. 1986c Pesticide residues in food: 1985 evaluations. Part II - Toxicology. FAO Plant Production and Protection Paper 72/2.

47. FAO/WHO. 1986d Pesticide residues in food - 1986. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 77.

48. FAO/WHO. 1986e Pesticide residues in food: 1986 evaluations. Part I - Residues. FAO Plant Production and Protection Paper 78.

49. FAO/WHO. 1987a Pesticide residues in food: 1986 evaluations. Part II - Toxicology. FAO Plant Production and Protection Paper 78/2.

50. FAO/WHO. 1987b Pesticide residues in food - 1987. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 84.

51. FAO/WHO. 1988a Pesticide residues in food: 1987 evaluations. Part I - Residues. FAO Plant Production and Protection Paper 86/1.

52. FAO/WHO. 1988b Pesticide residues in food: 1987 evaluations. Part II - Toxicology. FAO Plant Production and Protection Paper 86/2.

53. FAO/WHO. 1988c Pesticide residues in food - 1988. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 92.

54. FAO/WHO. 1988d Pesticide residues in food: 1988 evaluations. Part I - Residues. FAO Plant Production and Protection Paper 93/1.

55. FAO/WHO. 1989a Pesticide residues in food: 1988 evaluations. Part II - Toxicology. FAO Plant Production and Protection Paper 93/2.

56. FAO/WHO. 1989b Pesticide residues in food - 1989. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 99.

57. FAO/WHO. 1990a Pesticide residues in food: 1989 evaluations. Part I - Residues. FAO Plant Production and Protection Paper 100.

58. FAO/WHO. 1990b Pesticide residues in food: 1989 evaluations. Part II - Toxicology. FAO Plant Production and Protection Paper 100/2.

59. FAO/WHO. 1990c Pesticide residues in food - 1990. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 102.

60. FAO/WHO. 1991a Pesticide residues in food: 1990 evaluations. Part I - Residues. FAO Plant Production and Protection Paper 103/1.

61. FAO/WHO. 1991b Pesticide residues in food: 1990 evaluations - Toxicology. WHO/PCS/91.47.

62. FAO/WHO. 1991c Pesticide residues in food - 1991. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 111.

63. FAO/WHO. 1991d Pesticide residues in food: 1991 evaluations. Part I - Residues. FAO Plant Production and Protection Paper 113/1.

64. FAO/WHO. 1992 Pesticide residues in food: 1991 evaluations - Part II - Toxicology. WHO/PCS/92.52.

65. FAO/WHO. 1993a Pesticide residues in food - 1992. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 116.

66. FAO/WHO. 1993b Pesticide residues in food: 1992 evaluations. Part I - Residues. FAO Plant Production and Protection Paper 118.

67. FAO/WHO. 1993c Pesticide residues in food: 1992 evaluations - Part II - Toxicology. WHO/PCS/93.34.

CORRECTIONS TO REPORT OF 1992 JMPR

Additions and changes are shown **bold**. Minor typographical errors are not included.

P.13, para 4, l.5: Change "...were 0.05 mg/kg bw/day in the teratogenicity study in rats" to **"...was 0.05 mg/kg bw/day in the teratogenicity study in mice (CF1 strain)**".

P.19, para 4, ll.5&6: Change "4-12 mg/l" to "**4-12 µg/l**" and "400 mg/l" to "**400 µg/l**".

P.20, Entry 8: Under the heading "dosages", **underline 1.67** instead of 0.47.
Under the heading "NOAEL", change 0.12 to **0.47**.

P.29, last para, l.2: Change "1970 Joint Meeting" to "**1974 Joint Meeting**"

P.31, "Level causing no toxicological effect, mouse":
Change "...reviewed by 1990 JMPR" to"...**reviewed by 1987 JMPR**".

P.46, para 3, l.7: Change "1 mg/kg bw/day" to "**10 mg/kg bw/day**"

P.63, "Level causing no toxicological effect":
Move "dog" to the last line, **before "100 ppm, equal to..."**

P.78, para 2, l.3: Change "10 mg/kg bw/day" to "**2 mg/kg bw/day**".

P.79, "Level causing no toxicological effect, dog":
Change "10 mg/kg bw/day" to "**2 mg/kg bw/day**".

P.104, para 7, l.1: Change - 1977 to - 197**8**

P.105, para 9, l.2: Change 1986b to 1986**e**

P.106, para 5, l.2: Change 1990d to 1990**c**

P.106, para 9, l.3: Change 1113/1 to **113/1**

Annex I, p.123. In the new MRL for triadimenol in Milks, change 0.0^{*1,2} to 0.0**5***^{1,2}. (The error has been corrected in the evaluations.)

Annex III, p.134. In para 1, line 1, change 1966-91 to 1966-9**2**.

p.138. Insert in alphabetical position:

FAO Panel of JMPR, Manual on preparation of documents by 92-2.9

p.140. Under **Maximum residue level(s)**
Estimation of, add the ref. shown:
FAO Guide on.....91-2.5**; 92-2.7**

p.143. Under **Pesticide residue(s):**
Data on:
Evaluation of:, add the ref. shown:
FAO Guide on.....91-2.5**; 92-2.7**

ANNEX I

ACCEPTABLE DAILY INTAKES AND RESIDUE LIMITS PROPOSED AT THE 1993 MEETING

The table of recommendations includes maximum Acceptable Daily Intakes (ADIs) and Maximum Residue Limits (MRLs). It should be noted that MRLs include draft MRLS and Codex MRLs (CXLs). The MRLs recommended by the JMPR on the basis of its estimates of maximum residue levels enter the Codex procedure as draft MRLs. They become Codex MRLs when they have passed through the procedure and have been adopted by the Codex Alimentarius Commission.

In general, the recommended MRLs listed for compounds which have been reviewed previously are additional to, or amend, those recorded in Annexes to the reports of earlier meetings. For compounds re-evaluated in the CCPR periodic review programme however, both new and previous recommendations are listed because such re-evaluations are regarded as replacing the original evaluation rather than supplementing it.

Limits recommended at meetings from 1965 to 1977 inclusive are summarized in document FAO/WHO 1978c.

Some ADIs are temporary: this is indicated by the letter T and the year in which re-evaluation is scheduled in parenthesis below the ADI. All recommended MRLs for compounds with temporary ADIs are necessarily temporary, but some recommendations are designated as temporary (TMRLs) until required information has been provided and evaluated, irrespective of the status of the ADI. Such recommendations are followed by the letter T in the table. (See also the list of qualifications and abbreviations below.)

The following qualifications and abbreviations are used.

*	At or about the limit of determination
E	Extraneous Residue Limit (ERL).
F (following recommendations for milk)	The residue is fat-soluble and MRLs for milk and milk products are derived as explained in the introduction to Part 2 of the Guide to Codex Maximum Limits for Pesticide Residues and to Volume II of the Codex Alimentarius.
(fat) (following recommendations for neat)	The recommendation applies to the fat of meat.
Po	The recommendation accommodates post-harvest treatment of the commodity.
PoP (following recommendations for processed foods (classes D and E in the Codex Classification)	The recommendation accommodates post-harvest treatment of the primary food commodity.
T (following ADIs)	The ADI is temporary, and due for re-evaluation in the year indicated.
T (following MRLs)	The MRL is temporary, irrespective of the

status of the ADI, until required information has been provided and evaluated.

V (following recommendations for commodities of animal origin)	The recommendation accommodates veterinary uses.
W (in place of an MRL)	The previous recommendation is withdrawn.

If a recommended MRL is an amendment, the previous value is also recorded. The absence of a figure in the "Previous" column indicates that the recommendation is the first for the commodity or group concerned.

The table includes the Codex Classification Numbers (CCNs) of both the compounds and the commodities listed, to facilitate reference to the Guide to Codex Maximum Limits for Pesticide Residues.

Commodities are listed in alphabetical order. This is a change from previous practice where commodities were listed in the order of the "Types" in the Codex Classification of Foods and Animal Feeds, and in alphabetical order within each Type.

The change has been made to facilitate checking and comparison with the CCPR Tables of MRLs, which are in alphabetical order.

ACCEPTABLE DAILY INTAKES (ADIs) AND MAXIMUM RESIDUE LIMITS (MRLs)

Pesticide (Codex Ref. No.)	Max. ADI (mg/ kg bw)	Commodity		Recommended MRL or ERL (mg/kg)	
		CCN	Name	New	Previous
Aldicarb (117)	0.003	VB 0402	Brussels sprouts	0.1	0.05 T
		Residue:	sum of aldicarb, its sulphoxide and its sulphone, expressed as aldicarb		
Amitrole [1] (079)	0.0005 T (1997)	Note	TADI increased from 0.00003 mg/kg bw, conditional		
Azinphos-methyl (002)	0.005	AS 0654	Wheat straw and fodder, dry	W	1 T
		Residue:	azinphos-methyl		
Benalaxyl (155)	0.05	VR 0589	Potato	0.02*	0.01*
		Residue:	benalaxyl		
Bromopropylate [1] (070)	0.03	FP 0226	Apple	W	5
		FI 0327	Banana	W	5
		VP 0526	Common bean (pods and/or immature seeds)	3	-
		FS 0013	Cherries	W	5
		FC 0001	Citrus fruits	2	5
		SO 0691	Cotton seed	W	1
		VC 0424	Cucumber	0.5	-
		FB 0269	Grapes	2	5
		DH 1100	Hops, dry	W	5
		VC 0046	Melons, except Watermelon	0.5	-
		FS 0245	Nectarine	W	5
		FS 0247	Peach	W	5
		FP 0230	Pear	W	5
		FS 0014	Plums (including prunes)	2	5
		FP 0009	Pome fruits	2	-
		VC 0431	Squash, Summer	0.5	-
		FB 0275	Strawberry	2	5
		DT 1114	Tea, Green, Black	W	5
			Vegetables	W	1
		Residue:	bromopropylate		
		Note	ADI increased from 0.008 mg/kg bw		
Carbofuran (096)	0.01	FC 0001	Citrus fruits	W	2 T
		Residue:	sum of carbofuran and 3-hydroxycarbofuran		

[1] Re-evaluation in periodic review programme

Pesticide (Codex Ref. No.)	Max. ADI (mg/ kg bw)	Commodity		Recommended MRL or ERL (mg/kg)	
		CCN	Name	New	Previous
Carbosulfan (145)	0.01	FC 0001	Citrus fruits	W	2 T
		Residue:	carbosulfan		
Chlorothalonil [1] (081)	0.03	FI 0327	Banana	W	0.2
		GC 0640	Barley	0.1	0.2 (cereals)
		AS 0640	Barley straw and fodder, dry	20	-
		FB 0264	Blackberries	W	10
		VB 0400	Broccoli	W	5
		VB 0402	Brussels sprouts	5	5
		VB 0041	Cabbages, Head	1	5
		VR 0577	Carrot	1	1
		VB 0404	Cauliflower	1	5
		VS 0624	Celery	10	15
		GC 0080	Cereal grains	W	0.2
		FS 0013	Cherries	0.5	10
		FC 0001	Citrus fruits	W	5
		VP 0526	Common bean (pods and/or immature seeds)	5	5
		FB 0265	Cranberry	5	5
		VC 0424	Cucumber	5	5
		FB 0021	Currants, Black, Red, White	W	25
		VL 0476	Endive	W	10
		FB 0269	Grapes	0.5	10
		VL 0480	Kale	W	10
		VL 0482	Lettuce, Head	W	10
		VD 0534	Lima bean (dry)	W	0.5
		VC 0046	Melons, except Watermelon	2	5
		VA 0385	Onion, Bulb	0.5	5
		FS 0247	Peach	1	25
		SO 0697	Peanut	0.05	0.1
		SO 0703	Peanut, whole	W	0.5
		VO 0051	Peppers	W	10
		VR 0589	Potato	0.2	0.1
		VC 0429	Pumpkins	W	5
		FB 0272	Raspberries, Red, Black	W	10
		VC 0431	Squash, Summer	5	5
		VR 0596	Sugar beet	0.2	1
		AV 0596	Sugar beet leaves or tops	20	-
		VO 0447	Sweet corn (corn-on the-cob)	W	1
		VO 0448	Tomato	5	5
		GC 0654	Wheat	0.1	0.2 (cereals)
		AS 0654	Wheat straw and fodder, dry	20	-
		VC 0433	Winter squash	5	5
		VS 0469	Witloof chicory (sprouts)	W	10
Chlorpyrifos-methyl (090)	0.01	SO 0495	Rape seed	W	10 Po T
		Residue:	chlorpyrifos-methyl		
Cycloxydim (179)	0.07	VD 0071	Beans (dry)	2	-
		VB 0040	Brassica vegetables	2	-
		VR 0577	Carrot	0.5	-

[1] Re-evaluation in periodic review programme

		VP 0526	Common bean (pods and/or immature seeds)	1	-
Cycloxydim (contd.)		FB 0269	Grapes	0.5	-
		VA 0384	Leek	0.2	-
		VL 0482	Lettuce, Head	0.2	-
		VL 0483	Lettuce, Leaf	0.2	-
		VP 0063	Peas	1	-
		VP 0064	Peas, shelled	2	-
		VR 0589	Potato	2	-
		SO 0495	Rape seed	2	-
		VD 0541	Soya bean (dry)	2	-
		FB 0275	Strawberry	0.5	-
		VR 0596	Sugar beet	0.2	-
		AV 0596	Sugar beet tops or leaves	1	-
		Residue: sum of 3-thian-3-ylglutaric acid (TME) and 3-hydroxy-3-thian-3-expressed as cycloxydim			
DDT (021)	0.02	VR 0577	Carrots	0.2 E	-
		PE 0112	Eggs	0.1 E	0.5 E T
		MM 0095	Meat	1 (fat) E	5 (fat) E T
		ML 0106	Milks	0.02 F E	0.05 F E T
		Residue: Sum of p,p_-DDT, o,p_-DDT, p,p_-DDE and p,p_-TDE (DDD) (fat-			
Diazinon [1] (022)	0.002		Almond, hull	5	-
		TN 0660	Almonds	0.05	0.1
		GC 0640	Barley	W	0.1
		FB 0264	Blackberries	0.1	0.5[1]
		FB 4079	Boysenberry	0.1	0.5[1]
		VB 0400	Broccoli	0.5	0.5[2]
		VB 0041	Cabbages, Head	2	0.5[2]
		VC 4199	Cantaloupe	0.2	0.5[2]
		VR 0577	Carrot	0.5	0.5[2]
		FS 0013	Cherries	1	0.5[1]
		VL 0467	Chinese cabbage	0.05	0.7[3]
		FC 0001	Citrus fruits	W	0.7
		VP 0526	Common bean (pods and/or immature seeds)	0.2	0.5[2]
		SO 0691	Cotton seed	W	0.1
		VC 0424	Cucumber	0.1	0.5[2]
		FB 0021	Currants, Black, Red, White	0.2	0.5[1]
			Fruits (except as otherwise listed)	W	0.5
		VP 0529	Garden pea, shelled	0.2	0.5[2]
		TN 0666	Hazelnuts	W	0.1
		VL 0480	Kale	0.05	0.7[3]
		FI 0341	Kiwifruit	0.2	0.5[1]
		VB 0405	Kohlrabi	0.2	0.5[2]
Diazinon (contd.)		VL 0053	Leafy vegetables	W	0.7
		VL 0482	Lettuce, Head	0.5	0.7[3]
		VL 0483	Lettuce, Leaf	0.5	0.7[3]
		AF 0645	Maize forage	10	-
		GC 0645	Maize	0.02*	-
		MM 0097	Meat of cattle, pigs and sheep	W	0.7 (fat) V
		ML 0106	Milks	W	0.02 F V
		OC 0305	Olive oil, virgin	W	2
		FT 0305	Olives	W	2
		VA 0385	Onion, Bulb	0.05	0.5[2]

[1] Re-evaluation in periodic review programme

		FS 0247	Peach	0.2	0.7
		SO 0697	Peanut	W	0.1
		TN 0672	Pecan	W	0.1
		VO 0445	Peppers, Sweet	0.05	0.5[2]
		FI 0353	Pineapple	0.1	0.5[1]
		FS 0014	Plums (including Prunes)	1	0.5[1]
		FP 0009	Pome fruits	2	0.5[1]
		VR 0589	Potato	0.01*	0.5[2]
		DF 0014	Prunes	2	0.5[1]
		VR 0494	Radish	0.1	0.5[2]
		FB 0272	Raspberries, Red, Black	0.2	0.5[1]
		CM 1205	Rice, polished	W	0.1
		SO 0699	Safflower seed	W	0.1
		VL 0502	Spinach	0.5	0.7[3]
		VA 0389	Spring onion	1	0.5[2]
		VC 0431	Squash, Summer	0.05	0.5[2]
		FB 0275	Strawberry	0.1	0.5[1]
		AV 0596	Sugar beet leaves or tops	5	-
		VR 0596	Sugar beet	0.1	0.5[2]
		SO 0702	Sunflower seed	W	0.1
		VO 0447	Sweet corn (corn-on-the-cob)	0.02	0.7
		VO 0448	Tomato	0.5	0.5[2]
			Vegetables (except as otherwise listed)	W	0.5
		TN 0678	Walnuts	0.01*	0.1
		GC 0654	Wheat	W	0.1
			Residue: diazinon (fat-soluble)		
		Notes	ADI confirmed		
			[1] Fruits (except as otherwise listed) [2] Vegetables (except as otherwise listed) [3] Leafy vegetables		
Dichlorvos [1] (025)	0.004	VP 0061	Beans, except broad bean and soya bean	W	0.5 for Vegetables...
		SB 0715	Cacao beans	W	5
		GC 0080	Cereal grains	5 (Po)	2
		SB 0716	Coffee beans	W	2
Dichlorvos (contd.)		PE 0112	Eggs	W	0.05
			Fruits	W	0.1
		MM 0814	Goat meat	W	0.05
		VD 0533	Lentil (dry)	W	2
		VL 0482	Lettuce, Head	W	1
		MM 0097	Meat of cattle, pigs & sheep	W	0.05
		MM 0095	Meat	0.05*	
		ML 0106	Milks	0.02*	0.02
		VD 0541	Soya bean (dry)	W	2 Po
			Vegetables (except as otherwise listed)	W	0.5
		CM 0654	Wheat bran, unprocessed	10	-
		CF 1211	Wheat flour	1	-
		CF 1210	Wheat germ	10	-
		CF 1212	Wheat wholemeal	2	-
		Residue:	dichlorvos		

[1] Re-evaluation in periodic review programme

		Note	ADI confirmed		
Diquat (031)	0.002	Note	ADI is for diquat ion. Lowered from 0.008 mg/kg bw		
Dithiocarbamates [1]	See		Almond hulls	20	-
(105)	Note	TN 0660	Almonds	0.1*	-
		VS 0621	Asparagus	0.1	-
		FI 0327	Banana	2	1
		GC 0640	Barley	1	-
		AS 0640	Barley straw and fodder, dry	25	-
		VB 0041	Cabbages, Head	5	-
		VR 0577	Carrot	1	0.5
		VS 0624	Celery	W	5
		FS 0013	Cherries	W	1
		VP 0526	Common bean (pods and/or immature seeds)	W	0.5
		VL 0510	Cos lettuce	10	-
		FB 0265	Cranberry	5	-
		VC 0424	Cucumber	2	0.5
		FB 0021	Currants, Black, Red, White	10	5
		MO 0105	Edible offal (Mammalian)	0.1	-
		PE 0112	Eggs	0.05*	-
		VA 0381	Garlic	0.5	-
		FB 0269	Grapes	5	5
		VL 0480	Kale	15	-
		VA 0384	Leek	0.5	-
		VL 0482	Lettuce, Head	10	5
		AS 0645	Maize fodder	2	-
		FC 0003	Mandarins	10	-
		MM 0095	Meat	0.02*	-
		VC 0046	Melons, except Watermelon	0.5	1
		ML 0106	Milks	0.05*	-
		VA 0385	Onion, Bulb	0.5	-
		FC 0004	Oranges, Sweet, Sour	2	-
		FI 0350	Papaya	5	-
		FS 0247	Peach	W	3
		SO 0697	Peanut	0.1*	-
		AL 0697	Peanut fodder	5	-
		VO 0445	Peppers, Sweet	1	-
		FS 0014	Plums (including Prunes)	W	1
		FP 0009	Pome fruits	5	Apple 3 Pear 3
		VR 0589	Potato	0.2	0.1
		PM 0111	Poultry, Edible offal of	0.1	-
		PO 0110	Poultry meat	0.1	-
		VC 0429	Pumpkins	0.2	-
		VA 0389	Spring onion	10	-
		FB 0275	Strawberry	W	3
		VR 0596	Sugar beet	0.5	-
		AV 0596	Sugar beet leaves or tops	20	-
		VC 0431	Squash, Summer	1	-
		VO 0447	Sweet corn (corn-on-the-cob)	0.1*	-
		VO 0448	Tomato	5	3
		VC 0432	Watermelon	1	-
		GC 0654	Wheat	1	0.2
		AS 0654	Wheat straw and fodder, dry	25	-
		VC 0433	Winter squash	0.1	-

[1] Re-evaluation in periodic review programme

		Residue: CS_2 Notes 1. Group ADI for ethylenebis(dithiocarbamate)s (EBDCs) - mancozeb, 0-0.03 mg/kg bw, alone or in any combination. ADIs for mancozeb, maneb and zineb See also ethylenethiourea, propineb and propylenethi-ourea. 2. Recommendations for MRLs apply to total residues arising s of dithiocarbamates.			
Endosulfan (032)	0.006	VP 0522	Broad bean (green pods and immature seeds)	0.5	2[1]
		SB 0715	Cacao beans	0.1	-
		SB 0716	Coffee beans	0.1	-
		VC 0424	Cucumber	0.5	2[1]
			Fruits	W	2
		FB 0269	Grapes	1	2[2]
		GC 0645	Maize	0.1	-
		VC 0046	Melons, except Watermelon	0.5	2[1]
		FC 0004	Oranges, Sweet, Sour	0.5	2[2]
		FS 0247	Peach	1	2[2]
		FI 0353	Pineapple	2 Po	2[2]
		SO 0495	Rape seed	0.5	-
		VD 0541	Soya bean (dry)	1	2[1]
		VC 0431	Squash, Summer	0.5	2[1]
		SO 0702	Sunflower seed	1	-
		VO 0448	Tomato	0.5	2[1]
			Vegetables, except as otherwise listed	W	2
		GC 0654	Wheat	0.2	-
		Residue: sum of alpha- and beta-endosulfan and endosulfan sulphate (fat-soluble) Notes [1] Vegetables, except as otherwise listed [2] Fruits			
Ethephon (106)	0.05	Note As an ADI has now been allocated, previous GLs would normally be nce ethephon is now scheduled for residue evalu-ation in 1984 however, adoption of esent recommended			
Ethylenethiourea [1] (ETU, 108)	0.004	VR 0577	Carrot	W	0.01*
		VS 0624	Celery	W	0.01*
		VL 0482	Lettuce, Head	W	0.01*
		VR 0589	Potato	W	0.01*
		Residue: ethylenethiourea Note ADI increased from TADI of 0.002 mg/kg bw. There are no other TMRLs			
Etofenprox [2] (184)	0.03	FP 0009	Pome fruits	1	-
		VR 0589	Potato	0.01*	-
		Residue: etofenprox (fat-soluble)			
Fenbutatin oxide [1] (109)	0.03	TN 0660	Almonds	0.5	-
		FP 0226	Apple	W[1]	5
		AB 0226	Apple pomace, dry	40	20
		FI 0327	Banana	10	-
		FS 0013	Cherries	10	5
		PO 0840	Chicken, Edible offal of	0.05*	-
		PM 0840	Chicken meat	0.05*	-

[1] Re-evaluation in periodic review programme

		FC 0001	Citrus fruits	W[2]	5
		AB 0001	Citrus pulp, dry	25	7
		VC 0424	Cucumber	0.5	1
		MO 0105	Edible offal (Mammalian)	0.2	-
		VO 0440	Egg plant	W	1
		PE 0112	Eggs	0.05	-
		VC 0425	Gherkin	W	1
		FB 0269	Grapes	5	5
		FC 0203	Grapefruit	5	5 (citrus)
		AB 0269	Grape pomace, dry	100	-
		MO 1292	Horse, kidney	W[3]	0.2
		MO 1293	Horse, liver	W[3]	0.2
		MO 0098	Kidney of cattle, goats, pigs, and sheep	W[3]	0.2
		MO 0099	Liver of cattle, goats, pigs and sheep	W[3]	0.2
		FC 0206	Mandarin	5	5 (citrus)
		MM 0095	Meat	0.05*	-
		MM 0096	Meat of cattle, goats, horses, pigs and sheep	W[4]	0.02*
		VC 0046	Melons, except Watermelon	W	1
		ML 0106	Milks	0.05*	0.02*
		FC 0208	Orange, Sweet	5	5 (citrus)
		FS 0247	Peaches	7	7
		FP 0230	Pear	W[1]	5
		TN 0672	Pecans	0.5	-
		VO 0445	Peppers, Sweet	W	1
		FS 0014	Plums (including Prunes)	3	3
		FP 0009	Pome fruits	5	Apple 5 Pear 5
		DF 0014	Prunes [dried plums]	10	-
		DF 5263	Raisins	20	-
		FB 0275	Strawberry	10	3
		VO 0448	Tomato	1	1
		TN 0678	Walnuts	0.5	-
		Residue: fenbutatin oxide			
		Notes [1] Replaced by limit for Pome fruit [2] Replaced by separate limits for Grapefruit, Mandarin, and Orange, Sweet [3] Replaced by Edible offal (mammalian) [4] Replaced by revised limit for Meat			
Fenpropathrin [2] (185)	0.03	MO 0812	Cattle, Edible offal of	0.05	-
		MM 0812	Cattle meat	0.5 (fat)	-
		ML 0812	Cattle milk	0.1 F	-
		SO 0691	Cotton seed	1	-
		OC 0691	Cotton seed oil, crude	3	-
		PE 0112	Eggs	0.01*	-
		VO 0440	Egg plant	0.2	-
		VC 0425	Gherkin	0.2	-
		FB 0269	Grapes	5	-
		VO 0445	Peppers, Sweet	1	-
		FP 0009	Pome fruits	5	-
		PO 0111	Poultry, Edible offal of	0.01*	-
		PM 0111	Poultry meat	0.02 (fat)	-
		VO 0448	Tomato	1	-

[1] Re-evaluation in periodic review programme

		Residue:	fenpropathrin (fat-soluble)		
Fentin (040)	0.0005	DH 1100	Hops, dry	0.5	1
		Residue:	fentin, excluding inorganic tin and di- and mono-phenyltin		
Flucythrinate (152)	0.02	MM 0812	Cattle meat	W	0.5 (fat) T
		ML 0812	Cattle milk	W	0.1 F T
		MM 0814	Goat meat	W	0.5 (fat) T
		Residue:	flucythrinate (fat-soluble)		
Flusilazole (165)	0.001	FS 0240	Apricot	0.5	-
		FS 0245	Nectarine	0.5	0.1 T
		FS 0247	Peach	0.5	0.1 T
		Residue:	flusilazole		
Folpet (041)	0.01 T (1995)	FP 0226	Apple	W	10 T
		FS 0013	Cherries	W	15 T
		FC 0001	Citrus fruits	W	10 T
		VC 0424	Cucumber	W	2 T
		FB 0269	Grapes	2	25 T
		VL 0482	Lettuce, Head	W	15 T
		VC 0046	Melons, except Watermelon	W	2 T
		VA 0385	Onion, Bulb	W	2 T
		FB 0275	Strawberry	5	20 T
		VR 0589	Potato	0.02*	-
		VO 0448	Tomato	W	5 T
		Residue:	folpet		
		Note	Existing TADI extended until 1995		
Heptachlor (043)	0.0001	VR 0577	Carrots	W	0.2 E T
		VR 0596	Sugar beets	W	0.05 E
		VO 0448	Tomato	W	0.02 E T
			Vegetables	W	0.05 E T
		Residue:	sum of heptachlor and heptachlor epoxide (fat-soluble)		
Hexaconazole (170)	0.005	GC 0654	Wheat	0.1	0.1 T
		AS 0654	Wheat straw and fodder, dry	0.5	0.5 T
Mancozeb [1] (050)	0.03	Notes	1. ADI is group ADI for EBDCs: see Dithiocarbamates. Previous ADI 0-0.05 mg/kg bw 2. See Dithiocarbamates for recommended MRLs		
Maneb [1] (Dithiocarbamates, 105)	0.03	Notes	1. ADI is group ADI for EBDCs: see Dithiocarbamates. Previous ADI 0-0.05 mg/kg bw 2. See Dithiocarbamates for recommended MRLs		
Metiram [2] (186)	0.03	Note	ADI is group ADI for EBDCs: see Dithiocarbamates.		
Monocrotophos (054)	0.0006	Note	ADI increased from 0.00005 mg/kg bw		
(112)	0.0002	VR 0577	Carrot	0.2	0.5
		Residue:	sum of phorate, its oxygen analogue, and their sulphoxides and orate		
Phosalone (060)	0.001	Note	ADI lowered from 0.006 mg/kg bw		

[1] Re-evaluation in periodic review programme

Procymidone (136)	0.1	FP 0226	Apple	W	5
		FS 0013	Cherries	10	5
		VP 0526	Common bean (pods and/or immature seeds)	1	2
		FB 0021	Currants, Black, Red, White	W	10
		VO 0440	Egg plant	W	2
		FB 0269	Grapes	5	5 T
		FI 0341	Kiwifruit	W	7
		VC 0046	Melons, except Watermelon	W	1
		VA 0385	Onion, Bulb	0.2	0.2
		VR 0589	Potato	W	0.1
		CM 0649	Rice, husked	W	3
		CM 1205	Rice, polished	W	1
		SO 0702	Sunflower seed	0.2	2
		OR 0702	Sunflower seed oil, edible	0.5	-
Propineb [1]	0.007	Notes	1. previous TADI of 0.005 mg/kg bw was withdrawn in 1985 2. See Dithiocarbamates for recommended MRLs		
Propylenethiourea (PTU) (150)	0.0002 T (1999)	Note	As a TADI has now been allocated, GIs for propylenethiourea would as TMRLs, but the Meeting did not consider the GIs in this context		
Pyrazophos (153)	0.004	FP 0226	Apple	1	0.5
		AS 0640	Barley straw and fodder, dry	5	-
		VC 0424	Cucumber	0.1	0.1
		VC 0046	Melons, except Watermelon	0.1	0.1
		AS 0654	Wheat straw and fodder, dry	5	-
		Residue:	pyrazophos		
Triazophos (143)	0.001	FI 0327	Banana	W	1
		VR 0577	Carrot	0.5	0.1
		FC 0001	Citrus fruits	2 T	2
		VD 0541	Soya bean (dry)	0.05*	-
		FB 0275	Strawberry	0.05*	-
		Residue:	triazophos		
		Note	ADI increased from TADI of 0.0002 mg/kg bw. All previous TMRLs become MRLs		
Zineb [1] (Dithiocarbamates, 105)	0.03	Note	ADI is group ADI for EBDCs: see Dithiocarbamates. Previous ADI 0-0.05		

17

[1] Re-evaluation in periodic review programme

[1] Re-evaluation in periodic review programme

[1] Re-evaluation in periodic review programme

ANNEX II

INDEX OF REPORTS AND EVALUATIONS

Numbers in parentheses are Codex Classification Numbers.

	(R)
BROMIDE ION (047)	1968 (R), 1969 (T,R), 1971 (R), 1979 (R), 1981 (R), 1983 (R), 1988 (T,R), 1989 (R), 1992 (R)
BROMOMETHANE (052)	1965 (T,R), 1966 (T,R), 1967 (R), 1968 (T,R), 1971 (R), 1979 (R), 1985 (R), 1992 (R)
BROMOPHOS (004)	1972 (T,R), 1975 (R), 1977 (T,R), 1982 (R), 1984 (R), 1985 (R)
BROMOPHOS-ETHYL (005)	1972 (T,R), 1975 (T,R), 1977 (R)
BROMOPROPYLATE (070)	1973 (T,R), 1993 (T,R)
BUTOCARBOXIM (139)	1983 (R), 1984 (T), 1985 (T), 1986 (R)
BUPROFEZIN (173)	1991 (T,R)
sec-BUTYLAMINE (089)	1975 (T,R), 1977 (R), 1978 (T,R), 1979 (R), 1980 (R), 1981 (T), 1984 (T,R: withdrawal of TADI, but no evaluation)

CADUSAFOS (174)	1991 (T,R), 1992 (R), 1992 (R)
CAMPHECHLOR (071)	1968 (T,R), 1973 (T,R)
CAPTAFOL (006)	1969 (T,R), 1973 (T,R), 1974 (R), 1976 (R), 1977 (T,R), 1982 (T), 1985 (T,R), 1986 (corr. to 1985 rpt), 1990 (R)
CAPTAN (007)	1965 (T), 1969 (T,R), 1973 (T), 1974 (R), 1977 (T,R), 1978 (T,R), 1980 (R), 1982 (T), 1984 (T,R), 1986 (R), 1987 (R and corr. to 1986 evaluation), 1990 (T,R), 1991 (corr. to 1990 R evaluation)
CARBARYL (008)	1965 (T), 1966 (T,R), 1967 (T,R), 1968 (R), 1969 (T,R), 1970 (R), 1973 (T,R), 1975 (R), 1976 (R), 1977 (R), 1979 (R), 1984 (R)
CARBENDAZIM (072)	1973 (T,R), 1976 (R), 1977 (T), 1978 (R), 1983 (T,R), 1985 (T,R), 1987 (R), 1988 (R), 1990 (R)
CARBOFURAN (096)	1976 (T,R), 1979 (T,R), 1980 (T), 1982 (T), 1991 (R), 1993 (R)
CARBON DISULPHIDE (009)	1965 (T,R), 1967 (R), 1968 (R), 1971 (R), 1985 (R)
CARBON TETRACHLORIDE (010)	1965 (T,R), 1967 (R), 1968 (T,R), 1971 (R), 1979 (R), 1985 (R)
CARBOPHENOTHION (011)	1972 (T,R), 1976 (T,R), 1977 (T,R), 1979 (T,R), 1980 (T,R), 1983 (R)
CARBOSULFAN (145)	1984 (T,R), 1986 (T), 1991 (R), 1992 (corr. to 1991 rpt), 1993 (R)
CARTAP (097)	1976 (T,R), 1978 (T,R)
CHINOMETHIONAT (080)	1968 (T,R) (as oxythioquinox), 1974 (T,R), 1977 (T,R), 1981 (T,R), 1983 (R), 1984 (T,R), 1987 (T)
CHLORBENSIDE	1965 (T)
CHLORDANE (012)	1965 (T), 1967 (T,R), 1969 (R), 1970 (T,R), 1972 (R), 1974 (R),

	1977 (T,R), 1982 (T), 1984 (T,R), 1986 (T)
CHLORDIMEFORM (013)	1971 (T,R), 1975 (T,R), 1977 (T), 1978 (T,R), 1979(T), 1980(T), 1985 (T), 1986 (R), 1987 (T)
CHLORFENSON	1965 (T)
CHLORFENVINPHOS (014)	1971 (T,R), 1984 (R)
CHLORMEQUAT (015)	1970 (T,R), 1972 (T,R), 1976 (R), 1985 (R)
CHLOROBENZILATE (016)	1965 (T), 1968 (T,R), 1972 (R), 1975 (R), 1977 (R), 1980 (T)
CHLOROPICRIN	1965 (T,R)
CHLOROPROPYLATE	1968 (T,R), 1972 (R)
CHLOROTHALONIL (081)	1974 (T,R), 1977 (T,R), 1978 (R), 1979 (T,R), 1981 (T,R), 1983 (T,R), 1984 (corr. to 1983 rpt and T evaluation), 1985 (T,R), 1987 (T), 1988 (R), 1990 (T,R), 1991 (corr. to 1990 evaluation), 1992 (T), 1993 (R)
CHLORPROPHAM	1965 (T)
CHLORPYRIFOS (017)	1972 (T,R), 1974 (R), 1975 (R), 1977 (T,R), 1981 (R), 1982(T,R), 1983 (R), 1989 (R)
CHLORPYRIFOS-METHYL (090)	1975 (T,R), 1976 (R, Annex I only), 1979 (R), 1990 (R), 1991 (T,R), 1992 (T) and corr. to 1991, 1993 (R)
CHLORTHION	1965 (T)
CLOFENTEZINE (156)	1986 (T,R), 1987 (R), 1989 (R), 1990 (R), 1992 (R)
COUMAPHOS (018)	1968 (T,R), 1972 (R), 1975 (R), 1978 (R), 1980 (T,R), 1983(R),1987 (T), 1990 (T,R)
CRUFOMATE (019)	1968 (T,R), 1972 (R)
CYANOFENPHOS (091)	1975 (T,R), 1978 (T: ADI extended, but no evaluation), 1980, (T), 1982 (R), 1983 (T)
CYCLOXYDIM (179)	1992 (T,R), 1993 (R)
CYFLUTHRIN (157)	1986 (R), 1987 (T and corr. to 1986 rpt), 1989 (R), 1990 (R), 1992 (R)
CYHALOTHRIN (146)	1984 (T,R), 1986 (R), 1988 (R)
CYHEXATIN (TRICYCLOHEXYLTIN HYDROXIDE) (067)	1970 (T,R), 1973 (T,R), 1974 (R), 1975(R), 1977 (T), 1978 (T,R), 1980 (T), 1981 (T), 1982 (R), 1983 (R), 1985 (R), 1988 (T), 1989 (T), 1991 (T,R), 1992 (R)
CYPERMETHRIN (118)	1979 (T,R), 1981 (T,R), 1982 (R), 1983 (R), 1984 (R), 1985(R), 1986 (R), 1987 (corr. to 1986 evaluation), 1988 (R), 1990 (R)
CYROMAZINE (169)	1990 (T,R), 1991 (corr. to 1990 R evaluation), 1992 (R)

2,4-D (020)	1970 (T,R), 1971 (T,R), 1974 (T,R), 1975 (T,R), 1980 (R), 1985, (R), 1986 (R), 1987 (corr. to 1986 rpt, Annex I)
DAMINOZIDE (104)	1977 (T,R), 1983 (T), 1989 (T,R), 1991 (T)
DDT (021)	1965 (T), 1966 (T,R), 1967 (T,R),1968 (T,R), 1969 (T,R), 1978 (R), 1979 (T), 1980 (T), 1983 (T), 1984 (T), 1993 (R)
DELTAMETHRIN (135)	1980 (T,R), 1981 (T,R), 1982 (T,R), 1984 (R), 1985 (R), 1986, (R), 1987 (R), 1988 (R), 1990 (R), 1992 (R)
DEMETON (092)	1965 (T), 1967 (R), 1975 (R), 1982 (T)
DEMETON-S-METHYL (073)	1973 (T,R), 1979 (R), 1982 (T), 1984 (T,R), 1989 (T,R), 1992 (R)
DEMETON-S-METHYLSULPHON (164)	1973 (T,R), 1982 (T), 1984 (T,R), 1989 (T,R), 1992 (R)
DIALIFOS (098)	1976 (T,R), 1982 (T), 1985 (R)
DIAZINON (022)	1965 (T), 1966 (T), 1967 (R), 1968 (T,R), 1970 (T,R), 1975 (R), 1979 (R), 1993 (T,R)
1,2-DIBROMOETHANE (023)	1965 (T,R), 1966 (T,R), 1967 (R), 1968 (R), 1971 (R), 1979 (R), 1985 (R)
DICHLOFLUANID (082)	1969 (T,R), 1974 (T,R), 1977 (T,R), 1979 (T,R), 1981 (R),1982 (R), 1983 (T,R), 1985 (R)
1,2-DICHLOROETHANE (024)	1965 (T,R), 1967 (R), 1971 (R), 1979 (R), 1985 (R)
DICHLORVOS (025)	1965 (T,R), 1966 (T,R), 1967 (T,R), 1969 (R), 1970 (T,R), 1974 (R), 1977 (T), 1993 (T,R)
DICLORAN (083)	1974 (T,R), 1977 (T,R)
DICOFOL (026)	1968 (T,R), 1970 (R), 1974 (R), 1992 (T,R)
DIELDRIN (001)	1965 (T), 1966 (T,R), 1967 (T,R), 1968 (R), 1969 (R), 1970, (T,R), 1974 (R), 1975 (R), 1977 (T), 1990 (R), 1992 (R)
DIFLUBENZURON (130)	1981 (T,R), 1983 (R), 1984 (T,R), 1985 (T,R), 1988 (R)
DIMETHIPIN (151)	1985 (T,R), 1987 (T,R), 1988 (T,R)
DIMETHOATE (027)	1965 (T), 1966 (T), 1967 (T,R), 1970 (R), 1973 (R in evaluation of formothion), 1977 (R), 1978 (R), 1983 (R) 1984 (T,R) 1986(R), 1987 (T,R), 1988 (R), 1990 (R), 1991 (corr. to 1990 evaluation)
DIMETHRIN	1965 (T)
DINOCAP (087)	1969 (T,R), 1974 (T,R), 1989 (T,R), 1992 (R)
DIOXATHION (028)	1968 (T,R), 1972 (R)
DIPHENYL (029)	1966 (T,R), 1967 (T)

DIPHENYLAMINE (030)	1969 (T,R), 1976 (T,R), 1979 (R), 1982 (T), 1984 (T,R)
DIQUAT (031)	1970 (T,R), 1972 (T,R), 1976 (R), 1977 (T,R), 1978 (R)
DISULFOTON (074)	1973 (T,R), 1975 (T,R), 1979 (R), 1981 (R), 1984 (R), 1991 (T,R), 1992 (corr. to 1991 rpt, Annex I)
DITHIANON (180)	1992 (T,R)
DITHIOCARBAMATES (105)	1965 (T), 1967 (T,R), 1970 (T,R), 1983 (R, propineb and thiram), 1984 (R, propineb), 1985 (R), 1987 (T, thiram), 1988 (R, thiram), 1990 (R), 1991 (corr. to 1990 evaluation), 1992 (T, thiram), 1993 (T,R)
DNOC	1965 (T)
DODINE (084)	1974 (T,R), 1976 (T,R), 1977 (R)

EDIFENPHOS (099)	1976 (T,R), 1979 (T,R), 1981 (T,R)
ENDOSULFAN (032)	1965 (T), 1967 (T,R), 1968 (T,R), 1971 (R), 1974 (R), 1975 (R), 1982 (T), 1985 (T,R), 1989 (T,R), 1993 (R)
ENDRIN (033)	1965 (T), 1970 (T,R), 1974 (R), 1975 (R), 1990 (R), 1992 (R)
ETHEPHON (106)	1977 (T,R), 1978 (T,R), 1983 (R), 1985 (R), 1993 (T)
ETHIOFENCARB (107)	1977 (T,R), 1978 (R), 1981 (R), 1982 (T,R), 1983 (R)
ETHION (034)	1968 (T,R), 1969 (R), 1970 (R), 1972 (T,R), 1975 (R), 1982 (T), 1983 (R), 1985 (T), 1986 (T), 1989 (T), 1990 (T)
ETHOPROPHOS (149)	1983 (T), 1984 (R), 1987 (T)
ETHOXYQUIN (035)	1969 (T,R)
ETHYLENE DIBROMIDE	see 1,2-dibromoethane
ETHYLENE DICHLORIDE	see 1,2-dichloroethane
ETHYLENE OXIDE	1965 (T,R), 1968 (T,R), 1971 (R)
ETHYLENETHIOUREA (ETU) (108)	1974 (R), 1977 (T,R), 1986 (T,R), 1987 (R), 1988 (T,R), 1990 (R), 1993 (T,R)
ETOFENPROX (184)	1993 (T,R)
ETRIMFOS (123)	1980 (T,R), 1982 (T,R[7]), 1986 (T,R), 1987 (R), 1988 (R), 1989 (R), 1990 (R)
FENAMIPHOS (085)	1974 (T,R), 1977 (R), 1978 (R), 1980 (R), 1985 (T), 1987 (T)
FENBUTATIN OXIDE (109)	1977 (T,R), 1979 (R), 1992 (T), 1993 (R)

[7]R evaluation omitted. Published 1986.

FENCHLORPHOS (036)	1968 (T,R), 1972 (R), 1983 (R)
FENITROTHION (037)	1969 (T,R), 1974 (T,R), 1976 (R), 1977 (T,R), 1979 (R), 1982, (T) 1983 (R), 1984 (T,R), 1986 (T,R), 1987 (R and corr. to 1986 R evaluation), 1988 (T), 1989 (R)
FENPROPATHRIN (185)	1993 (T,R)
FENSULFOTHION (038)	1972 (T,R), 1982 (T), 1983 (R)
FENTHION (039)	1971 (T,R), 1975 (T,R), 1977 (R), 1978 (T,R), 1979 (T), 1980 (T), 1983 (R), 1989 (R)
FENTIN COMPOUNDS (040)	1965 (T), 1970 (T,R), 1972 (R), 1986 (R), 1991 (T,R), 1993 (R)
FENVALERATE (119)	1979 (T,R), 1981 (T,R), 1982 (T), 1984 (T,R), 1985 (R), 1986 (T,R), 1987 (R and corr. to 1986 rpt), 1988 (R), 1990 (R), 1991 (corr. to 1990 evaluation)
FERBAM	see dithiocarbamates, 1965 (T), 1967 (T,R)
FLUCYTHRINATE (152)	1985 (T,R), 1987 (R), 1988 (R), 1989 (R), 1990 (R), 1993 (R)
FLUSILAZOLE (165)	1989 (T,R), 1990 (R), 1991 (R), 1993 (R)
FOLPET (041)	1969 (T,R), 1973 (T), 1974 (R), 1982 (T), 1984 (T,R), 1986 (T), 1987 (R), 1990 (T,R), 1991 (corr. to 1990 R evaluation), 1993 (T,R)
FORMOTHION (042)	1969 (T,R), 1972 (R), 1973 (T,R), 1978 (R)

GLUFOSINATE-AMMONIUM (175)	1991 (T,R), 1992 (corr. to 1991 rpt, Annex I)
GLYPHOSATE (158)	1986 (T,R), 1987 (R and corr. to 1986 rpt), 1988 (R))
GUAZATINE (114)	1978 (T.R), 1980 (R)

HEPTACHLOR (043)	1965 (T), 1966 (T,R), 1967 (R), 1968 (R), 1969 (R), 1970 (T,R), 1974 (R), 1975 (R), 1977 (R), 1987 (R), 1991 (T,R), 1992 (corr. to 1991 rpt, Annex I), 1993 (R)
HEXACHLOROBENZENE (044)	1969 (T,R), 1973 (T,R), 1974 (T,R), 1978(T), 1985 (R)
HEXACONAZOLE (170)	1990 (T,R), 1991 (R and corr. to 1990 R evaluation), 1993 (R)
HEXYTHIAZOX (176)	1991 (T,R)
HYDROGEN CYANIDE (045)	1965 (T,R)
HYDROGEN PHOSPHIDE (046)	1965 (T,R), 1966 (T,R), 1967 (R), 1969 (R), 1971 (R)
IMAZALIL (110)	1977 (T,R), 1980 (T,R), 1984 (T,R), 1985 (T,R), 1986 (T), 1988 (R), 1989 (R), 1991 (T)

IPRODIONE (111)	1977 (T,R), 1980 (R), 1992 (T)
ISOFENPHOS (131)	1981 (T,R), 1982 (T,R), 1984 (R), 1985 (R), 1986 (T,R), 1988 (R), 1992 (R)
LEAD ARSENATE	1965 (T), 1968 (T,R)
LEPTOPHOS (088)	1974 (T,R), 1975 (T,R), 1978 (T,R)
LINDANE (048)	1965 (T), 1966 (T,R), 1967 (R), 1968 (R), 1969 (R), 1970 (T,R) (published as Annex VI to 1971 evaluations), 1973 (T,R), 1974 (R), 1975 (R), 1977 (T,R), 1978 (R), 1979 (R), 1989 (T,R)
MALATHION (049)	1965 (T), 1966 (T,R), 1967 (corr. to 1966 R), 1968 (R), 1969 (R), 1970 (R), 1973 (R), 1975 (R), 1977 (R), 1984 (R)
MALEIC HYDRAZIDE (102)	1976 (T,R), 1977 (T,R), 1980 (T), 1984 (T,R)
MANCOZEB (050)	1967 (T,R), 1970 (T,R), 1974 (R), 1977 (R), 1980 (T,R), 1993 (T,R)
MANEB	see dithiocarbamates, 1965 (T), 1967 (T,R), 1987 (T), 1993 (T,R)
MECARBAM (124)	1980 (T,R), 1983 (T,R), 1985 (T,R), 1986 (T,R), 1987 (R)
METALAXYL (138)	1982 (T,R), 1984 (R), 1985 (R), 1986 (R), 1987 (R), 1989 (R), 1990 (R), 1992 (R)
METHACRIFOS (125)	1980 (T,R), 1982 (T), 1986 (T), 1988 (T), 1990 (T,R), 1992 (R)
METHAMIDOPHOS (100)	1976 (T,R), 1979 (R), 1981 (R), 1982 (T,R[8]), 1984 (R), 1985 (T), 1989 (R), 1990 (T,R)
METHIDATHION (051)	1972 (T,R), 1975 (T,R), 1979 (R), 1992 (T,R)
METHIOCARB (132)	1981 (T,R), 1983 (T,R), 1984 (T), 1985 (T), 1986 (R), 1987 (T,R), 1988 (R)
METHOMYL (094)	1975 (R), 1976 (R), 1977 (R), 1978 (R), 1986 (T,R), 1987 (R), 1988 (R), 1989 (T,R), 1990 (R), 1991 (R)
METHOPRENE (147)	1984 (T,R), 1986 (R), 1987 (T and corr. to 1986 rpt), 1988 (R), 1989 (R)
METHOXYCHLOR	1965 (T), 1977 (T)
METHYL BROMIDE (052)	see bromomethane
METIRAM (186)	1993 (T,R)
MEVINPHOS (053)	1965 (T), 1972 (T,R)
MGK 264	1967 (T,R)
MONOCROTOPHOS (054)	1972 (T,R), 1975 (T,R), 1991 (T,R), 1993 (T)
MYCLOBUTANIL (181)	1992 (T,R)

[8]R evaluation omitted. Published 1989.

NABAM	see dithiocarbamates, 1965 (T), 1976 (T,R)
NITROFEN (140)	1983 (T,R)

OMETHOATE (055)	1971 (T,R), 1975 (T,R), 1978 (T,R), 1979 (T), 1981(T,R),1984 (R), 1985 (T), 1986 (R), 1987 (R), 1988 (R), 1990 (R)
ORGANOMERCURY COMPOUNDS	1965 (T), 1966 (T,R), 1967 (T,R)
OXAMYL (126)	1980 (T,R), 1983 (R), 1984 (T), 1985 (T,R), 1986 (R)
OXYDEMETON-METHYL (166)	1965 (T, as demeton-S-methyl sulphoxide), 1967 (T), 1968 (R), 1973 (T,R), 1982 (T), 1984 (T,R), 1989 (T,R), 1992 (R)
OXYTHIOQUINOX	see chinomethionat

PACLOBUTRAZOL (161)	1988 (T,R), 1989 (R)
PARAQUAT (057)	1970 (T,R), 1972 (T,R), 1976 (T,R), 1978(R), 1981 (R), 1982 (T), 1985 (T), 1986 (T)
PARATHION (058)	1965 (T), 1967 (T,R), 1969 (R), 1970 (R), 1984 (R), 1991 (R)
PARATHION-METHYL (059)	1965 (T), 1968 (T,R), 1972 (R), 1975 (T,R), 1978 (T,R), 1979 (T), 1980 (T), 1982 (T), 1984 (T,R), 1991 (R), 1992 (R)
PENCONAZOLE (182)	1992 (T,R)
PERMETHRIN (120)	1979 (T,R), 1980 (R), 1981 (T,R), 1982 (R), 1983 (R), 1984 (R), 1985 (R), 1986 (T,R), 1987 (T), 1988 (R), 1989 (R), 1991 (R), 1992 (corr. to 1991 rpt)
2-PHENYLPHENOL (056)	1969 (T,R), 1975 (R), 1983 (T), 1985 (T,R), 1989 (T), 1990 (T,R)
PHENOTHRIN (127)	1979 (R), 1980 (T,R), 1982 (T), 1984 (T), 1987 (R), 1988 (T,R)
PHENTHOATE (128)	1980 (T,R), 1981 (R), 1984 (T)
PHORATE (112)	1977 (T,R), 1982 (T), 1983 (T), 1984 (R), 1985 (T), 1990 (R), 1991 (R), 1992 (R), 1993 (R)
PHOSALONE (060)	1972 (T,R), 1975 (R), 1976 (R), 1993 (T)
PHOSMET (103)	1976 (R), 1977 (corr. to 1976 evaluation), 1978 (T,R), 1979 (T,R), 1981 (R), 1984 (R), 1985 (R), 1986 (R), 1987 (R and corr. to 1986 evaluation), 1988 (R)
PHOSPHINE	see hydrogen phosphide
PHOSPHAMIDON (061)	1965 (T), 1966 (T), 1968 (T,R), 1969 (R), 1972 (R), 1974 (R), 1982 (T), 1985 (T), 1986 (T)

PHOXIM (141)	1982 (T), 1983 (R), 1984 (T,R), 1986 (R), 1987 (R), 1988 (R)
PIPERONYL BUTOXIDE (062)	1965 (T,R), 1966 (T,R), 1967 (R), 1969 (R), 1972 (T,R), 1992 (T,R)
PIRIMICARB (101)	1976 (T,R), 1978 (T,R), 1979 (R), 1981 (T,R), 1982 (T), 1985 (R)
PIRIMIPHOS-METHYL (086)	1974 (T,R), 1976 (T,R), 1977 (R), 1979 (R), 1983 (R), 1985 (R), 1992 (T)
PROCHLORAZ (142)	1983 (T,R), 1985 (R), 1987 (R), 1988 (R), 1989 (R), 1990 (R), 1991 (corr. to 1990 rpt, Annex I, and evaluation), 1992 (R)
PROCYMIDONE (136)	1981 (R), 1982 (T), 1989 (T,R), 1990 (R), 1991 (corr. to 1990 Annex I), 1993 (R)
PROFENOFOS (171)	1990 (T,R), 1992 (R)
PROPAMOCARB (148)	1984 (T,R), 1986 (T,R), 1987 (R)
PROPARGITE (113)	1977 (T,R), 1978 (R), 1979 (R), 1980 (T,R), 1982 (T,R)
PROPHAM (183)	1965 (T), 1992 (T,R)
PROPICONAZOLE (160)	1987 (T,R), 1991 (R)
PROPINEB	1977 (T,R), 1980 (T), 1983 (T), 1984 (R), 1985 (T,R), 1993 (T,R)
PROPOXUR (075)	1973 (T,R), 1977 (R), 1981 (R), 1983 (R), 1989 (T), 1991 (R)
PROPYLENETHIOUREA (PTU) (150)	1993 (T,R)
PYRAZOPHOS (153)	1985 (T,R), 1987 (R), 1992 (T,R), 1993 (R)
PYRETHRINS (063)	1965 (T), 1966 (T,R), 1967 (R), 1968 (R), 1969 (R), 1970 (T), 1972 (T,R), 1974 (R)

QUINTOZENE (064)	1969 (T,R) 1973 (T,R), 1974 (R), 1975 (T,R), 1976 (Annex I, corr. to 1975 R), 1977 (T,R)

2,4,5-T (121)	1970 (T,R), 1979 (T,R), 1981 (T)
TECNAZENE (115)	1974 (T,R), 1978 (T,R), 1981 (R), 1983 (T), 1987 (R), 1989 (R)
TERBUFOS (167)	1989 (T,R), 1990 (T,R)
THIABENDAZOLE (065)	1970 (T,R), 1971 (R), 1972 (R), 1975 (R), 1977 (T,R), 1979 (R), 1981 (R)
THIODICARB (154)	1985 (T,R), 1986 (T), 1987 (R), 1988 (R)
THIOMETON (076)	1969 (T,R), 1973 (T,R), 1976 (R), 1979 (T,R), 1988 (R)
THIOPHANATE-METHYL (077)	1973 (T,R), 1975 (T,R), 1977 (T), 1978 (R), 1988 (R), 1990 (R)
THIRAM (105)	see dithiocarbamates, 1965 (T), 1967 (T,R), 1970 (T,R), 1974 (T),

	1977 (T), 1983 (R), 1984 (R), 1985 (T,R), 1987 (T), 1988 (R), 1989 (R), 1992 (T)
TOLYLFLUANID (162)	1988 (T,R), 1990 (R), 1991 (corr. to 1990 rpt)
TOXAPHENE	see camphechlor
TRIADIMEFON (133)	1979 (R), 1981 (T,R), 1983 (T,R), 1984 (R), 1985 (T,R), 1986 (R), 1987 (R and corr. to 1986 evaluation), 1988 (R), 1989 (R), 1992 (R)
TRIADIMENOL (168)	1989 (T,R), 1992 (R)
TRIAZOLYLALANINE	1989 (T,R)
TRIAZOPHOS (143)	1982 (T), 1983 (R), 1984 (corr. to 1983 rpt, Annex I), 1986 (T,R), 1990 (R), 1991 (T and corr. to 1990 evaluation), 1992 (R), 1993 (T,R)
TRICHLORFON (066)	1971 (T,R), 1975 (T,R), 1978 (T,R), 1987 (R)
TRICHLORONAT	1971 (T,R)
TRICHLOROETHYLENE	1968 (R)
TRICYCLOHEXYLTIN HYDROXIDE	see cyhexatin
TRIFORINE (116)	1977 (T), 1978 (T,R)
TRIPHENYLTIN COMPOUNDS	see fentin compounds

VAMIDOTHION (078)	1973 (T,R), 1982 (T), 1985 (T,R), 1987 (R), 1988 (T), 1990 (R), 1992 (R)
VINCLOZOLIN (159)	1986 (T,R), 1987 (R and corr. to 1986 rpt and R evaluation), 1988 (T,R), 1989 (R), 1990 (R), 1992 (R)

ZINEB (105)	see dithiocarbamates, 1965 (T), 1967 (T,R), 1993 (T)
ZIRAM (105)	see dithiocarbamates, 1965 (T), 1967 (T,R)

ANNEX III

INTAKE PREDICTIONS

At the request of the Meeting, WHO calculated the predicted intakes of residues of pesticides on the agenda of the Joint Meeting, based on the methods described in *Guidelines for Predicting Dietary Intake of Pesticide Residues*[v].

Detailed EMDI (Estimated Maximum Daily Intake) calculations were not performed on those pesticides for which the TMDI (Theoretical Maximum Daily Intake), based upon global diets, exceeded the ADI, because there was insufficient opportunity at the Joint Meeting to review the detailed processing data that had been supplied on the compounds of interest. The results of EMDI calculations will be made available to the Twenty-sixth Session of the Codex Committee on Pesticide Residues (CCPR) in April 1994.

The TMDI calculations were based on ADIs and MRLs proposed by the Meeting and existing and pending MRLs in the Codex system. For the following compounds the TMDI did not exceed the ADI:

aldicarb, benalaxyl, bromopropylate, carbofuran, chlorothalonil, cycloxydim, diazinon, DDT, dithiocarbamates (mancozeb and maneb), endosulfan, etofenprox, fenbutatin oxide, fenpropathrin, fentin, flucythrinate, flusilazole, folpet, hexaconazole, procymidone, propineb, and pyrazophos.

The TMDI exceeded the ADI for the following compounds (information on processing factors must be reviewed before EMDIs can be calculated):

azinphos-methyl, chlorpyrifos-methyl, dichlorvos, diquat, heptachlor, monocrotophos, phorate, phosalone and triazophos.

The TMDI was not calculated for the following compounds for which no MRLs have been proposed or where all existing MRLs have been proposed for withdrawal:

amitrole, carbosulfan, ethephon, ethylenethiourea, metiram, propylenethiourea and zineb.

The TMDIs calculated grossly over-estimate the true pesticide intake. It should, therefore, not be concluded that the MRLs proposed by the Meeting are unacceptable when the TMDI exceeds the ADI. Instead, TMDI calculations should be used as a screening tool that may eliminate the need for further calculations of the intake of a pesticide when its value is below the ADI. When the TMDI exceeds the ADI, EMDI and, if necessary, EDI (Estimated Daily Intake), calculations should be performed.

[i] *Principles for the toxicological assessment of pesticide residues in food*. WHO Environmental Health Criteria, No. 104. Geneva, World Health Organization, 1990.

[ii] *Pesticides in the diets of infants and children*. National Academy of Sciences. Washington, National Academy Press, 1993.

[iii] *Guidelines for Predicting Dietary Intake of Pesticide Residues*. Geneva, World Health Organization, 1989.

[iv] T = Toxicology

R = Residue and analytical aspects

[v.] *Guidelines for Predicting Dietary Intake of Pesticide Residues*, World Health Organization, Geneva 1989.

ALDICARB (117)

EXPLANATION

Aldicarb was evaluated in 1979, 1982, 1985, 1988 and 1990, and a complete re-evaluation is scheduled for 1994. At the 24th Session of the CCPR (1992) the draft MRL of 0.05 mg/kg for aldicarb in Brussels sprouts was held at step 7b to await new data from additional supervised trials (ALINORM 93/24, para 132).

The Meeting has received information on registered uses of aldicarb on Brussels sprouts from The Netherlands and the UK, and reports from supervised trials carried out in the UK.

USE PATTERN

In The Netherlands aldicarb is registered for use against nematodes on Brussels sprouts with broadcast application at planting at a dosage of 3 kg ai/ha. In the UK there is a general registration for the use of aldicarb against aphids, flea beetles and cabbage root fly on brassicas at a dosage of 5.1 g ai/100 m row, eqivalent to 0.67-0.84 kg ai/ha, and with one application in-furrow at planting. The pre-harvest interval in the UK is 10 weeks.

RESIDUES RESULTNG FROM SUPERVISED TRIALS

New data from supervised trials on Brussels sprouts carried out at 5 sites in the UK were available from Rhone-Poulenc (Table 1). In the trials aldicarb was applied at 3.8 g, 5.1 g, and 7.7 g ai/100 m row. Residues were determined in the whole plants and the edible parts, the "buttons". Residue levels in the buttons after a PHI of 155-198 days ranged from 0.01 to 0.03 mg/kg with one exception where the residue was 0.1 mg/kg. Residues in the whole plant at shorter PHIs (49 and 61 days) were considerably higher.

Table 1. Residues of aldicarb in Brussels sprouts from supervised trials in the UK.

{PRIVATE } Site, Year	Application No.	Application g ai/100 m row	PHI, days	Residues, mg/kg	Report
Sandy 1977	1	7.7	61 174	whole plants:0.44 buttons:0.01	UNC 78/ 79/80/ 78363
Cottenham 1977	1	7.7	49 155	whole plants:3.6 buttons:0.01	
Melbourne 1978	1	5.1 7.7	171 171	buttons:0.10 buttons:0.03	UNC 108/-112/ 79934
Fiskerton 1978	1	5.1 7.7	198 198	buttons:0.03 buttons:0.02	
Ombersley 1978	1	3.8 5.1 7.7	178 178 178	buttons:<0.01 buttons: 0.01 buttons: 0.09	

NATIONAL MAXIMUM RESIDUE LIMITS

The Meeting was informed that the national MRLs in Belgium and The Netherlands for aldicarb in Brussels sprouts were 0.05 mg/kg.

APPRAISAL

At the 24th Session of the CCPR (1992) a proposed MRL of 0.05 mg/kg for aldicarb in Brussels sprouts was held at step 7B awaiting more data from supervised trials.

Data from 5 sites in the UK were available to the Meeting. In the trials aldicarb was applied at 3.8, 5.1 and 7.7 g ai/100 m row: 5.1 g/100 m row is the registered use in the UK. Residues in the buttons were <0.01-0.03 mg/kg except in one trial at 5.1 g/100 m, where the level was 0.1 mg/kg, and one at 7.7 g ai/100m with a residue of 0.09 mg/kg.

RECOMMENDATIONS

On the basis of the data from new supervised trials with aldicarb on Brussels sprouts the Meeting concluded that the residue level shown below is suitable for establishing a maximum residue limit.

Definition of the residue: sum of aldicarb, its sulphoxide and its sulphone, expressed as aldicarb.

{PRIVATE }Commodity		Recommended MRL, mg/kg		PHI on which based, days
CCN	Name	New	Previous	
VB 0402	Brussels sprouts	0.1	0.05T	Application at planting

AMITROLE (079)

EXPLANATION

Amitrole (3-amino-1*H*-1,2,4-triazole) was originally evaluated by the JMPR in 1974. A conditonal ADI was established at 0-0.00003 mg/kg bw, and a conditional MRL for amitrole in raw agricultural commodities of plant origin was recommended at the limit of determination of 0.02 mg/kg. The ADI was confirmed by the JMPR in 1977, but the 17th Session of the CCPR (1987) recommended that the CXL (as it had become) at the limit of determination should be deleted and replaced by the statement that uses of amitrole should be restricted to those where residues in food would not be expected to occur.

The compound was scheduled for re-evaluation by the 20th Session of the CCPR (1990), and information on GAP was requested by a circular letter in 1991.

The Meeting was supplied with information on registered uses in several, mostly European, countries but data from residue trials were received only on grapes. The Meeting was provided with information on the fate of amitrole in animals, plants and soil, and on new analytical methods for the determination of amitrole in plant material, soil and water.

IDENTITY

Physical properties

The octanol/water partition coefficient was determined to be 0.108.

USE PATTERN

Information was supplied to the Meeting on registered uses of amitrole in eight countries (Table 1). Amitrole is used also on industrial land, roadsides, railways, ditches etc. Such uses are not quoted in the Table. In the 1974 evaluation detailed descriptions were given of the uses of amitrole on many crops and of non-agricultural uses. In all cases the compound should be applied in such a way that no residues are detectable in food commodities.

Table 1. Registered uses of amitrole

Country	Crop	Application			PHI, days
		No.	Rate, kg ai/ha	Spray conc., kg ai/hl	

Country	Crop	Application			PHI,
		No.	Rate, kg ai/ha	Spray conc., kg ai/hl	days
Australia	Vines and fruit	6-8 week intervals		0.14-0.28	
	Potatoes		1.4-2.8		28-35
	Cereals		0.7-1.4	0.58	sowing after > 5 days
	Pasture		0.75-1.0	0.58	graze after > 6 months
Belgium	Apple		3.0-5.0		
	Pear		5.0		
Canada	Apple orchards	1	to cover ground: 1.2 bands round trees: 0.03		> 60
		> 1	directly on ivy leaves: 2.2-4.6		30
	Maize Soya beans White beans	1	2.2-4.6		pre-sowing
	Cereals, Peas, Alfalfa, Clover	1	3.4-5.5		8 months
	Pasture	2	7.5-15		6 months
France	Apple, Pear	1	1.8		
	Other fruit	1	5.0		
Germany	Pome fruit	2	1.5-2.0		42
		1	5.0		in spring
	Plum	2	2.0		42
	Cherry	2	2.0		42
	Grape	1	2.0		42
Netherlands	Apple Pear	1-2	3.0-4.0		before blossom or after harvest
	Plum Cherry Currants	1	3.0-4.0		after harvest before 1 Nov.
Portugal	Grape	1	1.9-2.5		
Spain	Citrus fruits	1	2.5-6.0		post- and pre-emergence of weeds
	Pome fruit	1-2	2.5-6.0		as for citrus
	Grape	1	1.5-3.6		as for citrus
	Hazelnut	1	2.5-5.4		as for citrus
	Olive	1	2.4-3.2		as for citrus

RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting was informed of one report on completed trials with amitrole on grapes. New trials are being carried out on apples, pears and grapes but have not yet been completed. It was stated that reports on most of this work, including the study on grapes, would be supplied in the near future.

FATE OF RESIDUES

In the 1974 evaluation numerous studies on the degradation of amitrole in plants and soil were reviewed, but in some areas with some uncertainty about the extent of degradation and, especially in soil, about whether degradation is mainly a microbiological or a chemical process.

Several reports from new studies on the degradation of amitrole in animals, plants and soil were provided to the Meeting.

In animals

The metabolism of amitrole in rats was investigated and compared with its degradation in beans. Unaltered amitrole and three metabolites were present in the urine of treated animals, the metabolites being similar to those in beans. One of the major metabolites in rats was compatible with the structure of aminotriazolylalanine, 3-(3-amino-1,2,4-triazol-1-yl)-D-alanine, which had previously been identified as the major metabolite in plants (Franco and Municio, 1975).

In plants

In the evaluation of the 1974 JMPR several investigations of the fate of amitrole in plants were reviewed with the main conclusions that little or none of the compound is taken up from the soil via the roots to the leaves and fruit, and that if application is made directly to the leaves most of the absorbed compound is metabolized. Three or four metabolic products were observed, but only one was identified (as aminotriazolylalanine).

Further investigations of the possible uptake from soil to plant and of the metabolism in plants have been carried out.

In a study of the metabolism of some herbicides including amitrole by Field Horsetail (*Equisetum arvense*), 5-^{14}C-labelled amitrole sprayed on the plant showed evidence of conjugation in the shoots and rhizomes. In the shoots the main compound was the parent (>76%), together with aminotriazolylalanine and an unidentified metabolite. In the rhizomes the proportion of intact amitrole was lower. No further metabolites were identified. The quantity of aminotriazolylalanine decreased with time, with an increase of the unidentified metabolites.

This study also showed that conjugates with low mobility in the vascular system were formed (Marschall *et al.*,1987).

A report from Bayer AG (1982) on the degradation of amitrole in plants and soil describes experiments with treatments of *Ranunculus repens*, grapes and apples, the grapes and apples being grown in pots.

In the experiment with direct application to the weed (*Ranunculus*) 80% of the amitrole was metabolized within a few days, and the compounds were mainly present in the ends of the shoots.

For apples the soil surrounding the trees was treated with ^{14}C-labelled amitrole (about 4 kg ai/ha) twice a year for three years. The apples contained 0.1% of the radioactivity applied (about 0.2 mg/kg calculated as amitrole) in the first year of application. The level decreased to 0.02% in the second year. Approximately 18% of this was found to be the parent compound, at about 0.03 mg amitrole/kg.

A 3-year experiment was also carried out on grapes, where [^{14}C]amitrole was applied to the soil at a rate used in practice. After 90 days 1-2% of the applied radioactivity was found, mainly in the leaves. Radioactivity was present in the woody part of the vines 2 years after the last application, although in reduced quantities. The concentration of radioactivity in the grapes was approximately 0.2% of that applied during the last year of treatment. No radioactivity was detectable in the grapes after 2 years (Bayer AG, 1982).

In other experiments on the metabolism of amitrole in apples 3,5-[^{14}C]amitrole was applied to the soil in which apple trees were grown under outdoor conditions in tubs, to excised sprouts from apple trees, and to cell suspension cultures. Mature fruit from the outdoor experiment contained at most 0.05 mg/kg total residues, 75% water-soluble and 25% bound to insoluble material. No parent compound was detectable in the fruit. The major metabolite (maximum 0.012 mg/kg) was triazolylalanine (3-(1,2,4-triazol-1-yl)-D-alanine), which was present in the free form and as conjugates. More than 50% of the radioactivity was reassimilated ^{14}C incorporated into natural plant constituents.

In the tub experiment one apple tree absorbed 1.1% of the radioactivity applied to the soil, 0.07% was found within the mature fruit and about 42% remained in the soil after 5 months. In contrast to this the major metabolite in model experiments with excised apple sprouts and cell suspension cultures was not triazolylalanine but aminotriazolylalanine, although in excised sprouts small amounts of triazolylalanine were also present. In cell suspension cultures at high concentrations of amitrole, 3,5-dihydroxy-1,2,4-triazole was the main compound found (Schneider *et al.*, 1991).

In soil

The 1974 Meeting reviewed several reports on the degradation

of amitrole in soil. According to these degradation is rapid and the main product is CO_2. Some disagreement was obvious in the conclusions of these reports as to what extent degradation in soil is microbiological or chemical.

Since 1974 several experiments have been carried out on the degradation of amitrole in soil. In one, degradation and translocation were examined in sandy and loamy soils under northern German climatic conditions. Because of adsorption and rapid degradation amitrole was practically not translocated, but the possibility nevertheless exists of contamination of groundwater in sandy soils with a low content of organic material and a high water level (Drewes and Blume, 1976).

The degradation of amitrole was studied in the laboratory in two types of soil, an English loam and the German standard soil 2.2. Amitrole was labelled with ^{14}C in the 3 and 5 positions, and the soil was kept in darkness during the experiment. It was shown that the conditions exerted a marked influence on the degradation. Under aerobic conditions amitrole was extensively degraded to $^{14}CO_2$. After 28 days the production of $^{14}CO_2$ in German standard soil 2.2 amounted to 70-80% of the applied radioactivity, and in English loam soil to 40-50%, but in strictly anaerobic soil no volatile radioactivity was produced. In the aerobic soil most of the radioactivity which did not appear as $^{14}CO_2$ was not extractable and the major extractable radioactive compound was unchanged amitrole. No intermediates in the degradation to $^{14}CO_2$, including urea and cyanamide which were observed in other experiments, were detected in the soil extracts. In anaerobic soil the total non-bound radioactivity decreased to 60% of that applied after 28 days and to 25% after one year.

When soil under aerobic conditions was sterilized there was no significant loss of radioactivity, clearly indicating that the degradation of amitrole to CO_2 is strongly influenced by the presence of micro-organisms and oxygen. Similar results were obtained with [3-^{14}C] and [5-^{14}C]amitrole (Hawkins *et al.*, 1982a).

The degradation of ^{14}C-labelled amitrole was also studied in a field experiment. Field plots of loamy soil were treated with the labelled compound at 20 mg/plot (about 20 kg ai/ha) and exposed to the prevailing weather conditions.

Immediately after application the recovery of applied radioactivity was 97%, which decreased to 75% after 56 days and 53% after 112 days. Most of the radioactivity was in the top 5 cm layer of the soil at all times. After 112 days 43% of the applied radioactivity was present in this layer, about 6% in the 5-15 cm layer and about 4% in the 15-30 cm layer.

90% of the radioactivity in the extracts from the top 5 cm layer of soil was present as a compound corresponding to unchanged ^{14}C-amitrole at all sampling times. The level of ^{14}C-amitrole in the top layer declined from 22 mg/kg shortly after application to 3 mg/kg after 56 days and <0.1 mg/kg after 112 days (Hawkins *et al.*, 1982b).

A laboratory experiment was carried out on the degradation of amitrole in soil with the aim of identifying the intermediate compounds involved. Amitrole was labelled at the 3 and 5 positions. The soil was a clayish silt, which was treated with 0.1 and 1.0 mg [^{14}C]amitrole/100 g soil and incubated from 1 to 20 days. Amitrole (both labelled forms) was rapidly degradaded to CO_2. The residue in the extract of the soil consisted mostly of the parent compound, with <2.5% as metabolites. 5-hydroxy-amitrole was the primary metabolite, but was degraded very rapidly. Cyanamide and urea were also found as degradation products. Uracil presumely represents a secondary pathway in the metabolism. In the proposed degradation scheme the ring is opened between positions 1 and 5 after 5-hydroxy-amitrole is formed, CO_2 is eliminated at position 5 and hydrazine at positions 1 and 2, leaving cyanamide which is hydrolysed via urea to CO_2 and ammonia (Scholz, 1988).

In water

The stability of amitrole was studied in buffer solutions at 90 C at pH 4, 7 and 9 and a concentration of about 10 mg/l. Samples were taken 0, 19, 94 and 114 days after application. No degradation was observed, indicating that the half-life of amitrole in water at 20 C would be more than one year (Krohn, 1986).

Leaching

The mobility of amitrole and its metabolites was examined in two soil types: German standard soil 2.1 containing 0.6% organic carbon and 6.8% of the fraction <0.02 mm, and "Höpfchen" soil containing 2.9% organic carbon and 32% of the fraction <0.02 mm. ^{14}C-labelled amitrole was used at a concentration similar to that applied in practice of 10 kg ai/ha, and the tests were carried out after the soil had been incubated with amitrole for 0, 30 and 92 days, each test in duplicate. Incubated soil was placed on top of 27 cm of untreated soil in glass columns. A total of about 400 ml deionized water was used as the eluant over 2 days.

Only in the German standard soil 2.1 was more than 1% of the initially applied ^{14}C detected in the leachate. The percentage leached decreased from 24-31% from the 0-day soil to 1.5-1.9% from the 30-day and 0.7-1.6% from the 92-day. Most of the radioactivity in the 0-day leachate was from the parent compound (20-27%). Less than 0.1% of the parent compound was detected in the residue after 30 days aging. In the "Höfchen" soil, which had a considerably higher content of organic matter, the radioactivity in the leachate decreased from 0.8% from the 0-day soil to 0.1% from the soil aged for 92 days (Weller, 1987).

METHODS OF RESIDUE ANALYSIS

Analytical methods recorded in the evaluation of the 1974 JMPR were all colorometric, usually after derivatization of amitrole in cleaned up extracts. Several of them were modifications of an FDA method developed by Storherr and Burke (1961).

Gas-chromatographic methods have now been developed, using a nitrogen-specific detector, for the determination of residues of amitrole in apples and pears and in soil and water. An ethanol/water mixture is used for extraction, and after evaporation of the solvent the residue is acetylated with acetic anhydride and transferred to chloroform. Clean-up is by column chromatography on silica gel with ethyl acetate as eluant. The limit of determination in fruit and soil is 0.02 mg/kg and in water 0.002 mg/kg (Jarczyk, 1982, 1983).

A method has been developed to determine amitrole in water by TLC. After evaporation of water the residue is cleaned up by column chromatography on silica gel using an acetonitrile/NH_3/methanol mixture as eluant. Amitrole is detected on the chromatogram by diazotization and coupling to form an azo compound. Quantification is by densitometry. The **limit of determination is 0.05 ìg/l (Burger, 1986).**

HPLC methods have also been developed for the determination of residues of amitrole. In one method residues in vegetables are determined using the extraction and clean-up procedures described by Storherr and Burke (1961). The residue is then diazotized and cleaned up by column chromatography on polyamide and ion-pair HPLC. The limit of determination for residues in potatoes and beets was 0.01 mg/kg (Løkke, 1987).

Another HPLC method was developed to determine residues in blackberries. Residues are extracted with ethanol/water and, after pre-treatment with hydrogen peroxide, interfering substances are removed by ion exchange on an acidic cation-exchanger. The residue is then converted to an amitrole-fluorescamine complex and determined by HPLC with fluorescence detection. The limit of determination is 0.02 mg/kg (Dornseiffen and Verwaal, 1988).

A third HPLC method has been developed for the determination of residues in plant material, soil and water. Amitrole is extracted with acetone/water and organic components are separated by partitioning with dichloromethane. Amitrole is then isolated on a cationic ion-exchanger and purified by column chromatography on alumina. The residue is determined by HPLC with electrochemical detection. The limit of determination in vegetables is 0.01 mg/kg, in soil 0.005 **mg/kg, and in water 0.1 ìg/l (Weber, 1988).**

NATIONAL MAXIMUM RESIDUE LIMITS

National maximum residue limits reported to the Meeting are shown below.

Country	Crop	MRL, mg/kg

Australia	Avocados, bananas, cereal grains, citrus, grapes, meat, milk, milk products, passionfruits, paw-paws, pecans, pineapples, pome fruit, sugar cane	0.01
	stone fruit	0.02
	potatoes	0.05
	water	0.001
Netherlands	Agricultural food commodities	0.05 (limit of determination)
Spain	Citrus fruit, grapes, grapes, hazelnuts, Pome fruit, olives	0.05 (limit of determination)

APPRAISAL

Amitrole was evaluated by the JMPR in 1974 and 1977 and is included in the CCPR periodic review programme. A conditional ADI was allocated in 1974 and confirmed in 1977. An MRL for raw agricultural commodities was recommended at the limit of determination in 1974, but the 17th Session of the CCPR (1987) recommended that the MRL should be withdrawn and replaced by a note that uses of amitrole should be restricted to those where residues in food would not be expected to occur.

Information on registered uses was received from Australia, Belgium, Canada, France, Germany, The Netherlands, Portugal and Spain. The compound is applied to the ground and directly on to weeds and usually with a long PHI, so residues should not be detectable in crops grown on treated soil.

The Meeting received only one report from supervised trials, but was informed that new trials on apples, grapes, and pears were in progress. Most of the studies would be supplied to the JMPR in the near future.

Several reports were available from studies on the metabolism or degradation of amitrole (aminotriazole) in plants, animals and soil. In plants after direct applications to the leaves or stem the main metabolite was aminotriazolylalanine, 3-(3-amino-1,2,4-triazol-1-yl)-D-alanine. Two other metabolites were found, but not identified. The same metabolites were present in rats. After treatment of the soil surrounding plants only small amounts of aminotriazole and its metabolites were translocated to the plant. In apples residues of the parent compound and the metabolite triazolylalanine were undetectable or very low: when present the compounds were in both free and conjugated forms. In cell suspension cultures from apples 3,5-dihydroxy-1,2,4-triazole was produced.

In soil rapid degradation occurs with CO_2 as the main degradation product. Degradation in soil is strongly influenced by the presence of micro-organisms, and does not occur under anaerobic conditions. From laboratory experiments it was possible to propose a degradation scheme for amitrole in soil. The ring is opened after metabolism to 5-hydroxyaminotriazole, and via cyanamide the compound is decomposed to CO_2 and ammonia. Because of the rapid degradation only small amounts of aminotriazole are leached into soil. Leaching is most pronounced in sandy soil with a low content of organic material.

New analytical methods for the determination of residues of amitrole have been developed using gas chromatography with a nitrogen-specific detector, thin layer chromatography, and high performance liquid chromatography with fluorescence or electrochemical detection. The limits of determination are 0.01-0.02 mg/kg for residues in fruit, vegetables and soil.

A complete re-evaluation of amitrole has not been possible because new data from supervised trials were not available. Although the registered uses reported to the Meeting are similar to the application conditions in some supervised trials examined by the JMPR in 1974, the data from the trials currently in progress should be taken into consideration. No reports from studies of storage stability were available, but the Meeting was informed that the results of such studies will be available in 1995. Reports of animal transfer studies were also lacking, but as residues of amitrole in crops are obviously very low and usually below the limit of determination, there is a very limited need for such studies.

RECOMMENDATIONS

No residue limits have been established for amitrole in food commodities but a note states that uses of amitrole should be restricted to those where residues in food would not be expected to occur. The Meeting recommends the addition to this note of the statement: "A realistic limit of determination for the general monitoring of amitrole would be 0.05 mg/kg."

FURTHER WORK OR INFORMATION

Desirable

1. Residue data from supervised trials on apples, pears and grapes known to be in progress.

2. Reports from experiments on the storage stability of amitrole known to be in progress.

REFERENCES

1. Bayer AG. 1982. Metabolismus von Amitrol in Boden und Pflanzen. RA -578/85 B. Unpublished.

2. Burger, K. 1986. Methode zur Bestimmung von Amitrol in Wasser durch Dünnschichtchromatographie (AMD-Methode). Bayer AG, RA-531. Unpublished.

3. Dornseiffen, J.W. and Verwaal, W. 1988. Analysis of blackberries on contamination with amitrole used along railroad tracks. Med. Fac. Landbouww. Rijksuniv. Gent 53/3b, 1519-1530.

4. Drewes, H. and Blume, H.-P., 1976. Abbau, Bewegung und Sorption von Herbiciden in Böden. Landwirtsch. Forsch. Sonderh. 33, 104-113.

5. Franco, L. and Municio, A.M. 1975. Comparative Metabolism of 3-Amino-1,2,4-triazole. Gen. Pharmacol. 6, 163-169.

6. Hawkins, D.R., Kirkpatrick, D., Finn, C.M. and Conway, B. 1982a. The Biodegradation of ^{14}C-Aminotriazole in Soil (Laboratory Studies). Bayer AG. 123/81538. Unpublished.

7. Hawkins, D.R., Kirkpatrick, D., Finn, C.M. and Conway, B. 1982b. The Biodegradation of ^{14}C-Aminotriazole in Soil (Field Studies). Bayer AG. 124/801054. Unpublished.

8. Jarczyk, H.J. 1982. Methode zur gaschromatographischen Bestimmung von 3-Amino-1.2.4-triazol-Rückständen in Äpfeln und Birnen mit N-spezifischem Detektor. Bayer AG. RA-1328/159B. Unpublished.

9. Jarczyk, H.J. 1983. Methode zur gaschromatographischen Bestimmung von 3-Amino-1.2.4-triazol-Rückständen in Boden und Wasser mit N-spezifischem Detektor. Bayer AG. RA-362/182B. Unpublished.

10. Krohn, A. 1986. Hydrolysis of Amitrole in Buffer Solution. Bayer AG. M5179. Unpublished.

11. Løkke, H. 1987. Determination of amitrole by ion-pair high-performance liquid chromatography. J. Chromatog. 200, 234-7.

12. Marschall, G., Kirkwood, R.C. and Martin, D.J. 1987. Studies on the Mode of Action of Asulam, Aminotriazole and Glyphosate in Equesetum arvense (Field Horsetail). II: The Metabolism of [^{14}C]Asulam, [^{14}C]Aminotriazole and [^{14}C]Glyphosate. Pesticide Science 18, 65-77.

13. Schneider, B., Stock, M., Schütte H.R. and Schreiber, K. 1991. Untersuchungen zum Metabolismus von Amitrol in Äpfeln. Bayer AG. HM865. Unpublished.

14. Scholz, K. 1988. Metabolism of Amitrole in Soil under Aerobic Conditions. Bayer AG. M5977. Unpublished.

15. Storherr, R.W. and Burke, J. 1961. Determination of 3-amino-1,2,4-triazole in crops. J. Ass. Off. Anal. Chem. 44, 382-7.

16. Weber, E. 1988. Methode zur Bestimmung von Amitrol-Rückständen in Pflanzenmaterial, Boden und Wasser. Bayer AG, H600. Unpublished.

16. Weller, H. 1987. Leaching characteristics of soil-aged amitrole. Bayer AG. M1229, Unpublished.

AZINPHOS-METHYL

EXPLANATION

Azinphos-methyl was evaluated in 1965 and several times since. In 1991 a re-evaluation resulted in recommendations to withdraw or change several MRLs. New residue data from trials carried out according to GAP were required for apricots, black currants, citrus fruits, strawberries, kiwifruit and bulb and spring onions. The data from trials according to GAP on apricots, citrus fruits and kiwifruit were so limited that withdrawal of the existing MRLs was proposed, and this recommendation was accepted by the 25th Session of the CCPR (1993). Residue data from trials according to GAP on blueberries, cherries and grapes were also desirable as the data available were from only one country, the USA.

Information was provided to the Meeting on registered uses not available at the 1991 JMPR. Data were received from trials on cherries in Denmark and the USA, and on apricots, mandarins and oranges in Spain. The Meeting was informed that data from trials on grapes in Germany and Italy, including processing studies, would be available for the 1995 Meeting.

USE PATTERN

Registered uses of azinphos-methyl which were not available for the re-evaluation in 1991, or have been changed since, are listed in Table 1. Commodities for which no data from trials were supplied are not included.

RESIDUES RESULTING FROM SUPERVISED TRIALS

New data were supplied from trials on cherries in Denmark and the USA and on mandarins, oranges and apricots in Spain.

Table 1. Registered uses of azinphos-methyl.

{PRIVATE } Crop	Country	Application			PHI
		No	kg ai /ha	g ai/hl	days
Citus fruit	Australia	>1	2.0-2.5	25-50	14
	Spain	1-2	2.0-2.5	40-50	15
Pome fruit	Finland		0.25-0.75		21
Stone fruit	Denmark	2	0.75		21
	Spain	2-3	0.6-0.75	40-50	15
Apricot	New Zealand	5-8	max 1.5	50-60	21
Cherry	New Zealand	5-8	max 1.5	50-60	14
Nectarin	Australia	>1 (3-4 weeks)		38-50	14
	New Zealand	5-8	max 1.5	50-60	14
Peach	New Zealand	5-8	max 1.5	50-60	21
Plum	Australia	>1		50	14
Currants	Finland		0.2-0.5		21
Grape	New Zealand	6-9	max 1.0	50	14
Strawberry	Canada	>1	0.53-0.55		5
	Finland		0.2-0.5		21
Kiwifruit	Australia	>1 (3-4 weeks)		40	14

Cherries. Three trials were carried out on cherries in Denmark

at the registered dosage of 0.8 kg ai/ha. All residues were below the limit of determination, 0.04 mg/kg, but samples were taken 59, 81 and 99 days after the last treatment and the PHI in Denmark is 21 days.

Information on many trials on cherries in the USA was supplied in addition to the data reviewed by the 1991 JMPR. In most of the trials the application rate was 0.84 kg ai/ha, and samples were taken after 14 days, which is approximately the registered use in the USA. The trials were carried out over 2 years and in a number of States. Residues after 14-15 days in all trials at 0.84 kg ai/ha were from <0.01 to 2.3 mg/kg with a mean value of 0.60 mg/kg, which supports the MRL of 2 mg/kg proposed by the JMPR in 1991.

Mandarins and oranges. Four trials on mandarins were carried out in Spain. In 3 of them from two experimental stations the dosage applied was 2.4 kg ai/ha, which is within the registered use in Spain, and in the fourth the rate was 4.8 kg ai/ha.

In the trials with 2.4 kg ai/ha the residues after 14 days (the registered PHI in Spain is 15 days although it is being revised) were 0.28-0.48 mg/kg. Even at the rate of 4.8 kg ai/ha residues were within this range. Except for samples taken at day 0, residues decreased only slowly during the periods of the trials.

Residue levels in oranges were about the same as in mandarins. In 2 trials with the registered application rates of 2.2-2.4 kg ai/ha residues were 0.23-0.37 mg/kg after 14 days.

Apricots. Three trials were carried out on apricots in Spain with residues after 14 days at the level of 0.13-0.23 mg/kg, but the dosage used was about 3 times the registered application of 0.6-0.75 kg ai/ha.

Table 2. Residues of azinphos-methyl in cherries from supervised trials in Denmark and the USA. Underlined residues are from treatments approximating registered uses.

{PRIVATE } Country Year	Application			PHI,	Residues, mg/kg	Report
	No	kg ai/ha	g ai/hl	days		
Denmark 1988	1	0.8	400	59 81 99	<0.04 <0.04 <0.04 <0.04 <0.04 <0.04 <0.04 <0.04 <0.04	0335-88 0336-88 0337-88
USA 1984						
(New York)	4	0.56	120	7 14 21	3.7 0.80 0.43	151-GU014-84D
	4	0.84	180	7 14 21	5.0 <u>2.3</u> <u>1.2</u>	151-GU014-84D
(Michigan)	4	0.84	80	0 7 14	2.7 0.14 <u>0.35</u>	855-GU015-84D
USA 1985						

{PRIVATE } Country Year	Application			PHI,	Residues, mg/kg	Report
	No	kg ai/ha	g ai/hl	days		
(California)	2	0.56	140	3 7 14	0.42 0.71 0.90	455-GU030- 85D
	2	0.56	120	7 14	0.45 0.21	456-GU013- 85D
	2	0.56	120	7 14	0.55 0.20	456-GU009-85D
	2	0.56	600	3 7 14	0.36 0.16 0.30	455-GU027-85D
(Washington)	2	0.56	10	7 14 21	0.27 0.09 0.24	454-GU012- 85D
	2	0.56	10	7 14 21	0.18 0.11 0.21	451-GU008-85D
(Oregon)	2	0,56	120	3 7 14	1.6 0.42 0.52	451-GU029-85D
	2	0.56	120	3 7 14	1.2 0.29 0.73	451- GU026- 85D
(Michigan)	3	0.84	290	7 14 21	3.1 0.46 0.17	855- GU015- 85D
	3	0.84	290	7 14 21	0,51 0.29 0.38	855- GU031- 85D
	3	0.84	290	7 14 21	0.21 0.11 0.39	855- GU028- 85D
	3	0.84	1800	7 14	0.05 <0.01	855- GU011- 85D
(New York)	3	0.84	30	7 14	0.41 0.37	151- GU032- 85D
	3	0.84	30	7 14 21	1.5 1.4 0.31	151- GU007- 85D
(Wisconsin)	5	0.84	20	7 15 21	2.1 0.35 0.25	851- GU014- 85D
	5	0.84	20	7 15 21	1.7 0.40 0.20	851 GU010- 85D

Table 3. Residues of azinphos-methyl in mandarins from supervised trials in Spain. Underlined residues are from treatments approximating registered uses.

{PRIVATE } Crop Year	Application			PHI,	Residues, mg/kg	Report
	No	kg ai/ha	g ai/hl	days		
Mandarins/ satsumas 1986 (Algemesi)	>1	4.8	100	0 7 14 21 28	0.52 0.52 0.75 0.41 0.39 0.55 0.32 0.34 0.43 0.29 0.27 0.35 0.23 0.22 0.22	No information
(Pedrequer)	>1	2.4	50	0 7 14 21 28 35	0.42 0.35 0.45 0.38 0.40 0.42 0.30 0.35 0.34 0.25 0.30 0.30 0.21 0.23 0.22 0.18 0.21 0.21	

{PRIVATE } Crop Year	Application			PHI,	Residues, mg/kg			Report
	No	kg ai/ha	g ai/hl	days				
Mandarins/ clementines 1986	>1	2.4	50	0	0.84	0.80	0.65	No information
				7	0.61	0.55	0.43	
				14	0.48	0.35	0.32	
(Algemesi)				21	0.30	0.27	0.22	
				28	0.26	0.24	0.23	
				42	0.20	0.17	0.17	
(Pedrequer)	>1	2.4	50	0	0.53	0.47	0.38	
				7	0.40	0.41	0.36	
				14	0.28	0.36	0.28	
				21	0.24	0.28	0.26	
				28	0.18	0.20	0.23	
				35	0.18	0.20	0.19	
				42	0.13	0.13	0.15	
				56	0.12	0.12	0.12	

Table 4. Residues of azinphos-methyl in oranges from supervised trials in Spain (1986). Underlined residues are from treatments approximating registered uses.

{PRIVATE }Application			PHI,	Residues, mg/kg			Report
No	kg ai/ha	kg ai/hl	days				
>1	2.4	50	0	0.44	0,69	0.40	No infor-mation
			7	0.24	0.34	0.39	
			14	0.27	0.23	0.29	
			21	0.24	0.26	0.16	
			28	0.25	0.33	0.24	
			42	0.21	0.25	0.30	
			56	0.29	0.22	0.22	
>1	2.2	50	0	0.66	0.75	0.69	
			7	0.30	0.38	0.41	
			14	0.27	0.28	0.37	
			21	0.25	0.23	0.27	
			28	0.21	0.22	0.25	
			35	0.28	0.31	0.32	
			42	0.27	0.28	0.20	

Table 5. Residues of azinphos-methyl in apricots from supervised trials in Spain (1988).

{PRIVATE } Application			PHI,	Residues, mg/kg			Report
No	kg ai/ha	kg ai/hl	days				
>1	1.9	50	0	2,1	2.3	2.0	No infor-mation
			3	1.5	1.4	1.6	
			7	0.46	0.80	0.86	
			14	0.13	0.22	0.16	
			21	0.14	0.13	0.08	

APPRAISAL

Azinphos-methyl was evaluated in 1965 and several times since. In 1991 a re-evaluation resulted in recommendations to withdraw or change several MRLs. New residue data from trials carried out according to GAP were required for apricots, black currants, citrus fruits, strawberries, kiwifruit and bulb and spring onions. The data from trials according to GAP on apricots, citrus fruits and kiwifruit were so limited that withdrawal of the existing MRLs was proposed, and this recommendation was accepted by the 25th Session of the CCPR (1993). Residue data from trials according to GAP on blueberries, cherries and grapes were also desirable as the data available were from only one country, the USA.

The Meeting received summarized residue data from Spain from trials on mandarins and oranges according to registered use in Spain. Residue data on apricots were also available from Spain, but the dosage used was about 3 times the registered rate. The Meeting was unable to

recommend MRLs on the basis of the summarized data.

Residue data from trials on cherries were received from Denmark, but samples were taken more than 50 days after the last treatment whereas the registered PHI in Denmark is 21 days. Information was received on several trials on cherries carried out in the USA, which supported the MRL of 2 mg/kg proposed by the 1991 JMPR. The Meeting was informed that data from trials on grapes in Germany and Italy, including processing studies, would be available in 1995.

At the 1991 JMPR a temporary residue limit was proposed for wheat straw and fodder. As no supplementary data were received the Meeting proposes that the temporary limit should be withdrawn.

RECOMMENDATIONS

The residues found in supervised trials did not enable the Meeting to recommend MRLs. The withdrawal of the limit shown below is recommended.

{PRIVATE } Commodity		Recommended MRL (mg/kg)	
CCN	Name	New	Previous
AS 0654	Wheat straw and fodder, dry	W	1 T

W: the previous recommendation is withdrawn.

FURTHER WORK OR INFORMATION

Desirable

1. Detailed information from trials on citrus fruits carried out in Spain.

2. Residue data from trials on citrus fruits from other countries.

BENALAXYL (155)

EXPLANATION

Benalaxyl was first reviewed for residues by the 1986 JMPR which estimated Guideline Levels for several commodities and desired information on:

1. Residues in meat from pigs and cattle fed a diet containing benalaxyl
2. The effect of processing on residues in crops.
3. Information on national MRLs.
4. Additional information on levels of metabolites in plants after treatment with benalaxyl.

Guideline levels were changed to recommendations for MRLs when an ADI was allocated by the 1987 JMPR. Over several years CCPR delegations have questioned various limits, in particular in grapes (considered by some to be too high), potatoes (the 0.01 mg/kg limit of determination and the lack of a published method were questioned), hops, and tomatoes. The submission of data, including information on metabolites, was promised. The 1990 CCPR lowered the proposed grape limit from 0.5 to 0.2 mg/kg. Additional GAP information and summarized residue data were reviewed by the 1992 JMPR. The information was not adequate for revising limits, although it suggested the need for an increased limit for lettuce. The submission of additional unspecified data was promised.

Several submissions were made to the Meeting in response to the 1986 requests or the concerns expressed at the CCPR. These included current GAP information, national MRLs and residue data on several commodities (mainly on grapes, wine and must, but some on potatoes, tomatoes and cucumbers). Information was also provided on residues of conjugates in wine, and a published analytical method which had been considered desirable at the CCPR. Comments were provided on other items.

USE PATTERN

Information on current uses of benalaxyl on a number of crops is summarized in Table 1

Table 1. Nationally approved or registered uses of benalaxyl on selected crops.

Crop/ Country	Application			PHI (days)	Notes
	Formulation	Rate g ai/ha (g ai/hl)	No.		
Cucurbits					
Australia	WP ?[1]*	200 l. vol. (20) h. vol.	**	7	*Mixed formulation 80 g benalaxyl, 650 g mancozeb/kg. **2-spray sequence full coverage at 7-10-day intervals.
Austria	WP ?*	160		3*	*PHI for same mixed formulation as Australia
Hungary	?*	200-240		5*	*PHI for co-formulation with mancozeb
Spain				15	
melons, watermelon	WP*	160-240* (16-24)*			*Field or glasshouse
Grapes					
Australia	WP ?*	152-224 l. vol. (22.4) h. vol.	**	14	*See cucurbits **2-spray sequence at 7-21-day intervals. High vol. ≥500 l/ha before flower, 1000 l/ha at full foliage.
Greece	?	(12-16)	*	*	*Last treatment after blooming
Hungary	?	160-200	4*	30**	*At 14-day intervals **PHI for co-formulations with mancozeb
Italy	SC and ?	(16-20)	4*	7	*at 14-day intervals
Spain	WP*	160-240 (16-24)	-	30	*mixed formulation, 80 g benalaxyl, 650 g mancozeb/kg.
Pepper					
Spain	WP*	160-240 (16-24)	-	15	*mixed formulation, 80 g benalaxyl, 650 g mancozeb/kg.
Potatoes					
France	WP and ?	200 (20)	3*	-	*at 10-14-day intervals
Italy	SC and ?	(20-24)	3*	7	*at 14-day intervals
Greece	?	200-240	5*	28	*at 14-day intervals with co-formulation
Spain	WP*	160-240 (16-24)	-	30	*mixed formulation, 80 g benalaxyl, 650 g mancozeb/kg.
Tomatoes					
Italy	SC and ?	(20-24)	*	7	* at 12-14-day intervals
Spain	WP*	160-240 (16-24)	-	15	* mixed formulation, 80 g benalaxyl, 650 g mancozeb/kg

[1] Except for the Australian label which did not indicate the type of formulation a "?" indicates that labels were not in English and the summary translations did not include the type.

RESIDUES RESULTING FROM SUPERVISED TRIALS

In plants

The 1992 JMPR reviewed supervised trials data for several crops. It did not propose new or revised limits, but was informed that additional data would be provided. In addition to substantial supervised trials data on grapes, the present Meeting has received summary data on benalaxyl residues in cucumbers, potatoes and tomatoes. Because summary data without accompanying detailed reports are not suitable for estimating maximum residue levels, the Meeting did not further consider these summaries (ISAGRO, 1993). The Meeting also received additional information on levels of metabolites in plants.

Grapes. The current 0.2 mg/kg CXL for grapes was proposed by the 1990 CCPR, although the 1986 JMPR had estimated a 0.5 mg/kg Guideline Level. The change was based on the observation that German data (0.7 mg/kg maximum residue after 14 days) did not reflect GAP because the use was not approved

in Germany. Other residues were ≤0.22 mg/kg after 7 days. Substantial additional data from more recent supervised trials were provided to the Meeting, although many of the results do not strictly reflect GAP. Residue data from supervised trials on grapes are summarized in Table 2.

Of the 35 trials summarized in Table 2, 17 were Italian, 8 German, 2 Greek, 6 Australian and 2 Hungarian. Only two of the Italian trials were fully within GAP in terms of the number of applications and spray concentrations and these were at longer PHIs than the 7-day Italian GAP PHI (the shortest PHI in any of the trials was 11 days). The highest residue in these two trials was 0.2 mg/kg after 21 days. Using these results with a residue decline curve provided, a maximum residue of approximately 1.8 mg/kg after 7 days can be estimated. Other Italian trials involving 5 applications (instead of the 4 allowed by GAP) at GAP rates resulted in maximum residues of 0.6 mg/kg after 11 days (1.3 mg/kg after 7 days can be estimated from a residue decline curve). In other Italian trials with 4 applications, but at 32 g ai/hl instead of the approved maximum of 20 g/hl, the maximum residues were 0.09 mg/kg after 14 days (1.2-1.3 mg/kg after 7 days can be estimated from a residue decline curve).

Only two of the German trials approximated Italian GAP in terms of spray concentration (all the trials were at a higher rate/ha than is allowed by the GAP of other countries), with maximum residues of 0.5 and 0.6 mg/kg after 27/28 days (0.9-1 mg/kg after 7 days was estimated from a residue decline curve). The Meeting was not told whether this use is now GAP in Germany, although as noted above the 1990 CCPR was informed that it was not at that time.

The Australian trials do not reflect Australian GAP (1, 7 or 8 applications compared with the 4 approved). The highest residues at the Australian 14-day PHI were 0.2 mg/kg. In the Hungarian trials the highest residues after the longest sampling interval of 21 days were only 0.01 mg/kg. The approved Hungarian PHI is 30 days.

In the Table 2 trials in which wine, juice or must samples were also analyzed the maximum residues were 0.02 mg/kg in juice and must and 0.01 mg/kg in wine. In addition to the wine data summarized in Table 2, the Meeting was provided with the results of 6 other trials in which wine was made from similarly treated grapes. The residue levels in the treated grapes were not given; those in the wine ranged from not detected (<0.01 mg/kg) to 0.02 mg/kg. The interval from grape sampling to wine analysis was 1 to 2 years in these studies.

Table 2. Residues of benalaxyl in grapes and grape products resulting from application of a WP formulation in supervised trials.

Country/ Year/(Variety)	Application	Residues, mg/kg at intervals (days) after last application	Ref.

	No.	Rate kg ai/ha (g ai/hl)	Days / Residue				
Italy 1991			0	7[1]	11	38	56	82	
(Merlot)	5	0.28 (**20**)	3	1.3[2]	0.6	0.08	0.05	0.03	9/91/VIN
			0		22	44	66	77	
(Chardonnay)	**4**	0.22 (100)	0.9		0.05	0.02	≤0.01	≤0.01	7/91/VIN
1990						27	37	94	
(Barbera)	4	0.2 (50)				0.1	0.08	≤0.01	11/90/WIN
			Must and wine ≤0.01						
							54	114	
(Barbera)	4	0.16 (40)					0.03	≤0.01	12/90/WIN
			Must and wine ≤0.01						
1991			0	7[1]	21	43	65	90	
(Barbera)	4	2X 0.11 (20) + 2X 0.22 (20)	2.5	1.6	0.1	0.08	0.03	≤0.01	1/91/WIN
			Must and wine ≤0.01						
1990					27	37		80	
(Riesling)	4	0.2 (50)			0.3	0.2		0.02	13/90/WIN
			Must and wine ≤0.01						
							54	106	
(Riesling)	4	0.16 (40)					≤0.01	≤0.01	14/90/WIN
			Must and wine ≤0.01						
1991			0	7[1]	21	43	65	90	
(Riesling)	4	2X 0.11 (20) + 2X 0.22 (20)	2.8	1.8	0.2	0.1	0.04	0.03	2/91/WIN
			Must and wine ≤0.01						
1990								78	
(Merlot)	4	3X 0.15 (16) + 0.18 (16)						0.06	7/90/WIN
			Must and wine ≤0.01						
1991			0	7[1]	11	38	56	82	
(Merlot)	5	0.28 (20)	2.2	0.9	0.4	0.05	0.03	0.02	8/91/VIN
			Must and wine ≤0.01						
1990								79	
(M. Palieri)	2	0.16 (40)						≤0.01	8/90/VIN
1991			0	7[1]	14	30	48	69	
(M. Palieri)	4	0.16 (32)	2.8	1.3	0.09	≤0.01	≤0.01	≤0.01	3/91/VIN
1990								79	
(Italia)	2	0.16 (40)						≤0.01	9/90/VIN
1991			0	7[1]	14	30	48	69	
(Italia)	4	0.16 (32)	1.4	0.7	0.04	0.01	0.01	≤0.01	4/91/VIN
1990								79	
(Regina)	2	0.16 (40)						≤0.01	10/90/VIN

Table 2 (contd.)

Country/ Year/ (Variety)	Application		Residues, mg/kg at intervals (days) after last application	Ref.
	No.	Rate, kg ai/ha (g ai/hl)	Days / Residue	
Italy contd 1991 (Regina)	4	0.16 (32)	0 7[1] 14 30 48 69 2.6 1.2 0.09 0.01 ≤0.01 nd (<0.01)	5/91/VIN
(Chardonnay)	4	0.22 (100)	0 22 44 66 77 0.6 0.07 0.02 ≤0.01 ≤0.01 Must and wine ≤0.01	6/91/VIN
Germany 1986 (Portugieser)	4	0.32-0.48 (20)[3]	0 7[1] 27/28 42 56-59 69 1.1 1 0.6 0.3 0.14 Juice or past. juice 0.01 Wine ≤0.01 Pasteurized wine ≤0.01(95 days)	8/86/VIN 9/86/VIN
(Huxelrebe)	4	0.32-0.48 (80)[3]	2.3 0.6 0.3 0.04	10/86/VIN
(Müller-Thurgau)	4	0.32-0.48 (80)[3]	1.3 0.6 0.3 0.4	11/86/VIN
(Ruländer)	4	0.32-0.48 (80)[3]	2 0.41 0.3 0.2 Juice or past.juice 0.02 84 days Wine or past. wine ≤0.01	12/86/VIN 13/86/VIN
1987 (Müller-Thurgau)	4	0.32-0.48 (20)[3]	63 days 1.1 0.9 0.5 0.2 0.2 0.2 Must or past. must ≤0.01 Wine ≤0.01 (94 days) Past. wine ≤0.01 (83 days)	1/87/VIN 2/87/VIN 3/87/VIN
(Lemberger)	4	0.32-0.48 (80)[3]	2.3 0.5 0.4 0.3 Must or past. must 0.02 Wine or past. wine 0.01 (92 days)	4/87/VIN
(Morio-Muskat)	4	0.32-0.48 (80)[3]	1.8 0.3 0.2 0.3 0.2 (68 days)	5/87/VIN
(Ehrenfelser)	4	0.32-0.48 (80)[3]	1.5 0.5 0.5 0.3	6/87/VIN

Table 2 (contd.).

Country/Year/ Variey	Application		Residues, mg/kg at intervals (days) after last application	Ref.
	No.	Rate kg ai/ha (g ai/hl)	Days Residue	
Greece 1986 (Savatiano)	4	0.04-0.125 (16)	100 106 0.01 0.03	7/86/VIN 8/87/VIN
Australia 85 (Semillon)	7[4]	0.23 (15)	0 2 7 10 14 0.2 Wine 0.01	1/86/VIN
	7[4]	0.3 (20)	0.2 Wine 0.02	2/86/VIN
1986-87 (Cabernet Sauvignon)	8[4]	0.3 (20)	0.07 Wine must 0.02	5/86/VIN,3/86/VIN
	8[4]	0.36 (24)	0.2 Wine must 0.02	6/86/VIN, 4/86/VIN
1985 (b. Muscat)	1	0.6 (55)	0.8 0.6 0.4 0.2 0.09	1/85/VIN
	1	0.28 (28)	0.4 0.3 0.2 0.1 0.05	2/85/VIN
Hungary 1983	1	0.26 (32)	0 1 3 5 7/8 10/12 15/16 21 0.7 0.6 0.2 0.09 0.05 0.02 0.01 (Limit of detection 0.01)	2/83/VIN 3/83/VIN
	1	0.22 (28)	0.6 0.5 0.4 0.1 0.06 0.03 0.01 (Limit of detection 0.005)	

[1] No 7-day results were provided. Levels listed were interpolated from residue decline curves from the subject trial.

[2] Only references 1/91/WIN and 2/91/WIN of the Italian trials and the Greek and Hungarian trials closely reflect GAP. Results from these trials and from other trials most closely reflecting GAP are underlined, although in some cases the number of applications or the application rate exceeds GAP at least slightly. The 7-day interpolated values which would approximately reflect GAP are also underlined.

[3] Exaggerated rates and/or concentrations.

[4] Exaggerated number (GAP is 4).

Metabolite levels in plants. The manufacturer commented on unsuccessful efforts to determine the GX1a and GX1b glucoside metabolites in crops in response to a 1986 JMPR request. In plants these are formed by oxidation of a xylyl methyl group to -CH_2OH and subsequent conjugation with glucose. Information was provided on a method for the determination of residues of benalaxyl GX1 (unresolved mixture of GX1a and GX1b) glucosides in white wine by liquid chromatography (Agrimont, 1991). The method was not successful with red wine owing to interferences. See Methods of residue analysis for a description of the method. No data were provided except results of recovery studies.

In animals

The 1986 JMPR listed as desirable information on residues in meat from pigs and cattle fed a diet containing benalaxyl. No data were provided. The manufacturer expressed the view that residues in the meat of cattle and pigs are extremely unlikely, on the basis of the results of metabolism studies and the low residues likely to be in potential feed items (tomato MRL 1 mg/kg, grape MRL 0.1 mg/kg). In metabolism studies reviewed by the 1986 JMPR residues of the parent compound and metabolites were ≤0.1 mg/kg in muscle tissues of hens and goats fed with 50 ppm benalaxyl in the feed, up to 1.8 and 1 mg/kg in the respective livers and up to 1 and 0.4 mg/kg in the kidneys. Residues were up to 0.05 and 0.3 mg/kg in egg white and yolk respectively and <0.01 mg/kg in goat milk.

FATE OF RESIDUES

In storage and processing

The 1986 JMPR listed information on the effect of processing on crops as desirable. No information was provided except on residues in wine and must from supervised trials on grapes.

METHODS OF RESIDUE ANALYSIS

The 1986 monograph described a method by Farmoplant for the determination of benalaxyl *per se* based on acetone extraction, clean-up by liquid-liquid partitioning and column chromatography, and GLC with detection by an AFID. The method was applied to a number of commodities and a 0.01 mg/kg limit of determination was reported, although recoveries were determined only at 0.05 to 0.1 mg/kg. Concern was expressed at the CCPR at the lack of a published method suitable for enforcement, and the reported limit of determination was questioned. In response to these comments the manufacturer provided a recently published method for the determination of benalaxyl in crops and water (Crisippi and Zini, 1993).

The published method is similar to that previously described. Chopped crops are extracted with acetone and the residues partitioned into hexane. Filtered must and wine are eluted through an "Extrelut" column with hexane. Filtrates from both types of sample are further cleaned up by elution through an alumina column with 9:1 hexane:acetone and analyzed by GLC with NP detection. Recoveries of ≥95% with SDs of ≤6.5 were reported for several sample types at the following fortification levels:

Sample	Fortification Level (mg/kg)
grape	0.04-1.1
tomato	0.11-0.22
potato	0.01-0.3
tobacco	0.11-1.1
rapeseed	0.11-1.1
pineapple	0.04
	mg/l
wine	0.01-0.8
must	0.04-0.42
water	0.001-0.1

Blank contributions ranged from non-detectable (<0.003 mg/kg) in potatoes to 0.01 mg/kg in pineapples. Detection was possible at 0.003 mg/kg in potatoes and down to 0.1 ìg/l in water. The authors considered the limit of quantification to be 0.01 mg/kg in crops (0.01 mg/l in wine or must) and 0.1 ìg/l in water.

An analytical method was also provided for the determination of the glucoside metabolites GX1a and GX1b in white wines (Agrimont, 1991). It is

based on passing the wine through an "Extrelut" column, elution of the residues with methylene chloride, concentration of the sample and clean-up on a Florisil column by successive elution with acetone, 90:10 acetone:methanol and finally 80:20 acetone:methanol. The last eluate is concentrated and analyzed by HPLC with UV detection. Recoveries ranged from 78 to 86% from fortifications at 0.25 and 0.025 mg/l. A limit of detection of 0.01 mg/l was reported. Chromatograms suggest that routine analyses down to 0.05 mg/kg should be feasible.

A method has also been described for the determination of benalaxyl in wine (Agrimont, 1990). It is based on adsorbtion on an "Extrelut" column, elution with n-hexane, evaporation of solvent, application of the redissolved sample to an alumina column, elution with n-hexane:acetone (9:1 v/v), concentration and GLC determination with an AFID. Analytical recoveries were >92% from 0.01 to 1 mg/kg fortification levels. Sample chromatograms suggest that routine analyses at 0.02 to 0.05 mg/kg should be feasible, with detection down to 0.01 mg/kg.

NATIONAL MAXIMUM RESIDUE LIMITS

The following national MRLs were reported to the Meeting.

Commodity/Country	MRL (mg/kg)
All commodities	
The Netherlands	0* (0.02)
	Under consideration
Grapes	
France, Switzerland	0.1
Australia, Italy, Spain, Venezuela	0.5
Portugal	7.5
Lettuce	
Australia	0.01
Spain	0.1
Venezuela	0.5
Peru	1
Melons	
Australia	0.2
Italy, Spain, Venezuela	0.5
Onions	
Switzerland	0.01
Australia	0.1
Italy, Spain, Venezuela	0.5
Peru	1
Peppers	
Australia	0.2
Hungary, Italy, Spain, Venezuela	0.5
Potatoes	
Belgium, Great Britain, Hungary, Switzerland	0.01
France, Germany	0.02
Italy, Spain, Venezuela	0.05
Peru	1
Portugal	7.5
Strawberry	
Hungary, Italy	0.1
Tomato	
Hungary, Italy, Spain	0.5
Peru	1

APPRAISAL

Benalaxyl was first reviewed for residues by the 1986 JMPR, which estimated Guideline Levels and listed desirable information. Guideline Levels were changed to MRLs when an ADI was allocated by the 1987 JMPR. Over several years various limits (in particular on grapes) were questioned at the CCPR. Submission of additional unspecified data has been promised. Several submissions were made to the Meeting in response to requests of the 1986 JMPR or concerns expressed at the CCPR, some with and some without the detailed reports.

Grapes. The 0.5 mg/kg limit estimated by the 1986 JMPR was lowered to 0.2 mg/kg by the 1990 CCPR. Although no outstanding issues remained, extensive recent data from the use of benalaxyl on grapes were provided to the Meeting. Much of the summarized information could not readily be related to the more detailed reports provided owing to its code numbering format, except in the case of the Italian data.

Most of the submitted grape data do not closely reflect reported current GAP. In particular, most of the results (except at day 0) were at intervals significantly longer than the reported 7-day Italian GAP PHI (the shortest non-0-day PHI was 11 days and most PHIs were longer). The few results within GAP (GAP rates and $\geq$ 11 day PHI) were consistent with the current 0.2 mg/kg CXL. However, extrapolation from Italian residue decline curves strongly suggests that residues exceeding 1 mg/kg are likely to occur from Italian GAP at a 7-day PHI. Extrapolation of previously provided and additional data from German supervised trials also suggests that residues may approach 1 mg/kg when related to Italian GAP. However, the Meeting was informed that the manufacturer is to request that the 7-day Italian PHI be revised to 10-28 days. With that revision residues would be within the current limit. The Meeting was also informed that applications are only on small immature fruit.

Residues in must and wine were $\leq$0.02 mg/kg, mostly $\leq$0.01 mg/kg. No data were provided for grape pomace, a possible animal feed item.

Potatoes. The adequacy of previously submitted analytical methods to support the current 0.01 mg/kg CXL for potatoes has repeatedly been questioned at the CCPR. The Meeting concluded (see below) that 0.02 mg/kg is a reasonable limit of determination for the new analytical method reported, and noted that the limit of "detection" for much of the additional summary data reported (but not reviewed) is 0.02 mg/kg. The Meeting therefore proposed that the MRL should be increased to 0.02 mg/kg.

In addition to substantial supervised trials data for grapes, the Meeting received summary data on benalaxyl residues in cucumber, potatoes and tomatoes. Because summary data without accompanying detailed reports are not suitable for estimating maximum residue levels the Meeting did not review these summaries apart from considering the limit of determination for potatoes. The Meeting was informed that the detailed reports would be submitted for review at a future meeting.

The Meeting also received a limited response to the 1986 request for additional information on levels of metabolites in plants. Noting unsuccessful efforts to analyze the GX1a and GX1b glucoside metabolites in crops, the Meeting was informed of a method for the determination of these metabolites in white wine (unsuccessful in red wine). No data were provided except the results of recovery studies.

Residues in animal products. In response to a JMPR request for information on residues in cattle and pigs the manufacturer expressed the view that metabolism studies and the low residues expected in feed items would make residues in meat from cattle and pigs unlikely. Since (1) significant residues could occur: they have been found in the offal of goats and hens in metabolism studies (e.g. up to 1 and 1.8 mg/kg in the liver of goats and hens fed at 50 ppm in the feed); (2) information on the possible concentration of residues in feed items derived from processing was lacking; and (3) the duration of the metabolism study feeding periods (7

days for cattle, 14 days for hens) was relatively short, the Meeting could not with certainty come to the same conclusion. While the Meeting agreed that residues in animals would be likely to be low, there is the potential for finite residues.

Processing. Apart from data on residues of benalaxyl in wine and must, no information was provided in response to the 1986 JMPR request for information on the effect of processing on residues in crops. Processing studies would also provide insight into the likelihood of residues in animal products. The Meeting was informed that processing studies would be scheduled for 1994.

Analytical methods. A published analytical method based on acetone extraction, liquid-liquid partitioning, alumina clean-up and GLC with NPD detection was provided in response to CCPR concerns that no published enforcement method was available and doubt concerning the reported 0.01 mg/kg limit of determination in potatoes in the method previously reviewed. The published method was tested on several crops, wine, must and water. Recoveries of $\geq$95% were reported.

The Meeting received excellent documentation of what appears to be a suitable enforcement method. While the reported limits of determination (0.01 mg/kg in crops and 0.01 mg/l in wine and must) may be attainable in the author's laboratory, on the basis of sample chromatograms, reported control values and fortification levels, the Meeting concluded that a more realistic limit of determination for Codex purposes would be of the order of 0.05 mg/kg in crops (0.02 mg/kg in potatoes) and 0.05 mg/l in wine and must. Detection is possible at lower levels.

A description of an analytical method for the determination of the glucoside metabolites GX1a and GX1b in white wines was also provided (chromatograms suggest that routine analyses down to 0.05 mg/kg should be feasible). An analytical method based on column chromatography clean-up and GLC with AFID detection for determining benalaxyl in wine was also provided, with the capability of analyses at 0.02 to 0.05 mg/kg.

RECOMMENDATIONS

On the basis of the reported data on analytical methods the Meeting concluded that the residue level is suitable for establishing an MRL.

Definition of the residue: benalaxyl

Commodity		Recommended MRL (mg/kg)	
CCN	Name	New	Previous
VR 0589	Potato	0.02*	0.01*

*At or about the limit of determination.

FURTHER WORK OR INFORMATION

Desirable

1. Submission of revised Italian GAP for grapes for the next scheduled review.

2. Submission of detailed reports of trials on cucumber, potato and tomato corresponding to summary data provided to the 1993 Meeting, reported in a manner to permit easy comparison of the summary data and the detailed reports and in the working language of the Meeting.

3. Submission on completion of processing studies which are scheduled for 1994.

(From 1986 JMPR)

4. Information on residues in meat from pigs and cattle fed a diet containing benalaxyl.

REFERENCES

Agrimont, 1990. Determination of Benalaxyl Residue in Wine. Unpublished Agrimont report, July 16, 1990. Appendix E to ISAGRO report Annex 4, Fabbrini, 1992b (see below).

Agrimont, 1991. Determination of Residues of the Main Glucoside Conjugates of Benalaxyl in Wine. Report ZINI-13:X1VINO. ISAGRO Annex 12, July 29, 1991. The diasterioisomer GX1, i.e. methyl *N*-phenylacetyl-*N*-(2-glucopyranosylmethyl-6-methyl)phenyl-DL-alaninate was analysed.

Crisippi, T and Zini, G. 1993. Gas Chromatographic Determination of Benalaxyl Residues in Different Crops and Water. JAOAC International, *76*, 650-6.

Fabbrini, R. 1992a. Benalaxyl Residue Determination in Grape and Must Samples. Larpest 1992 Project AG-04/91. Enichem Final Report. May 28, 1992. ISAGRO Annex 3. Includes data for monograph Table 2 references 5/86/VIN (?), 1/91/WIN, 2/91/WIN and 7/90/WIN.

Fabbrini, R. 1992b. Benalaxyl Residue Determination in Wine Samples. Larpest 1992 Project IS-01/92. ISAGRO Final Report. July 21, 1992. Annex 4.

Fabbrini, R. 1993a. Benalaxyl Residue Determination in Grape Samples, Volume I. Larpest Project IS-02/92, January 25, 1993. ISAGRO Annex 1. Includes data for monograph Table 2 references 3/91/VIN and 4/91/VIN.

Fabbrini, R. 1993b. Benalaxyl Residue Determination in Grape Samples, Volume II Parts I and II (gas chromatograms of the Annex 1 study), Larpest Project IS-02/92. January 25, 1993. ISAGRO Annex 2.

ISAGRO*, 1993. (* Formerly Farmoplant, Agrimont, Enichem Agricoltura). Several volumes of Benalaxyl Unpublished information and data (some with and some without cited dates or authors) provided to the 1993 JMPR:

Report on Benalaxyl. Comments on various JMPR required and desirable information. 12/2/93 ISAGRO fax to FAO.

Analysis of Benalaxyl Residues in Wine. ISAGRO Final Report 2083, February 9, 1993. Includes Data references 10/91/WIN; 11/91/WIN; 12/91/WIN and 13/91/WIN (not included in Monograph Table 2 references).

Analysis of Benalaxyl Residues in Vine (grapes). ISAGRO Final Report 2085, February 9, 1993. Includes data for Monograph Table 2 references 8/91/VIN and 9/91/VIN.

Analysis of Benalaxyl Residues in Vine (grapes). ISAGRO Final Report 2087, February 9, 1993. Includes data for Monograph Table 2 references 6/91/VIN and 7/91/VIN.

Benalaxyl Residues Resulting from Supervised Trials - Grapes, Wine - (1) Italy (3) Germany (4) Greece (6) Australia (7) Hungary. ISAGRO Annex 5 Report. Summary Tables for monograph Table 2 references, plus wine sample references 1/89/VIN, 2/89/VIN, 6/90/VIN, 3/90/WIN, 4/90/WIN and 5/90/WIN (residues <0.01 to 0.018 mg/kg after 82 to 90 days) not included in monograph Table 2. Summarizes data from ISAGRO reports 2083, 2085, 2087, Annex 1, Annex 3 and probably others, although this was not readily discernible from the reports provided.

Benalaxyl Residues Resulting from Supervised Trials - Potatoes - (1) Italy (2) France (4) Greece. ISAGRO Annex 7.

Benalaxyl Residues Resulting from Supervised Trials - Tomatoes - (1) Italy (2) Greece. ISAGRO Annex 8.

Benalaxyl Residues Resulting from Supervised Trials - Tobacco - (3) Poland. ISAGRO Annex 10.

Benalaxyl Residues Resulting from Supervised Trials - Cucumber - (1) Austria (2) Australia (3) Hungary. ISAGRO Annex 11.

National MRLs. ISAGRO Annex 6.

BROMOPROPYLATE (70)

EXPLANATION

Bromopropylate was previously evaluated by the Joint Meeting in 1973. It was scheduled by the Codex Committee on Pesticide Residues for periodic re-evaluation at the 1993 JMPR (ALINORM 93/24A, para 93). An ADI of 0.008 mg/kg bw/day had been established and MRLs had been recommended for a range of food commodities.

In the last twenty years the use of bromopropylate has been extended to other crops and some uses have been discontinued. Additional residue trials data on crops such as artichokes, beans, celery, cucurbits, guava, onions, papaya, peaches, peanuts, peas, sweet peppers, pineapple and sugar beet have been submitted by the manufacturer and authorities in Spain and The Netherlands. The manufacturer had also submitted additional plant metabolism studies.

USE PATTERN

Bromopropylate is a contact acaricide (miticide). It is formulated as an emulsifiable concentrate at concentrations of 50% w/w and 25% w/w. The major crops on which it is recommended are pome and stone fruits for the control of mites such as the European red mite, two-spotted mite, carmine mite, apple rust mite and bryobia mite. It is also recommended on citrus, grapes, berries, cotton, hops, sugar beet, sugar cane, tea, ornamentals and certain vegetables.

Bromopropylate is registered or approved for use in many countries. However in Australia, although registered for use on pome fruits and stone fruits, it has not been marketed since 1986. Present indications in Australia are that it will not be returning to the market because of commercial factors, resistance problems and lack of compatibility with Integrated Pest Management systems. In Malaysia, bromopropylate is registered for use only on ornamentals. Its use for the control of varroa mites in honey bee colonies has caused some concern in The Netherlands. Details of the approved national recommendations in various crops were provided by the manufacturer, Spain, The Netherlands and Australia, and are summarized in Table 1.

Table 1. Registered uses of bromopropylate. All applications are foliar.

Crop	Country	Form.	No. of appl.	Application rate per treatment	PHI, days	Comments

				kg ai/ha	g ai/hl		
Apple/ pome fruit	Australia	500 EC	-		50-75	21	
	Belgium	500 EC	2		37.5-50	56	greenhouse
	Chile	500 EC	1		50-60	21	
	France	250 EC	1-2		50+3	15-28	mixed with bifenthrin
	Hungary	500 EC	1-2	0.75	75	30	
	Israel	500 EC	1		50-70	7	
	Italy	250 EC	1		39.3-52.4	21	
	Japan	450 EC	2		22.5-30	21-30	
	S. Korea	450 EC	3-4		45	21	
	N'lands	500 EC	-	0.65-0.98	65	21	
	New Zealand	500 EC	1-2		90	14	
	Peru	500 EC	HV 2-3	0.65-1	37.5-50	14	
	Poland	500 EC	-	0.57-0.75		21	
	Portugal	500 EC	1	0.5	50	21	
	Spain	500 EC	2-3	1.5	50-100	21	fruiting stage
	Switzerland	250 EC	>1		37.5	21	
	Turkey	500 EC	1-2		50	21	
	Uruguay	500 EC	1-2		37.5-50	21	
Apricot	Australia	500 EC	HV/LV		50-75	21	
Artichoke	Spain	500 EC	1-2	0.5-1.5	50-100	7	
Beans/Peas	Belgium	500 EC	2		37.5-50	14	greenhouse
	Israel	500 EC	1	0.75-1.0		3	
	Italy	250 EC	1		39.3-52.4	21	
	Peru	500 EC	HV 2	0.15-0.2	37.5-50	14	
	Spain	500 EC	2-3	0.5-1.5	50-100	7	fruiting stage
Berries	Switzerland	250 EC	>1		37.5	21	
Black currant	New Zealand	500 EC	1-2	0.5-0.75		14	
Cane fruit	New Zealand	500 EC			90	14	
Citrus	Brazil	500 EC		0.8	40	14	flowering stage
					75	14	
	Chile	500 EC	1		50-60	21	
	China	500 EC	2-3		33.3-50	14	
	Cuba	500 EC		0.25-0.3		5	
	Honduras	500 EC	2		50	15	
	Iran	250 EC	1-2		25	21	
	Israel	500 EC	1		15-25	21	
	Italy	250 EC	>1		39.3-52.4	21	
	Japan	450 EC	2		22.5	30	
	Jordan	500 EC	1-2		100-125	14	
	Kenya	500 EC	2	0.375	37.5	21	
	S. Korea	450 EC	3-4		45	30	
	Mozambique	500 EC	2		15-50	21	

Crop	Country	Form.	No. of appl.	Application rate per treatment		PHI, days	Comments
				kg ai/ha	g ai/hl		
	Peru	500 EC	HV 2	0.5-0.73	25-37.5	21	
	Portugal	500 EC	2	0.5	50	28	
	Spain	500 EC	1	2-6	50-100	21	fruiting stage
	Taiwan	250 EC	2	0.25-0.125		21	
	Thailand	250 EC			37.5-50	21	
	Turkey	500 EC	1-2		50	21	
	Uruguay	500 EC			15-50	21	
	Venezuela	500 EC	2-3		10-15		
	Zimbabwe	500 EC	1	0.5-1.5	12.5	10	
		500 EC	1	1.5-2		10	Aerial
Cotton	Brazil	500 EC	2-3	0.4-1.0		28	
	Colombia	500 EC			100	20	
				0.75-1.0		20	Aerial
	Kenya	500 EC	2	0.75	37.5	14	
	Pakistan	500 EC	HV 1	0.75		15	
	Peru	500 EC	HV 1	0.2	50-75	14	
	Spain	500 EC	2-3	0.5-1.0	50-100	21	all stages
	Turkey	500 EC	1	0.5-1.0		21	
	Uruguay	500 EC	1-2	0.5		14	
Cucurbits	Belgium	500 EC	2		50	14	
		500 EC	2		37.5	14	greenhouse
	France	250 EC	1-2		50	15	melon
	Italy	250 EC	>1		39.3-52.4	21	cucumber, melon, zucchini
	Japan	450 EC	2		22.5	7	
	Poland	500 EC	2		25-50	4	
	Spain	500 EC	1-2	0.5-1.5	50-100	7	fruiting stage
Currants	Poland	500 EC		0.75		21	
Egg plant	Israel	500 EC	1	0.75-1.0		3	
	Italy	250 EC	>1		39.3-52.4	21	
	Japan	450 EC	2		22.5	7	
	Spain	500 EC	1-2	0.5-1.5	50-100	7	fruiting stage
Fruits	Austria	500 EC		0.25-0.5	37	21	
	Iran	250 EC	1-2		25	21	
	Pakistan	500 EC	HV 1-2	0.75		15	
Grapes	Chile	500 EC	1		50-60	21	
	France	250 EC	1-2		50	15	
		250/15 EC			37.5+ 2.25	28	mixed with bifenthrin
	Hungary	500 EC	1-2	0.75	75	30	
	Israel	500 EC	2		50-75	7	
	Italy	250 EC	>1		39.3-52.4	21	

Crop	Country	Form.	No. of appl.	Application rate per treatment		PHI, days	Comments
				kg ai/ha	g ai/hl		
	Jordan	500 EC	2-3		100-125	14	
	Portugal	500 EC	2	0.5	50	21	
	Spain	500 EC	1-2	0.6	50-100	21	fruiting stage
	Switzerland	250 EC	>1		37.5	21	
	Thailand	250 EC			37.5-50	21	
	Turkey	500 EC	1-2		50	21	
	Venezuela	500 EC	>1		10-15		
Hops	Belgium	500 EC	2		37.5-50	56	
	Japan	450 EC	2		22.5	60	
Nectarines	Australia	500 EC	HV/LV		50-75	21	
Peach	Australia	500 EC	HV/LV		50-75	21	
	Chile	500 EC	1		50-60	21	
	Italy	250 EC	>1		39.3-52.4	21	
	Japan	450 EC			22.5	14	
	New Zealand	500 EC	1-2		90	14	
	Portugal	500 EC	2	0.5	50	28	
Peppers (sweet)	Belgium	500 EC	2		37.5-50	14	
	Poland	500 EC	2		25-50	4	
Plums	Australia	500 EC	HV/LV		50-75	21	
	N'lands	500 EC		1	65	21	
Raspberry	Poland	500 EC	-	0.75		21	
Strawberry	Belgium	500 EC	2		50	14	3d PHI for continuous culture
		500 EC	2		37.5	14	
	Italy	250 EC	>1		39.3-52.4	21	
	New Zealand	500 EC	1-2	0.5-0.75		14	
	Poland	500 EC	-	1.5			
	Spain	500 EC	2-3	0.5-1.5	50-100	14	
	Thailand	250 EC	>1		37.5-50.0	21	
	Venezuela	500 EC	2-3		10-15	-	
Stone fruits	Switzerland	250 EC	>1		37.5	21	
	Spain	500 EC	1-2	1.5	50-100	21	
Sugar beet	Iran	250 EC	1	0.25		14	
Sugar cane	Pakistan	500 EC	HV 1-2	0.75		15	
Tea	Bangladesh	500 EC	2	0.5		7-10	
	Mozambique	500 EC	2		37.5-50	7	
Tomato	Belgium	500 EC	2		50-37.5	14	
	Dominic Rep.	500 EC	2	0.6	125	-	
	Israel	500 EC	1	0.75-1		3	
	Italy	250 EC	>1		39.3-52.4	21	
	Mozambique	500 EC	2	0.5-0.75		14	

Crop	Country	Form.	No. of appl.	Application rate per treatment		PHI, days	Comments
				kg ai/ha	g ai/hl		
	Poland	500 EC	2		25-50	4	
	Spain	500 EC	1-2	0.5-1.5	50-100	7	
Vegetables	Chile	500 EC	1	0.75-1.0		14	
	Jordan	500 EC			100-125	14	
	Kenya	500 EC	2-3	0.75		14	
	Turkey	500 EC			50	14	
	Uruguay	500 EC	1-2	0.5		14	
Walnut	Chile	500 EC	1		50-60	21	

RESIDUES RESULTING FROM SUPERVISED TRIALS

Residue data from a number of supervised trials were evaluated in 1973. Since then more than 150 supervised trials have been carried out in 18 countries. Unfortunately some of the residue data could not be evaluated because many of the registered uses as provided by the manufacturer were in terms of spray concentration (g ai/hl) or dose per plant (g ai/tree) while the trials data were usually specified in terms of application rate (kg ai/ha). Nevertheless where information was available, conversions were made to allow comparison with the recommendations listed in Table 1. Summaries of the residue trial reports are presented in Tables 2-20 as listed below. Underlined residues in the Tables are from treatments approximating GAP.

Table 2. Apples and pears - Brazil, Canada, Chile, France, Germany, Hungary, Japan and The Netherlands
Table 3. Artichokes and celery - Italy and Spain
Table 4. Beans and peas - Italy, Spain and Switzerland
Table 5. Citrus fruits - Australia, Brazil, China, Greece, Israel, Japan, Morocco and Spain
Table 6. Cotton - Brazil and South Africa
Table 7. Cucurbits - Italy, Japan and Poland
Table 8. Fruiting vegetables - Brazil, Israel, Italy, Japan and South Africa
Table 9. Grapes - Australia, France, Hungary, Israel, South Africa and Switzerland
Table 10. Guava - South Africa
Table 11. Hops - Germany and Japan
Table 12. Maize - Italy and Spain
Table 13. Onions - Italy
Table 14. Papaya - Brazil and the Philippines
Table 15. Pineapple - Brazil
Table 16. Peanuts - The Philippines
Table 17. Peaches and Plums - Brazil, Germany and Switzerland
Table 18. Strawberries - Brazil, Israel, Italy, Japan and

Spain
Table 19. Sugar beet - Italy
Table 20. Tea - India

In all the trials the residues were determined as the parent compound bromopropylate. Bromopropylate was usually applied to the foliage by either hand-held or motorized sprayers.

Apples. Trials data have been submitted from Brazil, Canada, Chile, France, Germany, Hungary, Japan and The Netherlands. No GAP information was available from Brazil, Canada or Hungary to evaluate the trials there. Application rates in Chile and Japan were expressed differently in the trials and the registered uses. The data from France, Germany and The Netherlands were evaluated on the basis of GAP in The Netherlands. Residues at 21 and 28 days after the last application ranged from 0.4 to 1 mg/kg and 0.2 to 1.4 mg/kg respectively.

Pears. Trials were available only from Germany. Residue levels were 0.4-1.6 and 0.3-1.4 mg/kg after 21 days and 28 days respectively.

Table 2. Bromopropylate residues in pome fruits (apples and pears) from supervised trials in Brazil, Canada, Chile, France, Germany, Hungary, Japan and The Netherlands.

Crop/ Country	Application		Int. (weeks)	Residue, mg/kg, at days after last						Ref.
	No.	Rate, g ai/ ha		0	7-10	14	21	28-30	>30	
Apple										
Brazil	1	500					<0.02			1059/82
	1	500					<0.02			1059/82A
	1	600							0.91	1191/85
	1	750					0.18			1060/82
	1	750					0.18			1060/82A
	1	900							0.96	1192/85
	1	0.65/tree				1.1	1.0			2012/85
	1	1.3/tree				2.2	2.0			2013/85
	1	850		0.88	0.76	0.68		0.46	0.36	1163/80
	1	850		0.66	0.25	0.31	0.17			1165/80
	1	1700		0.84	0.44	0.54	0.34	0.24	0.19	1164/80
	1	1700		0.72	0.68	0.47	0.58			1166/80
Chile	3	1864-1966	3	2.7	2.3	2.4	2.1	2.0		1096/90
	3	1108-1170	3				1.2			1097/90
	3	1429-1499	3				1.4-2.5			1098/90
	3	1420-1548	3				1.3-1.6			1099/90
							<0.02 (juice)			

Crop/ Country	Application		Int. (weeks)	Residue, mg/kg, at days after last						Ref.
	No.	Rate, g ai/ ha		0	7-10	14	21	28-30	>30	
France	2	450	4					0.18		95/89
	2	600	4					0.24		95/89
	1	563							0.04	94/89
	1	750							0.08	94/89
	1	675							0.05	91/89
	1	900							0.09	91/89
	2	750	4						0.20	90/89
	2	1000	4						0.13	90/89
In France all applications are in mixtures with various rates of bifenthrin										
Germany	3	563	4	0.7	1.0	1.0	1.0	1.0		1076/86
	3	563	4-8	1.0	1.1	1.3	0.6	0.7		1077/86
	3	563	3-4	0.6	0.4	0.5	0.4	0.5		1088/85
	3	563	4	0.5	0.5	0.5	0.7	0.2		1087/85
	3	578	4-3	1.4	1.0	1.0	0.8	1.4		1089/85
Hungary	2	1500	2	1.3	1.1	0.6	0.5			9/10/84
Japan	2	450	1		2.8	3.1	2.2			PH la/lb/81
	2	450	1		1.7	1.5	1.4			PH la/lb/81
N'lands	1	910					0.8	1.2		1039/83
N'lands	1	1300					0.6	1.0		1037/83
	1	1170					0.5	1.3		1038/83
Pear										
Germany	3	525-750	4	1.6	1.6*	0.96	1.0	0.27		1091/85
	3	563	3-7	0.67	0.62*	0.59	0.45	0.37		1090/85
	3	563	4-5	2.2	2.2*	1.9	1.6	1.4		1079/86
	3	563	4	1.6	0.8*	0.9				1078/86
	3	500-600	4	1.6	2.2*	2.3	0.6	0.8		1080/86

*All 7 days

Artichokes. Data from two trials in Spain were submitted. Spain is the only country where bromopropylate is recommended on artichokes. Under Spanish GAP the application rate is 0.5-1.5 kg ai/ha and the recommended pre-harvest interval is 7 days. The trial rates were below the maximum Spanish GAP rate but residues at 7 days ranged from 2.8 to 5.7 mg/kg.

Celery was not considered in 1973. Only two residue trials from Italy were reported. Bromopropylate is not known to be used on celery in Italy or elsewhere. From the trials data there was no reduction in the residue level from days 0 to 10. It was not possible to deduce when residues started decreasing to the 0.07-0.2 mg/kg reached by day 28. The use rates in the trials were much higher than national recommendations for the vegetables group.

Table 3: Bromopropylate residues in stalk and stem vegetables (artichokes and celery) from supervised trials in Italy and Spain.

Crop/ Country	Application	Plant part	Residue, mg/kg, at days after last applicn.	Ref.

	No.	g ai/ha		0	7	10	14	21	28	
Artichoke										
Spain	1	625 (50 g ai/hl)	whole	9.2	2.8		0.2	0.1		Barba, 1991
			heart	0.88	0.31		0.04	not detected		
	1	937 (75 g ai/hl)	whole	15.3	5.7		0.5	0.1		
			heart	1.4	0.66		0.08	<0.001		
Celery										
Italy	1	562		1.2		4.0			0.07	1091/86
	1	250-370		3.7		0.8			0.2	1090/86

Beans were not considered in 1973. Residue trial reports were submitted from Italy, Spain and Switzerland. Since no GAP inforrmation on beans and peas was available from Switzerland, the trial results from Switzerland could not be evaluated.

Peas. Only two trials in Italy were reported, of which one was within GAP in Spain and was evaluated. Residues in the whole pea decreased from an initial level of 8.3 mg/kg to 1.4 mg/kg on day 21.

Table 4: Bromopropylate residues in beans and peas from supervised trials in Italy, Spain and Switzerland.

Crop, Country	Application		Intvl. (wks.)	Residue, mg/kg, at days after last applicn.						Ref.
	No.	g ai/ha		0	7	14	21	28	30	
Beans										
Italy	1	375	whole	2.5	1.5	0.69	0.66	0.28		1105/87
	1	250-375		7.6	2.46	0.66	0.04	<0.02		1094/86
	1	937		1.4	0.36	0.41	0.02	0.02		1095/86
Spain	1	625	whole	0.54	0.26					Barba, 1991
	1	937		0.78	0.45					
		500		0.54	0.28	0.26				
		750		0.78	0.54	0.45				
		1500			1.69	1.28				Almeria
	1	1500		4.77	2.24	0.99				Almeria
Switzer-land	3	0.3-0.5 g/l						0.08-0.13		
Peas										
Italy	1	625	whole	8.3	6.0	3.2	1.4			1089/86
		1750-3125	peas	4.7	0.9	1.7	0.13		<0.04	1088/86
		(63.5 g ai/hl)	hulls	2.1	1.0	2.9	0.42		0.08	

Citrus. The residue data from Australia, South Africa and Morocco could not be evaluated as there was no informaiton on GAP in those countries. The national use rates in Spain, China and Israel were generally expressed differently from those in the trials so that the data could not be evaluated, but some Spanish trials on lemons could be directly related to GAP. Trials data from Greece could not be evaluated because samples were only taken 85 days after the last application.

Table 5. Bromopropylate residues in citrus fruits from supervised trials in Australia, Brazil, China, Greece, Israel, Japan, Morocco and Spain.

Crop/	Applicn.		Int.	Plant	Residue, mg/kg, at days after last applicn.						Ref.
Country	No	g ai/ha	Wks.	part	0-5	6-7	14	21	28	>32	
Oranges											
Aus-tralia	2	187-375	8	pulp	<0.04						73/7/413
				peel	1.45					0.8	
Brazil	1	7.5 g/tree		pulp	0.02	<0.02	<0.02	<0.02	<0.02		1025/87
				peel	2.0	1.9	1.7	1.5	1.9		
		4.2 g/tree		pulp			0.4			<0.02	1175/83
		(30 g ai/hl)		peel			3.0			1.6	
		4.2 g/tree		pulp			0.12		0.1	0.07	1152/83
				peel	1.3		1.5		0.9	0.7	
		8.4 g/tree		pulp			0.2			0.12	1176/83
Brazil		(65 g ai/hl)		peel			5.8			2.6	
		3.8 g/tree		pulp	<0.02				<0.02		1024/87
				peel	0.61	0.78	0.56	0.59	0.74		
	2	2.25-2.81 g/tree	12	whole	0.9	0.9	1.0	0.9	1.0		1182/89
	2	3.75-3.37	8	pulp				<0.02			1185/89
				peel				2.55			
China	3	66-200	1-4	pulp	0.03		0.007	0.01	0.01	0.007	20/12/84
		g ai/Mu		peel	5.9		1.8		1.7	0.71	
		(1000-3000)		whole	4.2		1.3		1.3	0.51	
Greece	1	480+240*		whole						1.9 (85 days)	1209/86
Israel	1	1200		pulp	0.07	0.05	0.05	0.04	0.04	0.04 (42 days)	63/73
				peel	0.2	1.9	1.4	1.2	1.6	1.6	
Morocco	2	1500	2	pulp	<0.02	<0.02	<0.02	<0.02		0.021-0.026	1003/91
				peel	2.12	2.36	4.95	2.64		4.30-4.65	
Spain	3	2.5 g/tree	3	pulp						0.03 (31 days)	1193/89
				peel						3.9 (31 days)	
	3	3 g/tree	3	whole			1.8				
	3	2.75 g/tree	8	whole		2.3					1191/89
Spain	3	7.5 g/tree	3	whole	3.8	3.19	4.1	2.98	2.4		1190/89
South Africa	1	1500		pulp			<0.06		<0.06	<0.06	14/73
				peel			<0.06	<0.06	0.12		
Mandarin orange											

Crop/ Country	Applicn. No	Applicn. g ai/ha	Int. Wks.	Plant part	Residue, mg/kg, at days after last applicn. 0-5	6-7	14	21	28	>32	Ref.
Greece	1	480+240*		whole						1.3 (85 days)	
Japan	2	450		juice				<0.006-0.04			AC-2
Spain	3	3.5 g/tree	3	whole		3 (8 days)					1201/89
	3	4.5 g/tree	3	whole		3.5 (8 days)					1200/89
	3	4 g/tree	3	whole			1.06				
	3	7.5 g/tree	3	whole	4.6	4.9	4.7				
Lemon											
Greece	2	480+240*		whole						2.0 (85 days)	1208/86
Spain	1	1600			0.96		0.76	0.64		0.66	Camara *et al.*, 1991
	1	2003			0.22		0.46	0.37			
	1	5 g/tree		whole	2.0	2.1	2.2	1.5	1.4		1194/89
	1	5200		whole	1.2	0.88	0.76			0.44	Alicante 1986
Spain (contd.)	1	5200		whole	2.34	1.56	1.27			0.63	Alicante 1986
	1	5200		whole	1.4	1.08	0.88			0.48	Alicante 1986

*applied in a mixture with tetradifon

Cotton seed. A 1973 trial in South Africa had resulted in <0.02 mg/kg residues at the two sampled intervals. In a trial in Brazil within the recommended use rate a residue of 0.04 mg/kg was found after 28 days.

Table 6. Bromopropylate residues in cotton seed from supervised trials.

Country	Application No.	Application g ai/ha	Intvl. (wks.)	Residue, mg/kg, at days after last applicn. 3-7	14-16	28	Ref.
Brazil	3	500				0.04	1090/80
South Africa	2	0.1% ai	1	<0.02	<0.02		5216

Table 7. Bromopropylate residues in cucurbits (cucumber, melon, squash) from supervised trials in Italy, Japan and Poland.

Crop Country	Application	Intvl. (wks.)	Residue, mg/kg, at days after last applicn.	Ref.

	No.	g ai/ha		0	7	14	21	28	
Cucumber									
Italy	1	375-500		0.26	0.08	0.05	0.02	<0.02	1108/86
				0.68	0.02	0.02	0.02	<0.02	1109/86
Poland	1	2000		0.82	0.26	0.10	0.04		10/9/84
Melon									
Italy	1	500		0.29	0.05	0.07	0.06	0.03	1103/87
	1	500		0.45	0.16	0.15	0.21	0.24	1104/87
	1	563		0.08	0.08	0.05	0.06	0.02	1098/86
	5	1250-1875	3	0.04	0.03	0.02	0.27		
	5	375-500		0.26	0.07	0.02	<0.02	<0.02	1097/86
	1	375-500		0.26	0.08	0.05	0.02	<0.02	1108/86
	1	459		0.68	0.02	0.02	0.2	<0.02	1109/86
Japan	4	600	1		0.004	0.004	0.009		AC-3
	2	600	1		<0.002	<0.002	<0.002		
Japan	4	480-600	1-2		0.003	0.003	0.004		
	2	480-600	1.5		0.071	<0.002	<0.002		
Poland	7	1000			0.82	0.26	0.10	0.04	
Squash									
Italy	1	375-500		0.26	0.07	0.02	<0.02	<0.02	1097/86
	1	1250-1875		1.0	0.04	0.03	0.02	0.27	1096/86

Cucumbers, melons and squash. Residue data were submitted from Italy, Poland and Japan. The results from Japan and Poland could not be evaluated because trial application rates were differently expressed from those in the approved uses. Data from Italy were evaluated in relation to Italian GAP. Residues disapeared quite rapidly: on the day of application the highest residue obtained was 1.0 mg/kg in squash and lower in melons and cucumbers.

Egg plant. Additional data from Israel, Italy and Japan were submitted. Applications in Japan were at higher than GAP rates. The data were not evaluated, but residues were <0.001 mg/kg at intervals of 7, 14 and 21 days after application.

Sweet peppers. Only two trials, from Italy and Spain, were reported. No information on GAP was available from these countries.

Tomatoes. Residue trials data were submitted from Brazil, Israel, Italy and South Africa. Results from Brazil and South Africa were not evaluated because of lack of information on registered uses. The data from Israel and Italy (excluding the trial in which a higher application rate was used) showed residues of 0.04-0.1 mg/kg at 7 days.

Table 8. Bromopropylate residues in fruiting vegetables (egg plant, sweet peppers, tomatoes) from supervised trials in Japan, Italy, Israel, Brazil and South Africa.

Crop Country	Application	Intvl. (wks.)	Residue, mg/kg, at days after last applicn.	Ref.

	No.	g ai/ha		0	7	14	21	28	
Egg plant									
Japan	4	330-510	1		<0.001	<0.001	<0.001		AC-4
	2	510	1		<0.001	<0.001	<0.001		
	4	540	1		<0.001	<0.001	<0.001		
	2	540 (30 g ai/hl)	1		<0.001	<0.001	<0.001		
Italy	1	625		1.6	0.86	0.29	0.16	0.08	1100/86
	1	937 (62.5 g ai/hl)		0.42	0.17	0.04	0.05	0.04	1101/86
Israel	1	1000			<0.1	<0.1	<0.1		10/72
Sweet Peppers									
Italy	1	438		4.7	3.4	0.03	1.6	1.5	1102/86
	1	438 (62.5 g ai/hl)		5.1	1.3	1.2	1.3	0.46	1103/86
Spain	1	1500		1.47	0.58	0.36			
Tomato									
Brazil	3	500 (80 g ai/hl)	0.5			1.2			1084/80
	3	750 (187.5 g ai/hl)	1			1.4			1085/80
Israel		1000			0.1	0.1	0.1		RVA 9/72
Italy	1	375		0.29	0.04	0.19	0.09	0.02	1092/86
	1	1250 (62.5 g ai/hl)		1.7	1.3	1.1	0.83	0.92	1093/86
South	2	69	1	0.13	0.16	0.14			1225/86
Africa	2	113	1	0.08	0.35	0.17		0.13	1226/86
	2	90	1	0.33	0.11	0.38		0.10	1229/86
	2	150	1	0.03	0.62	0.15		0.31	1227/86
	2	60 (20-50 g ai/hl)	1	0.02	0.03	0.02		0.06	1228/86

Grapes. Additional data from trials in Australia, France, Hungary, Israel, South Africa and Switzerland was submitted. The data from Australia and South Africa could not be evaluated because no GAP information was available. Trial rates in Hungary were expressed differently from the registered application rates. The data from France, Israel and Switzerland were evaluated.

Table 9: Bromopropylate residues in grapes from supervised trials in Australia, France, Hungary, Israel,South Africa and Switzerland.

Country	Application		Int,	Commod-	Residue, mg/kg, at days after last applicn.						Ref.
	No	g ai/ha	wks.	ity	0	7	14	21	27-28	(No. of days)	

Country	Application		Int,	Commod-	Residue, mg/kg, at days after last applicn.						Ref.
	No	g ai/ha	wks.	ity	0	7	14	21	27-28	(No. of days)	
Aus-tralia	1	1.6 g/vine (37.5 g ai/hl)			1.5	1.1	0.26	0.26			79/10/754
France	1	1000		grapes						0.92 (40)	28/90
				wine						<0.02	
	1	1500		grapes						0.24 (34)	27/90
				wine						<0.02	
	2	550	4	grapes					1.02		26/90
				wine					<0.02		
	2	650	4-6	grapes					0.42		25/90
				wine					<0.02		
	2	410+33*	4	grapes					1.43		111/89
France				wine					<0.02		
	2	487+39*	6-8	grapes					0.93		110/89
				wine					<0.02		
	1	500		grapes						0.34 (70)	13/89
				wine						<0.02	
	2	500	1	grapes						2.16 (77)	14/89
				wine						<0.02	
	1	750+60*	1	grapes						1.75 (40)	04/90
				wine						<0.02	
	1	1125+90*		grapes						0.5 (34)	03/90
				wine						<0.02	
	1	500		grapes						0.11 (73)	15/89
				wine						<0.02	
* applied in a mixture with bifenthrin											
Hungary	2	1500	3		1.8	0.6	0.3	0.09	<0.005		10/10/84
Israel	2	2250	1		4.0	3.4	1.5				112/72
South Africa	3	250-375	2	grapes				1.1 (23 days)			1001/90
				wine				<0.02			
South Africa	3	500-750	2	grapes				3.9 (23 days)			1002/90
				wine				<0.02			
	3	250-375	2-5							0.46 (35)	1244/82
	2	250-375	2-5							0.16 (87)	1243/82
	3	250	16		0.58	0.04	0.18	0.13	0.12		1001/88
	3	250	16		0.27	0.28	0.32	0.21	0.17		1002/88
	3	500	16		0.73	0.47	0.92	0.56	0.48		1003/88
	3	500	16		0.65	0.37	0.55	0.66	0.31		1004/88

Country	Application		Int,	Commod-	Residue, mg/kg, at days after last applicn.						Ref.
	No	g ai/ha	wks.	ity	0	7	14	21	27-28	(No. of days)	
Switz-erland	1	40 g ai/hl		grapes						1.6 (50)	12/72
				Wine						<0.1	

Guava. Since no information on national recommendations was available the report from South Africa could not be evaluated.

Table 10. Bromopropylate residues in guava from supervised trials in South Africa.

Application		Intvl. (wks.)	Plant Part	Residue, mg/kg, at days after last applicn.					Ref.
No.	g ai/hl			0	7	14	21	28	
3	37.5	3	whole	0.11	0.35	0.26	0.17	0.25	1172/89
			pulp					<0.02	
			peel					0.47	
3	37.5	3	whole	0.28	0.25	0.22	0.24	0.21	1173/89
			pulp					<0.02	
			peel					0.44	

Hops. Residue data were submitted from Japan and Germany. The data from Japan could not be evaluated because the application rates were not clearly reported. The trials from Germany showed residues at 28 days after the last application ranged of 2.2-4.9 mg/kg.

Table 11. Bromopropylate residues in hops from supervised trials in Germany and Japan

Country	Application		Intvl. (wks.)	Residue, mg/kg, at days after last applicn.					Ref.
	No.	g ai/ha		0	14	21	28	>32, in beer	
Germany	2	1150-1400 (37.8 g ai/hl)	2	12.0	3.2		4.9	<0.005	1097/88
	2	1125-1350 (37.5 g ai/hl)	2	6.7	4.5		2.2	<0.005	1095/88
	2	1125-1350	2	16.0	3.8		3.4	<0.005	1096/88
Japan	2	30 g ai/hl	4				0.09		25.7.1972
	4	30 g ai/hl	2				0.41 (37 days)		
	2	30 g ai/hl	2		0.07				
	4	30 g ai/hl	1			0.23			

Maize. Data from Spain and Italy were not evaluated as there was no information on GAP.

Table 12. Bromopropylate residues in maize from supervised trials in Italy and Spain.

Country	Application		Plant	Residue, mg/kg, at days after last applicn.						Ref.
	No.	g ai/ha	part	0	7	14	21	28	75-106	
Italy	1	313	whole	6.3	4.6	3.9	4.8	3.9		1095/87
	1	625	whole	9.6	6.2	6.5	3.5	2.3		1096/87
	1	375	whole	11.0	5.2	9.9	3.6	6.5		1104/86
Spain	1	1000	grain						<0.02	1018/91
	1	1000	grain						<0.02	1019/91
	1	1000	grain						<0.02	1020/91
Spain	1	1000	grain						<0.02	1021/91
	1	1000	grain						<0.02	1022/9
	1	1000	grain						<0.02	1023/91

Onions. Residue data from Italy were submitted but not evaluated because there was no GAP information.

Table 13. Bromopropylate residues in onions from supervised trials in Italy.

Application		Plant part	Residue, mg/kg, at days after last applicn.					Ref.
No.	g ai/ha		0	7	14	21-25	28	
1	375	whole	3.3	0.93	0.37	1.0	0.39	1253/86
1	531	whole	15.8	16.9	5.5	3.7		1254/86

Papaya, pineapples. No information on national GAP was submitted. GAP for the fruits group in Austria, Iran and Pakistan could not be used to evaluate the data from Brazil and the Philippines.

Table 14. Bromopropylate residues in papaya from supervised trials in Brazil and the Philippines.

Country	Application		Int. (wks)	Residue, mg/kg, at days after last applicn.							Ref.
	No.	g ai/ha		0	3	7	14	21	28	>32	
Brazil	2	500	2				1.0				1082/80
	2	750	2				1.9				1083/80
	1	500					0.03		0.2		1108/84
	1	1000					0.43		0.31		1109/84
Philippines	6	280	1	0.18	0.36	0.51	0.53				1005/91
	6	140	1	0.12	0.13	0.29	0.44				1006/91

Table 15. Bromopropylate residues in pineapples from supervised trials in the Philippines.

Application		Int. (wks.)	Plant part	Residue, mg/kg, at days after last applicn.				Ref.
No.	g ai/ha			0	14	28	31	

2	0.15 g/l	1	pulp	<0.02	<0.02	<0.02	<0.02	1069/90
			peel	0.72	0.17	0.13	0.09	
2	0.3 g/l	1	pulp	<0.02	<0.02	<0.02	<0.02	1068/90
			peel	1.5	0.75	0.34	0.15	

Peanuts. The residue data from Brazil were not evaluated because there was no information on GAP.

Table 16. Bromopropylate residues in peanuts from supervised trials in Brazil.

Application		Int. (wks)	Plant part	Residue, mg/kg, at 37 days after last applicn.	Ref.
No.	g ai/ha				
2	500	7	seeds	0.02	1086/80
			pods	0.09	
2	750	7	seeds	<0.02	1087/80
			pods	0.12	

Plums and peaches. Data were submitted from Brazil, Switzerland and Germany. The data from Switzerland and Germany were evaluated on the basis of the GAP of Switzerland and The Netherlands.

Table 17. Bromopropylate residues in stone fruits (peaches and plums) from supervised trials in Brazil, Germany and Switzerland.

Crop Country	Application	Int. (wks.)	Residue, mg/kg, at days after last applicn.	Ref.

	No.	g ai/ha		0	7	14	21	28	78	
Peaches										
Brazil	1	500				0.75(18 days)				1131/81
	1	750				1.72(18 days)				
Swit-zerland	1	375							0.09	104/73
	1	500							0.18	
Plums										
Germany	3	563	4	3.1	0.88	0.92	0.63	1.5		1082/86
	3	563	4	1.1	1.2	0.78	0.85	0.4		1083/86
	3	563	4	1.7	0.9	0.9	0.9	0.7		1085/86
	3	563	4	2.0	2.2	1.6	1.2	0.69		1081/86
	3	563	4	3.9	2.0	1.9	1.4	1.3		1067/87
	3	563	4	1.7	2.0	1.9	1.5	1.3		1066/87
	3	563	4-3	0.82	0.62	0.52	0.27	0.3		1065/87
	3	563	5-4	3.5	0.95	1.9	1.6	1.6		1064/87
	3	563	4-3	1.5	1.7	1.1	0.57	0.66		1063/87
(Prunes)	3	563	4	2.3	1.4	2.0	1.2	1.1		1084/86

Strawberries. Residue trial data from Brazil, Israel, Italy, Japan and Spain were submitted. Residue levels in the trials in Spain and Italy at 14 and 21 days were respectively 0.03-1.5 mg/kg and 0.5-1.6 mg/kg.

Table 18. Bromopropylate residues in strawberries from supervised trials in Brazil, Israel, Italy, Japan and Spain.

Country	Application	Int. (wks.)	Residue, mg/kg, at days after last applicn.	Ref.

	No.	g ai/ha		0-3	7-9	14	21	28	
Brazil	4	500	1		0.72(9 days)				1195/80
	4	750	4 days		1.1(9 days)				1196/80
Israel	2	0.75 g/l	1	8.2 (3 days)					110/79
	3	0.75 g/l	10 days	15.4 (3 days)					111/79
		1	0.75 g/l	5.4 (3 days)					109/79
Italy	1	500		1.8	0.8	0.6	0.48	0.49	1101/87
	1	625		7.1	2.9	1.5	1.6	0.3	1102/87
Japan	2	0.3 g/l			1.71	1.42			AC-5
	2	0.3 g/l			1.16	0.28			
Spain		1000		3.6-4.2	1.82	1.18			Huelve
		1000		0.7-2.4	0.81	0.45			Huelve
	1	1000		0.9-1.4	0.6	0.3			Huelve
	4	625		4.2-11.9	2.3	0.53			Malaga
	4	937		7.5-13.8	2.6	0.03			Malaga

Sugar beet. As no national GAP was available, the data from Italy could not be evaluated.

Table 19. Bromopropylate residues in sugar beet from supervised trials in Italy.

Application		Plant part	Residue, mg/kg, at days after last applicn.					Ref.
No.	g ai/ha		0	7	14	21	28	
1	313	roots	0.07	0.2	0.06	0.1	0.08	1097/87
		leaves	5.4	2.0	3.0	1.1	1.3	
1	750	roots	0.38	0.5	1.0	0.26	<0.02	1098/87
1	375	roots	0.09	0.09	0.09	0.14	0.1	1106/86
		leaves	12.0	2.0	0.31	1.1	0.28	

Tea. The additional data from India were not sufficient to recommend an MRL.

Table 20. Bromopropylate residues in tea, seven days after last application, from a supervised trial in India.

Application		Commodity	Residue, mg/kg	Ref.
No.	g ai/ha			
1	62.5	dried leaves	6.5	AG-A 1888
		manufactured tea	2.4	

FATE OF RESIDUES

The fate of bromopropylate in plants, animals and soil was reviewed in 1973. Studies on cows and calves were conducted using radiolabelled bromopropylate (Cassidy *et al*., 1968). Cows were fed 9.7 ppm bromopropylate in the feed for 5 days. In tissues bromopropylate was found at significant levels only in the fat, from which it was eliminated rapidly when dosing was stopped.

In plant studies on soya beans and apples, bromopropylate was found to remain on

the treated surface, with hardly any penetration or translocation. The main residue was the parent compound but the metabolite 4,4′-dibromobenzilic acid was also detected. Additional information on the fate in citrus and tomatoes has since been submitted.

In plants

Citrus. A 3-year old Valencia orange tree in a glasshouse was treated with 266 mg ^{14}C-ring-labelled bromopropylate at a rate equivalent to about 1 kg ai/ha (Spare, 1989). After 62 days, samples of the ripe fruit, leaves and soil were collected for analysis. The ripe fruits were processed to obtain the juice before the peel and the pulp sacs were separated. All the samples were extracted in organic solvents and radioactivity was determined in both the organo- and water-soluble extracts. Leaves were found to contain 19 mg/kg of residues, the fruit peel 1.8 mg/kg, the pulp <0.026 mg/kg, the juice <0.008 mg/kg and the soil 0.08 mg/kg.

The organosoluble fraction contained 66-79% of the radioactivity, the aqueous extract about 1.2-1.8% and the non-extractable fraction 13-29%. Thin-layer chromatography was used to characterize the organosoluble fraction and showed that about 60-68% of the total radioactivity in the citrus leaves and peel was due to the parent compound. This study supported the submission in 1973 that bromopropylate was not metabolized to any significant extent in the edible parts of plants. Bromopropylate might therefore be considered as the toxicologically important residue.

Tomato. Tomato plants in the field were treated three times with ^{14}C- bromopropylate at a rate corresponding to 500 g ai/ha (Galicia, 1991). Samples of the fruits and leaves were taken at 0, 22 and 58 days after the last (third) application. Total ^{14}C levels in the fruit declined over this period more rapidly than in the leaves. The data also suggested that there was little or no translocation from leaves to fruit. The distribution of the ^{14}C residues in the fruits and leaves is shown in Table 21.

Table 21. Distribution of ^{14}C in tomato fruit and leaves from plants treated with [^{14}C]bromopropylate at 0.5 kg ai/ha.

Nature of residue	Fruit, days after final application			Leaves, days after final application		
	0	22	58	0	22	58
Total ^{14}C, mg/kg*	5.0	1.2	0.055	115	47	34
Extractable ^{14}C, mg/kg*	5.0		0.061	112		35
Non-extractable ^{14}C, mg/kg*			<0.006			0.5
% of ^{14}C on surface	93		18	51		34

*bromopropylate equivalants

Surface radioactivity determined from the washings of the tomatoes and leaves also declined; for tomatoes very little of the residues became bound. Surface radioactivity was 93% of the total ^{14}C at day 0, declining to 18% at day 58. Surface radioactivity on the leaves at these times was 51% and 34% respectively. Extractable radioactivity in or on the leaves and fruits was very high, ranging respectively from a maximum of 112 and 5.0 mg/kg on day 0 to 35 and 0.061 mg/kg at harvest. Non-extractable radioactivity was low for both the leaves (0.5 mg/kg) and fruit (<0.006 mg/kg) at harvest.

Characterization of the radioactive residues in the fruit and leaves was by thin-layer chromatography. Tomato fruits at harvest contained 0.050 mg/kg of the parent compound (89% of the radioactivity), 0.002 mg/kg of 4,4′-dibromobenzilic acid and the remainder as 7 other minor metabolites. Leaf samples at harvest were found to contain 11 radioactive fractions, the most abundant being the parent compound (65%). The metabolite 4,4′-dibromobenzilic acid accounted for 8%, the rest being minor metabolites. The washings from the tomatoes and leaves at harvest were also characterized and found to contain mainly the parent compound in both instances, (82% for tomatoes and 90% for the leaves).

In animals

No further work was reported on the fate of bromopropylate in animals but the manufacturer indicated that some information on the fate in animals and toxicological properties of bromopropylate had been submitted to WHO in 1992 for evaluation.

In soil

The degradation of bromopropylate under aerobic and anaerobic laboratory conditions was studied to characterize the degradation products (Ercegovic *et al*., 1976) ^{14}C-ring-labelled bromopropylate was applied to silty loam and sandy loam soils which were incubated under controlled conditions for 52 weeks. Samples were periodically taken for determination of radioactivity. Degradation was predominantly due to biological activity and was more rapid in silty than sandy loam under both aerobic and anaerobic conditions as indicated by the decreasing percentage of organo-extractable radioactivity and increasing radioactivity that remained in the silty loam after extraction. This was also shown in the higher percentage of [^{14}C]bromopropylate that was converted to $^{14}CO_2$ in the silty loam, although the degradation was not very pronounced. Tables 22 and 23 summarize some of the results for silty and sandy loam respectively. The half-life of bromopropylate in silty loam soils under the test conditions was determined to be 45 days. The major extractable degradation product in both soils under aerobic conditions was 4,4′-dibromobenzophenone, while under anaerobic conditions the major degradation product was 4,4′-dibromobenzhydrol in silty loam and 4,4′-dibromobenzophenone in sandy loam. In another study using similar soils from different sources under aerobic conditions (Suter, 1982a), the half-life of ^{14}C-bromopropylate was determined to be about 47 days for sandy loam and 70 days for silty loam. Degradation was thought to follow first order kinetics. The major metabolite was 4,4′-dibromobenzophenone. Carbon dioxide, probably produced by further degradation, accounted for 56-60% of the radioactivity after 270 days. Another study on the persistence of bromopropylate in soils was conducted in Florida (Rothwell *et al*., 1971). Fine sand samples (10-15 cm) were taken from an area where citrus had been planted for many years. The soil was air-dried, screened and thoroughly mixed before bromopropylate was added at rates of 0.0, 0.25, 0.5 and 1.00 mg/kg active ingredient. All samples were incubated at 28°C for 16 weeks. Residues of bromopropylate decreased with time but the higher concentrations seemed to decrease at a faster rate. The estimated half-life for bromopropylate in both soils was about 60 days.

Table 22. Percentage of radioactivity at day 0 recovered from silty loam soil treated with [^{14}C]bromopropylate after various intervals under sterile, aerobic and anaerobic conditions.

Sample	Percentage recovery of radioactivity at weeks after application						
	0	4	8	12	24	39	52
Sterile incubation							
Soil before extraction	100.0	102.0	99.9	101.2	99.4	99.4	99.9
Acetonitrile- water extraction	97.9	98.1	99.1	97.8	93.2	93.7	90.7
Soil residue after extraction	3.6	3.5	3.3	3.1	4.1	7.8	7.8
Volatile trappings	-	<0.1	<0.1	<0.1	0.7	<0.2	<0.1
Total recovery	100.5	101.6 102.4	94.3	98.0	98.5	98.6	
Aerobic incubation							
Soil before extraction	100.0	96.0	94.2	93.1	83.8	76.2	73.5
Acetonitrile-water extraction	97.0	83.8	80.6	77.6	65.0	53.9	49.1
Soil residue after extraction	2.9	6.6	10.0	12.6	15.4	18.0	20.0
Volatile trappings	-	3.3	3.7	4.1	3.2	2.0	0.0
Total recovery	99.9	93.5	93.9	94.3	83.6	74.0	70.8
	Aerobic incubation			Anaerobic incubation			
Soil before extraction	100.0	94.1	88.9	88.8	85.6	82.5	79.3
Acetonitrile-water extraction	97.0	75.1	74.4	74.9	64.9	60.7	58.9
Soil residue after extraction	-	10.3	10.0	9.7	21.3	16.0	12.7
Volatile trappings	-	5.7	5.6	5.3	1.0	4.5	8.8
Total recovery	99.9	93.5	94.3	89.6	87.2	74.0	70.0

Table 23. Percentage of radioactivity at day 0 recovered from sandy loam soil treated with [^{14}C]bromopropylate after various intervals under sterile, aerobic and anaerobic conditions.

Sample	Percentage recovery of radioactivity at weeks after application						
	0	4	8	12	24	39	52
Sterile incubation							
Soil before extraction	100.0	99.2	99.9	100.6	100.3	100.8	102.3
Acetonitrile- water extraction	99.2	99.2	96.8	99.5	98.5	99.6	99.0
Soil residue after extraction	0.7	0.3	0.4	0.4	1.0	1.4	1.7
Volatile trappings	-	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Total recovery	99.9	99.5	97.2	99.9	99.6	101.2	101.6
Aerobic incubation							
Soil before extraction	100.0	98.3	95.6	93.7	96.3	96.1	96.0
Acetonitrile-water extraction	99.3	95.1	95.6	93.1	92.6	91.9	91.6

Sample	Percentage recovery of radioactivity at weeks after application						
	0	4	8	12	24	39	52
Soil residue after extraction	0.1	0.6	1.5	1.2	2.9	3.7	4.6
Volatile trappings	<0.1	0.5	0.9	1.1	0.3	0.7	0.4
Total recovery	100.2	96.3	98.0	95.4	95.9	96.2	96.7
	Aerobic incubation			Anaerobic incubation			
Soil before extraction	100.0	99.3	98.4	95.3	93.8	93.4	92.7
Acetonitrile-water extraction	99.3	97.3	95.5	91.2	91.4	90.6	89.4
Soil residue after extraction	0.9	0.6	1.5	1.0	2.5	2.0	2.3
Volatile trappings	-	0.5	0.9	1.1	0.4	1.1	1.8
Total recovery	100.2	98.5	97.8	93.3	94.4	93.8	93.6

Mobility studies

The leaching characteristics of bromopropylate were studied according to German requirements (Guth, 1974). Soil columns of sandy loam, silty loam and sandy soils were used. After applying the equivalent of 200 mm of rain in 2 days, no bromopropylate (<0.2 ìg/l) was detected in the eluate from any of the columns.

In another study on the leaching properties of bromopropylate in silty loam and sandy soil with [^{14}C]bromopropylate (Suter, 1982b), the compound was applied to the soil column at a rate equivalent to 1 kg ai/ha and residues were monitored. Only 6.5-13.1% of the applied radioactivity remained after 6 months. The "aged" soil was then added to the top of similar but untreated soils as a 2 cm layer and rain was simulated for 45 days (total 571 mm). No significant radioactivity was detected in the leachate of either soil (1.34% and 0.13% of the applied radioactivity). These studies suggested that bromopropylate and its metabolites have low mobility in the two soils.

In water

The hydrolysis of bromopropylate in aqueous systems at pH values of 1 to 10 was studied at a concentration of 1 mg/l (Burkhard, 1977). No significant hydrolysis was detected under neutral conditions (pH 5 and 7), but at pH 9 and 10 the half-life of bromopropylate was determined to be 34 days and 4.4 days respectively.

Photolysis studies were carried out with [^{14}C]bromopropylate in aqueous solution under artificial sunlight (Frank *et al.*, 1992). Only 30% of the initial radioactivity was recovered after 51

hours. The main metabolite was 4,4′-dibromobenzilic acid which accounted for 27% of the radioactivity. Under natural sunlight, no decomposition was detected after 2 weeks.

In another study, [^{14}C]bromopropylate (0.5 mg/kg) was applied to soil as a sediment in well water at 18◦C and allowed to age for 28 days (EG & G Bionomics, 1976). Catfish were kept in the water for 49 days. The concentration in the unfiltered water increased from <0.2 ìg/l to 1 ìg/l at the end of 49 days. Radioactivity in the whole fish was calculated to be 1.05 mg/kg, corresponding to a bioaccumulation factor of 1050, but 86% of the radioactivity had been eliminated by day 7. [^{14}C]bromopropylate in the aquatic system was also studied using river from the Rhine and a pond with about 1% sediment (Cuth, 1984). Initially radioactivity in the waters decreased rapidly owing to adsorption to the sediment. Bromopropylate was subsequently degraded and the half-life was determined to be about 20 and 40 days in river and pond water respectively. The parent compound accounted for only about 5.6% and 3.4% of the initial radioactivity in the waters respectively after 77 days. The main metabolites detected were 4,4′-dibromobenzophenone and 4,4′-dibromobenzilic acid. Extraction of the sediment recovered 21.4% and 53.7% radioactivity in the river and pond water respectively. The parent compound was the major residue. Small amounts of carbon dioxide were also detected. No significant difference was found in the types or numbers of the aquatic micro-organisms present in the untreated and treated systems suggesting that bromopropylate had no harmful effects on aquatic micro-organisms.

In processed commodities

In some of the residue trials the crop was further processed and the products analysed for bromopropylate. As shown in Table 2, apple juice from apples with residues of 1.3-1.6 mg/kg was analysed and no measurable residues (<0.02 mg/kg) were detected. Juice from mandarin oranges obtained from a trial in Japan after a PHI of 21 days was analysed and found to contain <0.006-0.04 mg/kg residues (Table 5). Wine produced from grapes in trials carried out in France and South Africa was also found to contain no measurable residue (<0.02 mg/kg, Table 9). Beer was brewed from dried cones after residue trials on hops. The dried cones contained 2.2-4.9 mg/kg residues but <0.005 mg/kg was detected in the beer (Table 11).

Stability of residues in stored analytical samples

The stability of bromopropylate residues in samples of tea, tomatoes, tomato puree, oranges, grapefruit, orange juice and orange oil, apples, peaches and cherries during storage under freezer conditions was investigated.

In all the studies, the crop or processed crop was first homogenized and then fortified with bromopropylate at 0.5 mg/kg. A sample was taken for analysis before it was stored at -18◦C in glass and plastic containers. Subsequent samples were taken at

3, 6, 12, 18 and 24 months for analysis. In both glass and plastic containers, bromopropylate residues remained stable in the various matrices after storage for 24 months. The percentage changes in all commodities in both glass and plastic containers are shown in Table 24.

Table 24. Storage stability of bromopropylate in crop samples (% change corrected for recovery).

Crop/ processed crop	Storage time (Months)									
	3		6		12		18		24	
	G	P	G	P	G	P	G	P	G	P
black tea	-3	+11	+3	+3	+3	+7	-4	-6	-1	-2
tomato	-6	-4	-5	-7	-15	-16	-6	-10	-6	-15
tomato puree	+5	+12	-6	-10	-7	-4	-7	+7	0	+9
orange	-7	+4	+5	+15	+3	+16	+3	+6	-3	0
grape fruit	-4	+6	+5	+12	+2	+6	+2	+11	+1	+7
orange juice	+6	-2	+7	0	-3	-10	0	-7	-2	-9
orange oil	+8	-	+7	+12	+2	+5	+3	+7	+4	+6
apple	+2	+13	+1	0	+4	0	-6	0	+1	+6
peach	+4	+7	-4	+8	-6	-2	-2	-1	-4	0
cherry	-3	-4	-13	-15	-11	-2	-6	-10	0	+11

G - Glass; P - Plastic

METHODS OF RESIDUE ANALYSIS

Methods for the determination of bromopropylate residues using gas chromatography and thin-layer chromatography were described in the 1973 review. The clean-up procedures have been modified by the use of "BondElut" silica cartridges. Recoveries were 88-113% and the limit of determination 0.02 mg/kg for apples, cherries, citrus fruits, peaches, tomatoes, tomato puree and tomato ketchup. For tea and hops the limit of determination was 0.1 mg/kg. For water samples, "BondElut" C_{18} cartridges were used for extraction, followed by determination on a gas chromatograph fitted with an electron-capture detector. The limit of determination was 0.05 ìg/l.

NATIONAL MAXIMUM RESIDUE LIMITS

Since the last review in 1973, many countries have established MRLs for bromopropylate in various food crops. These are summarized below.

Country	Crop	MRL (mg/kg)
Australia	pome fruits	5

Country	Crop	MRL (mg/kg)
	stone fruits	5
Germany	banana pulp	0.2
	banana, whole	3.0
	citrus juice	0.2
	citrus, whole	5
	cotton	1
	grapes	2
	hops	5
	pome fruit	2
	stone fruit	2
Germany cont.	strawberries	2
	tea	5
	vegetables	1
Hungary	apples	2
	grapes	1
	pears	2
Israel	apples	5
	apricots	5
	banana pulp	0.2
	banana, whole	5
	beans	1
	cherries	5
	citrus fruit pulp	0.2
	citrus fruit, whole	0.2
	cotton seed	1
	egg plant	5
	grapes	5
	peaches	5
	pears	5
	plums	5
	strawberries	5
	tomatoes	1
	vegetables	1
Italy	banana	3
	citrus fruit	3
	fruits, except as specified	0.05
	grapes	2
	pome fruit	2

Country	Crop	MRL (mg/kg)
	stone fruit	2
	strawberries	2
	vegetables	1
Japan	apple	2
	citrus fruit	2
	fruits, except as specified	2
	egg plant	0.5
	hops	1
	watermelon	0.5
	oranges	5
	peaches	2
	pears	2

Country	Crop	MRL (mg/kg)
	vegetables	0.5
Jordan	citrus fruit	0.2
	grapes	5
	vegetables	1
Netherlands	banana	3
	citrus fruit	3
	grapes	2
	honey	05*
	hops, dry	5
	pome fruit	2
	stone fruit	2
	strawberries	
	tea	5
	vegetables	1
* under consideration		
New Zealand	apples	3
	blackcurrants	3
	cane fruit	3
	fruit	3
	peaches	3
	strawberries	3
Poland	cucumbers	1
	currants, black, red, white	1
	peppers	1
	pome fruit	1
	raspberries	1
	stone fruit	1
	strawberries	1
	tomatoes	1
South Africa	banana	3
	citrus fruit	0.2
	cotton seed	0.2
	grapes	1
Spain	artichokes	1 (proposed)
	beans, green	1 (proposed)
	eggplant	1 (proposed)
	tomatoes	1 (proposed)
Switzerland	fruit	1.5

Country	Crop	MRL (mg/kg)
	honey	0.2
Turkey	citrus fruit	0.1
	pome fruit	0.2
	vegetables	0.5

APPRAISAL

Bromopropylate was scheduled by the CCPR for periodic review at the 1993 JMPR (ALINORM 93/24A, para 93). It was first considered by the JMPR in 1973 when residue data on apples, pears, plums, grapes, bananas, strawberries, citrus, hops, tea, cotton, egg plant and tomatoes were evaluated and MRLs for apple, banana, cherry, citrus fruits, cotton seed, grapes, hops, nectarine, peach, pear, plum, strawberry, tea and vegetables were established. Since then, more residue trials on some of the same crops as well as additional ones such as artichokes, beans, celery, cucurbits, guavas, maize, onions, papaya, peaches, peanuts, peas, sweet peppers, pineapples and sugar beet have been conducted by the manufacturer in various countries as well as the authorities of The Netherlands and Spain. Further information has also been provided by the manufacturer, Spain, The Netherlands and Australia on current uses. Australia has also indicated that the pesticide had not been marketed since 1986.

The manufacturer has indicated that there were no current uses on nectarines, bananas or cherries. The Meeting recommended the withdrawal of the MRLs for these commodities.

Additional plant metabolism studies on apples and citrus showed that the parent compound was the residue of importance, particularly in the edible parts, although minor metabolites, mainly 4,4′-dibromobenzilic acid, were found in the leaves.

No information on the fate in animals has been submitted but the Meeting noted that adequate information on animal transfer studies for dairy cows and beef cows had been reported by the 1973 JMPR.

In water, bromopropylate was found to have a half-life of 20-40 days. Bromopropylate and its metabolites were concluded to have low mobility in sandy loam, silty loam and sandy soils on the basis of leaching studies. The half-life in silty loam and sandy loam soils was about 45 days, the major metabolite being 4,4′-dibromobenzophenone.

Residues in the juice of apples and mandarin oranges, and in wine and beer were reported to be below the limit of detection, (0.02 mg/kg in all cases, except beer 0.005 mg/kg).

Bromopropylate residues in samples of tea, tomatoes, tomato puree, oranges, grapefruit, orange juice, orange oil, apples, peaches and cherries were found to remain stable up to 2 years under freezer conditions at -18°C.

GAP information was not available for guavas, papayas, pineapples, onions, celery, maize, peanuts or sugar beet, so residue data on these crops could not be evaluated.

GAP information and residue trials data on peas, tomatoes, egg plants, artichokes and sweet peppers were too limited for the Meeting to estimate maximum residue levels.

The available information for cotton seed, hops and tea was also too limited to support the present MRLs. The Meeting agreed to withdraw the recommendations for these commodities.

For citrus, the trials data on residues in the pulp and peel confirmed earlier findings that most of the residues are concentrated in the peel. Results from Australia, South Africa and Morocco were not supported by GAP information, while in data from Spain, China and Israel trials rates were expressed differently from the national GAP. Six trials in Brazil on oranges and mandarin oranges were evaluated in the light of the national GAP. At 14 days after the last application, residues ranged from 0.6 to 5.8 mg/kg in the peel and 0.2 to 0.4 mg/kg in the pulp. Assuming that the peel weight is 30% of the fruit's weight the calculated residues in whole fruit would be less than 2 mg/kg. In trials on lemons in Spain rates between 1.6 and 5.2 kg ai/ha were used, which were within Spanish GAP. At 14 days, residues ranged from 0.5 to 1.3 mg/kg. The Meeting estimated a maximum residue level of 2 mg/kg for bromopropylate in citrus fruits with a PHI of 14 days, to replace the current MRL of 5 mg/kg.

The residue trials data on apples from Brazil and Canada were not evaluated because no information on registered uses was available from these countries. Trials from Chile were also not evaluated because the registered use was supplied in terms of spray concentration while the trials application rates were expressed as kg ai/ha. Trials in The Netherlands, France and Germany on apples and pears were within the GAP of The Netherlands and France, and residues at 21 days were within the range of 0.18-1.6 mg/kg. Data on apples and pears were mutually supporting. The Meeting recommended an MRL of 2 mg/kg for pome fruits at a pre-harvest interval of 21 days, based on the data from France, Germany and The Netherlands.

For peaches, trials data from Brazil and Switzerland were submitted, and for plums, data from ten trials in Germany. Data from Switzerland and Germany were evaluated on the basis of the GAP of Switzerland (stone fruits) and The Netherlands (plums). At 21 days after the last application, the highest residue obtained was 1.6 mg/kg. The Meeting estimated a maximum residue level of 2 mg/kg for plums at a pre-harvest interval of 21 days. Data from peach trials were insufficient to recommend an MRL but

provided some additional support for the plum estimate.

Additional data on grapes from trials in Australia, France, Hungary, Israel, South Africa and Switzerland were submitted. As there was no information on registered uses in Australia and South Africa, the data from these countries could not be evaluated. Trials rates in Hungary were expressed differently from GAP in Hungary. The trials rates in France, Israel and Switzerland covered the national recommendations. The data from France showed that by 27 days residues in grapes were all less than 2 mg/kg, and in wine <0.02 mg/kg. The Meeting recommended an MRL of 2 mg/kg for grapes at a pre-harvest interval of 28 days.

Residue trials data on strawberries had been submitted from Brazil, Israel, Italy, Japan and Spain. Although trial rates in Spain and Italy did not cover the maximum rates under the GAP of the two countries, on the basis of the trials data the Meeting recommended an MRL of 2 mg/kg for strawberries with a PHI of 14-21 days.

For beans, evaluation of the residue trials data from Italy and Spain was based on the GAP of Spain. Residue levels at 7 days ranged from 0.26 to 2.5 mg/kg. The Meeting recommended an MRL of 3 mg/kg for common beans at a pre-harvest interval of 7 days.

Residue data from Italy on cucumbers, melons and summer squash were evaluated on the basis of the GAP of Spain. The Meeting recommended an MRL of 0.5 mg/kg for cucumber, melons and summer squash at a pre-harvest interval of 7 days.

The Meeting recommended withdrawal of the MRL for vegetables, to be replaced by MRLs for the specific commodities beans, cucumber, melons and summer squash.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits.

Definition of the residue: bromopropylate.

Commodity		Recommended MRL, mg/kg		PHI on which based, days
CCN	Name	New[1]	Previous	
FP 0226	Apple	W	5	
FI 0327	Banana	W	5	
VP 0526	Common bean (pods and/or immature seeds)	3	-	7
FS 0013	Cherries	W	5	
FC 0001	Citrus fruits	2	5	14
SO 0691	Cotton seed	W	1	
VC 0424	Cucumber	0.5	-	7
FB 0269	Grapes	2	5	28
DH 1100	Hops, dry	W	5	
VC 0046	Melons, except Watermelon	0.5	-	7
FS 0245	Nectarine	W	5	
FS 0247	Peach	W	5	
FP 0230	Pear	W	5	
FS 0014	Plums (including prunes)	2	5	21
FP 0009	Pome fruits	2	-	21
VC 0431	Squash, Summer	0.5	-	7
FB 0275	Strawberry	2	5	7-14
DT 1114	Tea, Green, Black	W	5	
A01 0001	Vegetables	W	1	

[1] W: the previous recommendation is withdrawn.

FURTHER WORK OR INFORMATION

Desirable

1. Information on the occurrence of bromopropylate residues in food in commerce or at consumption.

2. Information on residues in the pomace of citrus fruit, apples and grapes, which may be used as animal feeds.

REFERENCES

Australia. Submission on GAP and label text.

Barba, A. *et al*. 1991. Disappearance of bromopropylate residues in artichokes, strawberries and beans. J. Environ. Sci. Health, B 26(3), 323-332.

Burkhard, N. 1997. Rate of hydrolysis of GS 19851 under laboratory conditions. Proj. Rep. 21/77, Ciba-Geigy, Basle, April 1977 - unpublished.

Camara *et al*. 1991. Residuos de bromoproilato en citrus limon, variedad Verna. Bol. San. Veg. Plagas 17, 165-171.

Cassidy, J.E., Mattson, A., Cullen, T. and Min, B. 1968. The metabolic fate of ^{14}C GS 19851 administered to a cow by capsules. Ciba-Geigy, New York - unpublished.

China. 1984. The determination of residues of bromopropylate in citrus. 20 December. Project Report 20/12/84.

Ciba-Geigy. 1978-1990. Reports on Methods of analysis of bromopropylate residues in various substrates by gas chromatography - unpublished.

Ciba-Geigy. 1989. Gas chromatographic determination of unpolar pesticides in plant material. Basle, August 1989, Method Description REM-119-01 - unpublished.

Ciba-Geigy. 1989-1992. Reports on residue storage stability studies under freezer conditions for tomatoes, tomato puree, citrus fruits, orange juice , orange oil, apples, cherries and peaches, 1989-1992. Study director: G. Formica - unpublished

Ciba-Geigy. 1993. Working Paper on Bromopropylate for the Panel of Experts on Pesticide Residues (3 volumes).

Ciba-Geigy. Unpublished reports from Basle on residue trials on apples, beans, celery, citrus, cotton, cucumber, eggplant, grapes, guava, hops, maize, melons, onion, papaya, peach, peanut, pears, peas, pepper (sweet), pineapple, plums, squash, strawberries, sugar beet, tea and tomatoes.

Ciba Geigy Australia. Determination of GS-19851 Residues in oranges TR No. 73/7/413 - unpublished.

Cuth, J.A. 1984. Degradation of bromopropylate in aquatic systems. Project 024625, RCC Itingen - unpublished

EG & G Bionomics. 1976. Kinetics of aged Acarol in model aquatic ecosystem - Aquatic Toxicology Lab., Wareham Mass. - unpublished.

Ercegovic, C.D. and Bogus, E.G. 1976. Fate of Isopropyl-4,4′-dibromobenzilate (bromopropylate) in soil under aerobic and anaerobic laboratory conditions. Rep. No. E-2/1-76, Pest. Res. Lab., Pennsylvannia, USA - unpublished.

Frank, J. and Balu, K. 1973. Film photolysis of GS-19851 under natural and artificial sunlight conditions. GAAC-73023, Ciba-Geigy Corp., USA - unpublished.

Frank, J. *et al*. 1992. Photolysis of GS- 19851 in aqueous solution under natural and artificial conditions. GAAC - 72140, Ciba-Geigy, Corp., USA.

Galicia, H. 1991. ^{14}C-GS 19851 (bromopropylate): Plant metabolism study in field grown tomato after foliar treatment with Bis(Phenyl -U^{14}C)Gs 19851 labelled material. RCC Project 247634, RCC, Itingen, Switzerland - unpublished.

Guth, J.A. 1974. Leaching behaviour of the acaricide bromopropylate in three German standard soils. SPR 25/74, Ciba-Geigy Basle - unpublished.

Netherlands. Submission on GAP, MRLs and residue data on apples, method of analysis on apples and honey and residues in commercial honey.

Rothwell, D.F. *et al*. 1971. Persistence and microbiological effects of Acarol and chlorobenzilate in two Florida soils. Department of Soil Science and Food Science, Univ. of Florida, Gainesville - unpublished.

Spain. Submission on GAP, MRLs, residue study reports on lemons and oranges, eggplant, artichoke, strawberry, beans, squash, cucumber, pepper and melons.

Spare, W.C. 1989. Metabolism of bromopropylate in greenhouse grown citrus spray treated with [^{14}C]bromopropylate. Agrisearch Proj. No. 12151, Ciba-Geigy Corp., USA - unpublished.

Suter, P. 1982a. Degradation of bromopropylate in two soils under aerobic conditions. Proj. Rep. 34/82, Ciba-Geigy Basle - unpublished.

Suter, P. 1982b. Leaching characteristics of aged [^{14}C]bromopropylate residues in two soils. Proj. Rep. 31/82, Ciba-Geigy, Basle, August 1982, unpublished.

CARBOFURAN (096)

EXPLANATION

A 2 mg/kg limit for the sum of carbosulfan, carbofuran, 3-hydroxy-carbofuran and 3-keto-carbofuran in citrus fruits was estimated by the 1984 JMPR. The 1991 JMPR revised the definition of the residue to separate the carbosulfan limit from that of its metabolites. This resulted in separate 2 mg/kg limits, one for carbosulfan and one for the sum of carbofuran and 3-hydroxy-carbofuran to replace the original proposal. The estimate for the sum of carbofuran and 3-hydroxy-carbofuran was intended to accommodate residues resulting from the use of carbosulfan. Keto-carbofuran was deleted from the definition. The numerical levels were not revised, pending the evaluation of additional information submitted too late for review. Critical supporting information had been requested since 1984 and additional requirements added in 1991. Some delegations to the CCPR had expressed the view that the proposed limits were not supported.

The Meeting received additional data on residues of carbosulfan and its metabolites in citrus and other commodities resulting from the use of carbosulfan, and comments from the German government explaining its view that the 2 mg/kg proposals were not supported by the data provided.

Summary residue data and information on Spanish GAP for applications of carbofuran to potatoes, onions and tomatoes were also received (Spain, 1993).

USE PATTERN

The Meeting received information on Spanish and Australian GAP for carbofuran on several commodities which is summarized in Table 1.

Table 1. Summary of registered or authorized uses of carbofuran on selected commodities.

Country/Crop	Application			PHI (days)	Comments
	Formu-lation	Rate, kg ai/ha	No.		
Spain					
Banana	SL	5.6	?	60	Drop irrigation
Cotton	GR	0.6-0.75	?	60	In soil at seeding
Maize	GR	0.6-0.75	?	60	same
Potato	GR	0.6-0.75	?	60	same
Sorghum	GR	0.6-0.75	?	60	same
Sugar beet	GR	0.6-0.75	?	60	same
Sunflower	GR	0.6-0.75	?	60	same
Tomato	SL	0.8	?	45	Drop irrigation (Field or glasshouse)
Australia					
Rice	GR	1	2	42	Not for upland rice
Sugar cane	GR	3 or (4.5 gai/ 10 M row)	1	30 wk.	Incorporate by irrigation or tillage in 21 cm band either side of row at 3-5 leaf stage.

RESIDUES RESULTING FROM SUPERVISED TRIALS

Summary data were provided by the Spanish government from supervised trials on potatoes, onions and tomatoes (Spain, 1993).

Potatoes. In one of two supervised trials in Germany in 1972 residues of carbofuran were 0.05 mg/kg 160 days after one application of a granular formulation at 5 kg ai/ha. In the other residues were 0.23 mg/kg after 106 days. No data were provided at the 60-day Spanish GAP PHI, nor was other additional supporting information provided.

Onions. In a 1984 supervised trial in Spain on onions carbofuran residues were 0.19 and 0.7 mg/kg and 3-hydroxy-carbofuran 0.28 and 0.39 mg/kg on onion plants 106 days after the application of a granular formulation at 0.92 mg/kg. Residues in the bulb were <0.05 mg/kg for both carbofuran and 3-hydroxy-carbofuran. No GAP information was provided for onions.

Tomatoes. Summary data were available from 4 supervised trials in Spain (Table 2). Residues of both carbofuran and 3-hydroxy-carbofuran were <0.05 mg/kg after 30 and 60 days from applications of 6.3 kg ai/ha compared to the Spanish GAP PHI of 45 days and application rate of 0.8 kg ai/ha. No other supporting information was provided.

Table 2. Residues in tomatoes resulting from supervised trials in Spain.

Year	Application			PHI (days)	Residues (mg/kg) at given Interval		Ref.
	Formu-lation	Rate, kg ai/ha	No.		Carbofuran	3-OH-Carbofuran	
Not given	Not given	4.2	Not given	0 7 15	<0.05 <0.05 8.1	<0.05 <0.05 <0.05	HUK 73/70 B
		8.4	Not given	0 7 15	<0.05 <0.05 0.12	<0.05 <0.05 <0.05	
1982	SC	6.3[1]	2	30	<0.05	<0.05	FC 47
	SC	6.3[1]	2	60	<0.05	<0.05	

[1] Indoor. Not indicated for other trials.

NATIONAL MAXIMUM RESIDUE LIMITS

National MRLs reported to the Meeting are summarized below.

Crop	Country	MRL (mg/kg)
Banana	Australia	0.1
	Spain	0.1
Cotton	Spain	0.1
Edible offal	Australia	0.05
Eggs	Australia	0.05
Maize grain	Spain	0.1
Maize fodder	Spain	2.0
Meat	Australia	0.05
Meat of poultry	Australia	0.05
Milk	Australia	0.05
Potato	Spain	0.2
Rice	Australia	0.2
Sugar cane	Australia	0.1
Water	Australia	0.03
Wheat	Australia	0.2

APPRAISAL

The 1992 CCPR held the 2 mg/kg carbofuran citrus limit at Step 7B pending a JMPR review of country comments to be submitted. The Meeting considered comments from Germany on the proposed citrus limit, as well as supervised trials data for potatoes, onions and tomatoes. In addition to being only summarized, the data for potato did not reflect the GAP PHI. No GAP information was provided for onions. For tomato important information was missing for two of four trials. The Meeting did not consider the summary data with no additional supporting information from limited trials sufficient for estimating maximum residue levels.

RECOMMENDATIONS

Because the current 2 mg/kg temporary limit for carbofuran in citrus fruits was recommended to accommodate residues resulting from the use of carbosulfan and because withdrawal of the limit for carbosulfan is recommended (see the monograph on carbosulfan), the Meeting also recommends withdrawal of the current temporary limit for carbofuran in citrus fruits.

Definition of the residue: sum of carbofuran and 3-hydroxycarbofuran

CCN	Crop	Maximum residue limit (mg/kg)	
		new	previous
FC 0001	Citrus fruits	W[1]	2 T

[1] withdrawn

REFERENCES

Spain, 1993. Information on GAP and summary data for potato, onions and tomato:

Crop	Report No.
Potato	197/72; 62/72
Onion	FCC 68/1; ?/72
Tomato	HUK 73/70 B; FCC 47

CARBOSULFAN (145)

EXPLANATION

Carbosulfan was first reviewed for residues by the 1984 JMPR. Although substantial data were reviewed for a number of commodities, the Meeting recommended only a temporary limit for citrus fruits (as the sum of carbosulfan, carbofuran, 3-hydroxy-carbofuran and 3-keto-carbofuran) pending the submission of required information. Critical supporting studies and GAP information relevant to citrus were especially needed.

At the request of the CCPR the 1991 Meeting reconsidered the definition of the residue with a view to making it compatible with that of its major metabolite carbofuran (also a pesticide). It recommended that limits for carbosulfan should be for carbosulfan *per se* and that separate limits should be set for the sum of carbofuran and 3-hydroxy-carbofuran to accommodate residues resulting from the use of carbosulfan or carbofuran. Keto-carbofuran was deleted from the definition.

The 1991 JMPR reviewed additional GAP and residue data for citrus, but other data on citrus and other crops were received too late for consideration. The Meeting decided not to propose a new limit for citrus, pending review of the additional data and of additional required information. Information required for the 1993 JMPR included:

1984 JMPR

1. GAP information relevant to data from supervised tirals.
2. Root crop metabolism studies.
3. Identification of residues found in ruminant tissues and milk.
4. A conventional ruminant feeding study.
5. Identification of residues in eggs from metabolism studies with ring-labelled carbosulfan.
6. Further information on the times and conditions of storage of Brassica samples.

1991 JMPR

7. Clarification of conflicting information on Spanish citrus GAP.
8. Information on sampling-to-analysis intervals in Israeli trials on oranges (FMC, 1991b).

The present Meeting reviewed data on a number of crops submitted too late for the 1991 JMPR, a manufacturer's response to requirements 2, 3, 4, 5, 7 and 8 above (an index of relevant reports with a summary/overview and/or selected pages of each), and comments from the German government explaining its view that the current 2 mg/kg TMRLs for carbosulfan and carbofuran in citrus fruits were not justified.

USE PATTERN

The information provided is summarized in Table 1. Because the use patterns available to the 1984 JMPR are out of date and because they were in any case incomplete, Table 1 includes only the new information provided to the present Meeting and that submitted too late for review in 1991. If conflicting GAP information has been supplied, it has been assumed that the most recently submitted is current and only that is listed.

Table 1. Summary of registered or authorized uses of carbosulfan on selected commodities

Crop/ Country	Formu-lation	Application		PHI (Days)	Comments
		kg ai/ha (g ai/hl)	No.[1]		
Citrus					
Brazil	250EC	(10-12.5)		7	foliar, 8l/tree
Cyprus	250EC	(12.5-50)	>1	14	
Israel	250EC	(12.5-25) 1.25		60	high volume low volume
Spain	250EC[2] 25%LE[2]	(25-37.5) (50-75)	repeat	28	
Tunisia	250EC	(50)		20	
Greece	250EC	(25-50)		60	
Thailand	250EC	(37.5-62.5)		15	
Cotton seed					
Spain	250EC 25%LE	(37.5-50) (50-75)	-	28	
Hops					
Spain	25%LE	(50-75)	-	28	
Maize					
Spain	GR	0.6[3]	-	60	application to soil
Melons					
Spain	25%LE	(50-75)	-	21	
Pome fruit					
Spain	250EC 25%LE	(37.5-50) (50-75)	-	28	
Potatoes					
Spain	250EC 25%LE	0.38-0.5[3] (50-75)	- -	28	
Sorghum					
Spain	GR	0.6[3]	-	60	application to soil
Stone fruit					
Spain	250EC 25%LE	(50)* (50-75)	-	28	* peaches
Sugar beet					
Spain	GR	0.6[3]	-	60	
Watermelon					
Spain	25%LE	(50-75)	-	21	

[1] In most cases the number of applications was not given.

[2] For Spanish GAP, LE refers to uses of an EC formulation reported by the Spanish government and usually confirmed by one of two labels. EC refers to uses reported by the manufacturer and and confirmed by the other label. All

Spanish information refers to registered uses to be considered by the EU. There is at least the appearance of a conflict where both LE and EC are recorded, as noted by the 1991 JMPR. No explanation was provided to the present Meeting.

[3] Corrects an obvious error in the 1991 JMPR evaluation (Table 1, p.215).

RESIDUES RESULTING FROM SUPERVISED TRIALS

In plants

The 1984 JMPR required further information on storage conditions and times for Brassica samples. Additional field trials with shorter PHIs might be needed, depending on the answer. No further information was provided.

Extensive data on a variety of crops were examined by the Meeting. Unfortunately, most of it was merely summary information, much of it had been previously reviewed and some submitted several times. Summaries without accompanying detailed reports were not useful to the Meeting, and generally were not evaluated. Those data which included full field reports or were otherwise sufficiently detailed were considered.

Citrus. A 2 mg/kg temporary MRL the sum of carbosulfan, carbofuran, 3-hydroxy-carbofuran and 3-keto-carbofuran in citrus was recommended by the 1984 JMPR pending the submission of required information. The 1991 JMPR reviewed additional data and replaced the recommendation by separate limits for carbosulfan alone and for the sum of carbofuran and 3-hydroxycarbofuran, both at the previous 2 mg/kg level, pending the evaluation of required information and of data submitted too late for review by the 1991 JMPR. Clarification of apparently conflicting Spanish GAP was requested (see Table 1 notes above). The German government provided comments to the Meeting on its view that a 2 mg/kg limit is not justified for carbosulfan or carbofuran.

As noted in Table 1 GAP information is available for Brazil, Cyprus, Israel, Spain, Tunisia, Greece and Thailand. The Meeting considered data reviewed in 1984 (from the USA and Italy) and in 1991 (from Brazil and Israel), together with data submitted too late for review in 1991 (a Spanish submission of Italian data (Report FFC40A/82583) reviewed by the 1984 JMPR) and Spanish data on peel and pulp residues (Report FCC 408/82584), apparently not previously reviewed. Brazilian and Israeli data reviewed by the 1991 JMPR were re-submitted, but the re-submitted Israeli results included residue levels in the whole fruit which were not in the 1991 submission. The 1991 JMPR assumed the peel to pulp ratio in the Israeli results in order to estimate whole fruit residue levels, but could not do so for the Brazilian data since only pulp residues were provided. A brief summary of the available data with comments is provided below.

1984 JMPR (see 1984 Monograph, Table 3c). Most data were from US trials, although the use is not GAP in the United States. The results could not be linked to application rates reported for other countries, since the rates in the trials were 0.94 to 4.6 kg ai/ha whereas the reported GAP was in terms of g ai/hl. The Israeli GAP was in terms of kg ai/ha (1.25), but the Israeli PHI is 60 days compared to the maximum of 28 days in the US trials. The GAP PHI is 7 days for Brazil and 28 days for Spain. Maximum residues in the US trials at 7 and 28 days were as shown below.

	Residues, mg/kg			
	Carbosulfan	Carbofuran (A)	3-OH Carbofuran (B)	$\overline{A}$+B
Oranges:				
7 days	0.01	0.05	0.03	
0.08				
	0.03	0.3	0.4	0.7
	0.02	0.2	0.4	0.6
	0.06	0.6	0.6	1.2
	0.05	0.2	0.7	0.9
	0.01	0.05	0.02	
0.07				
	0.9	0.9	0.4	1.3
	0.4	0.9	0.2	1.1
28 days	ND	0.03	0.01	
0.04				
	ND	0.28	0.41	
0.69				
	0.01	0.17	0.34	
0.51				
	0.02	0.26	0.34	0.6
	<0.01	0.01	0.02	
0.03				
	0.11	0.31	0.22	
0.53				
	0.1	0.51	0.09	0.6
Grapefruit:				
7 days	0.01	0.2	0.2	0.4
	0.09	0.7	0.5	1.3
28 days	ND	0.17	0.23	0.4
	0.02	0.62	0.46	1.1
	ND	0.19	0.42	0.6

Residues in oranges in Italian trials which conformed to reported Spanish GAP of 75 g ai/ha and a 28-day PHI were as shown below.

Day	mg/kg			
28	0.5	1.5	0.2	1.7
	0.3	0.8	0.08	0.9
42	0.3	1.1	0.06	1.2
	0.3	1	0.05	1.1
56	0.7	0.08	--	
0.08				
	0.3	0.7	0.05	0.8

Spanish peel and pulp residues (FMC 1983). Field reports were not available to determine the formulation used, the PHIs or other important information. Application rates were referred to as 0.15 or 0.2%, but the meaning of this could not be determined nor related to Spanish GAP. Residues of carbosulfan, carbofuran and 3-hydroxy-carbofuran were reported as <0.05 mg/kg in pulp, although one sample gave an unquantifiable positive response for the hydroxy metabolite. Peel residues were reported as <0.05 mg/kg carbosulfan and 0.6/0.2, 0.6/0.2, and 0.8/0.3 mg/kg carbofuran/3-hydroxy-carbofuran. If it could be assumed that the trials represented GAP and that the peel to pulp ratio was 30:70, a residue in the whole fruit of approximately 0.3 mg/kg could be estimated for the sum of carbofuran and 3-hydroxy-carbofuran. This estimate is not in good agreement with estimates based on the Italian results under the conditions of Spanish GAP shown above.

Israeli data. Although the Israeli data were summarized by the 1991 JMPR, the levels in the whole fruit were only estimates based on an assumed peel

to pulp ratio. The new whole fruit data (FMC, 1984) for single applications are summarized below to show the sum of carbofuran and 3-OH carbofuran.

		mg/kg			
Rate	PHI, days	carbosulfan	carbofuran (A)	3-OH-carbofuran (B)	A+B
12.5 g ai/hl	90	<0.05	<0.05	<0.05	0.1
		<0.05	<0.05	0.07	0.12
		<0.05	0.06	0.08	0.14
		<0.05	0.21	0.18	0.39
		<0.05	0.07	0.13	0.2
		<0.05	0.12	0.08	0.2
1.25 k gai/ha	60	<0.05	0.11	0.08	0.19
		<0.05	0.06	<0.05	0.11
		<0.05	0.05	0.13	0.18
	120	<0.05	0.05	0.13	0.18
		<0.05	<0.05	0.08	0.13
		<0.05	0.07	0.12	0.19
Controls		<0.05	<0.05	<0.05	

All of these results are from treatments within Israeli GAP, although only from a single application, and 12.5 g ai/hl is only half the maximum concentration permitted Israel. The difference between a 60-day and a 120-day PHI appears to have little effect on the levels. The maximum residues within Israeli GAP (half the maximum GAP concentration) were therefore <0.05 mg/kg carbosulfan and 0.4 mg/kg carbofuran + 3-OH carbofuran.

Brazilian data. As noted above the 1984 submission was re-submitted to the Meeting, but with more detail including the whole fruit values. No residues (<0.05 mg/kg) were reported for carbosulfan, carbofuran or 3-hydroxy-carbofuran 7 to 14 days after single applications of a 250 EC formulation at 150, 200 or 400 g ai/ha. However, these application rates could not be compared to the g ai/hl concentrations reported as Brazilian GAP.

Hops. Extensive data from Germany were available to the 1984 JMPR on green, dry and spent hops and beer, based on supervised trials in Germany with 6 applications of an EC formulation at 9.75 kg ai/ha and sampling at 0, 7, 14, 21 and 28 days after the last application. No MRL was recommended because no GAP information was provided. Information on Spanish GAP (50-75 g ai/hl, 28-day PHI) and what appear to be the data reviewed by the 1984 JMPR were provided to the Meeting (Spain, 1993). The 14-, 21- and 28-day results are shown in more detail below in Table 2, which also gives the g ai/hl concentrations which can be compared to Spanish GAP. All 28-day residues reflect Spanish GAP, although 37.5 g ai/hl is only half the reported maximum allowable concentration. Maximum residues in any hops after 28 days were 2 mg/kg carbosulfan, 0.2 mg/kg carbofuran and 7.8 mg/kg 3-hydroxy-carbofuran, and in dry hops 2.1 mg/kg carbosulfan, 0.2 mg/kg carbofuran and 6.5 mg/kg 3-hydroxy-carbofuran. The highest combined residues of carbofuran and 3-hydroxy-carbofuran were 8 mg/kg in green hops and 6.7 mg/kg in dry hops. Considerable variation was observed in the results. As also indicated in the 1984 monograph, the maximum residues in beer and spent hops 21 days after the same treatments to hops were <0.05 mg/kg.

Table 2. Residues of carbosulfan and metabolites in hops from supervised trials in Germany, all with 6 applications at 9.75 kg ai/ha and 37.5 kg ai/hl. Spanish GAP is 50-75 g ai/hl with a 28-day PHI.

Year Location /Variety	Green or Dry	PHI, Days	Residues (mg/kg)	Ref.

			carbosulfan	carbofuran	3-OH-carbofuran	
1981						
Pischlsdorf /Hersbrucker	green	14	1.4	0.3	22	73/21
		21	0.6	0.2	8	
		28	0.4	0.1	6	
	dry	14	2	0.8	17	
		21	1.1	0.3	7.4	
		28	0.6	0.2	5.3	
Pischlsdorf /Hallertauer	green	14	1.1	0.3	9.8	73/21
		21	2.2	0.4	9.5	
		28	0.3	0.07	4.8	
	dry	14	0.9	0.3	12	
		21	3.4	0.9	7	
		28	0.4	0.09	5.1	
Pischlsdorf /N. Brewer	green	14	1.4	0.9	18	73/21
		21	1.9	0.2	7.6	
		28	2	0.2	7.8	
	dry	14	1.7	1.6	19	
		21	1.4	0.3	11	
		28	2.1	0.2	6.5	
1982						
Durren Mungenau /Spalter	green	14	0	0	0.08	73/32
		21	0	0	0.2	
		28	0	0	0.2	
	dry	14	0.09	0	0.4	
		21	0.4	0.1	1.5	
		28	0.07	0	0.3	
Pischlsdorf /N. Brewer	green	14	0.2	0.3	0.8	73/32
		21	0.08	0	0.6	
		28	0	0	0.7	
	dry	14	0.3	0.09	0.8	
		21	0.2	0.09	0.7	
		28	0.1	0.06	1.4	
Pischlsdorf /Hallertauer	green	14	0.3	0.07	1.9	73/32
		21	0	0.1	1	
		28	0.6	0.06	0.8	
	dry	14	0.4	0.1	8.1	
		21	0.7	0.5	2.8	
		28	0.2	0.06	1.2	
Obermecken-Beuren /Tettnanger	green	14	0.1	0	1.2	73/32
		21	0	0	1.5	
		28	0	0	1.1	
	dry	14	0.3	0.07	2.5	
		21	0.2	0.2	1.2	
		28	0.08	0	0.3	
Pilschsdorf /Hersbrucker Spät	green	14	0.2	0.06	2.1	73/32
		21	0.1	0	2.8	
		28	0.2	0.06	2.5	
	dry	14	0.6	0.1	33	
		21	0.04	0.3	3	
		28	0.2	0	0.06	
Controls	green	21	<0.05-0.04	<0.05	<0.05	
	dry	21	<0.05	<0.05-0.1	<0.05-0.08	

Melons. Residue trials with carbosulfan on melons have not previously been reviewed. The Spanish government provided Spanish GAP (50-75 g ai/hl, 21-day PHI) and data from 1989 indoor supervised trials in Spain (Spain, 1993, Table 3). Maximum residues of carbosulfan in whole fruit under Spanish GAP conditions were 0.01 mg/kg and no residues (<0.001 mg/kg) of carbofuran were detected. Samples were not analyzed for 3-hydroxy-carbofuran.

Table 3. Residues of carbosulfan and carbofuran in melons resulting from 1989 supervised trials in Spain (Spain, 1991). All single applications of EC.

Spray concn. (g ai/hl)	Portion of crop	Interval, days	Residue, mg/kg

			Carbosulfan	Carbofuran
(38)	whole fruit	0	0.2, 0.2	-
	peel	0	0.3, 0.4	-
	pulp	0	-	-
	whole fruit	2	0.1, 0.2	-
	peel	2	0.2, 0.4	NQ, NQ
	pulp	2	-	-
	whole fruit	14	NQ[1]	-
	peel	14	NQ, 0.002	-
	pulp	14	-	-
	whole fruit	21	-	-
	peel	21	-	-
	pulp	21	-	-
(74)	whole fruit	0	0.3, 0.2	-
	peel	0	-	NQ, NQ
	pulp	0	-	-
	whole fruit	2	0.2, 0.3	-, 0.083
	peel	2	0.4, 0.7	-, 0.2
	pulp	2	-	-
	whole fruit	14	0.03, 0.04	-
	peel	14	0.05, 0.09	-
	pulp	14	-	-
	whole fruit	21	0.01, 0.01	-
	peel	21	0.03, 0.02	-
	pulp	21	NQ, NQ	-

[1] Not quantifiable. A dash is presumed to mean that no residue was detected at the reported 0.001 mg/kg limit of detection for carbosulfan or carbofuran.

Pome fruit. The 1984 JMPR reviewed apple studies from the United States, the United Kingdom and Italy. No limits were recommended because (1) the use is not authorized in the USA, (2) critical supporting information was not provided for many of the studies and (3) the information on GAP was not relevant to the trials. Desirable information on storage conditions and intervals for pome fruit was not provided.

The Meeting received information on Spanish GAP, and summarized reports of supervised trials on apples in Italy, the UK and the USA (Spain, 1993). Most of the trials reports had been reviewed by the 1984 Meeting, as had a summary report of US pear trials which was also supplied.

Of the Italian data only the trials reviewed in 1984, for which there was no critical supporting information, include the Spanish application rates and 28-day PHI (maximum residues reflecting GAP of 1.2 mg/kg carbosulfan, 0.9 mg/kg carbofuran, 0.4 mg/kg 3-hydroxy-carbofuran). Additional summarized results from 1983 Italian trials provided to the present Meeting were from applications at Spanish GAP rates, but the PHI was 100 days. After 28 days in the UK trials reviewed in 1984 maximum residues were 1.1 mg/kg carbosulfan, 1.1 mg/kg carbofuran and 0.8 mg/kg 3-hydroxy-carbofuran, but the kg ai/ha application rates could not be compared with the g ai/hl concentrations specified in the Spanish GAP. In the additional UK trials reported to the Meeting maximum residues after 28 days were 0.6 mg/kg carbosulfan, 0.25 mg/kg carbofuran and 0.17 mg/kg 3-hydroxy-carbofuran, but again only summaries were provided and the application rates could not be compared with Spanish GAP.

Potatoes. Data from French, Italian and United Kingdom trials were reviewed by the 1984 JMPR. Critical supporting information was sketchy and metabolism studies on a root crop after both foliar and soil applications were required. The Meeting received summary information on metabolism (see "Fate of residues") and a summary of the French and United Kingdom supervised trials data which were reviewed in 1984 (Spain, 1993). Of 13 trials, the PHIs in two were shorter than the Spanish 28-day PHI, two were close to it and the rest were 66 to 207 days.

Stone fruit. Carbosulfan trials on stone fruit have not previously been reviewed by the JMPR. The Meeting was provided with Italian data on nectarines, French data on peaches and summary Spanish data on peaches (Spain, 1993, Reports E88-312 and E88-314). Only the Italian and French data included adequate field reports giving critical information such as storage interval and conditions. However, it could not be determined from

the French data whether the residues (maximum 0.2 mg/kg after 37 days) were of carbosulfan alone or carbosulfan and metabolites (probably the latter). The Spanish Report E88-312 showed maximum total residues of carbosulfan and (unidentified) metabolites of 0.44 mg/kg from Spanish GAP rates and at the 28-day Spanish PHI. Maximum residues of 1 mg/kg after 28 days were reported in E88-314, but whether these were total residues or only carbosulfan was not indicated. Only the Italian results were marginally adequately reported, and these are summarized in Table 4.

Table 4. Residues of carbosulfan, carbofuran and 3-hydroxycarbofuran in nectarines resulting from supervised trials in Italy (1983) with an EC formulation (Spain, 1991).

Application		Interval (days)	Carbosul-fan	Carbofuran (A)	3-OH Carbofuran (B)	A+B	Report
Rate kg ai/ha (g ai/hl)	No.						
0.7 (50)	1	83	<0.05	<0.05	0.05	<0.1	73/39
	1	83	<0.05	<0.05	0.07	<0.12	
0.53 (30)							
	1	83	<0.05	<0.05	<0.05	<0.1	
			<0.05	<0.05	<0.05	<0.1	
Controls							
			<0.05	<0.05	<0.05	<0.1	

Sugar beet. Extensive data from supervised trials in France and the United Kingdom were reviewed by the 1984 JMPR, but MRLs were not recommended because information on relevant GAP and critical supporting studies were lacking and a root crop metabolism study was required. The Meeting received summary Italian data indicating residues (whether of carbosulfan or carbosulfan plus metabolites was not indicated) of <0.05 mg/kg 79 to 120 days after treatments at 0.6 to 1 kg ai/ha (Spain, 1993). Spanish GAP requires 0.6 kg ai/ha soil applications and a PHI of 60 days. Most results from in-furrow treatments reviewed by the 1984 JMPR were after 104 days or longer. Maximum residues were 0.3 mg/kg (most were ≤0.06 mg/kg at these extended PHIs) from Spanish GAP rates and 0.4 mg/kg from double rates. In the trials reviewed in 1984 residues of both carbofuran and 3-hydroxy-carbofuran in the roots were <0.05 mg/kg.

In animals

The 1984 JMPR required, or listed as desirable, information on residue levels in various animal tissues and milk. The Meeting received an index of relevant reports not previously provided, together with a manufacturer's summary or overview and selected pages or tables from them.

A conventional ruminant feeding study was specifically required. Summaries and selected tables were provided on carbosulfan and cholinesterase-inhibiting metabolites in cow tissues (Tilka, 1982) and milk (Leppert, 1982); carbosulfan phenolic residues in cow milk and tissues (Witkonton, 1982a); residues of dibutylamine in cow milk and tissues (Witkonton, 1982b) and methods of analysis for dibutylamine in milk and tissues (Witkonton, 1982c). These are all referenced under FMC, 1993, Volume 2.

Desirable information on residues of 3-hydroxy-*N*-hydroxy-carbofuran and dibutylamine in poultry tissues and eggs was not provided.

FATE OF RESIDUES

The 1984 Meeting required or desired information on the fate of residues in specific plants and animals. The requests were reiterated in 1991. The index of reports with the summary and selected pages or tables mentioned above included references to the fate in plants and animals.

In animals

The 1984 JMPR required the identification of residues found in ruminant tissues and milk and in eggs resulting from feeding ring-labelled carbosulfan. Summaries aand selected extracts of relevant reports were provided but were not reviewed in the absence of the complete reports.

In plants

The 1984 JMPR required a metabolism study on a root crop, after uptake from both foliar and soil treatments. A summary and selected information from a carbosulfan metabolism study on sugar beet (Robinson, 1982) were provided but were not reviewed in the absence of the complete reports.

A citrus metabolism study, recorded as desirable in 1984, was not provided.

In storage and processing

Desirable studies were not provided.

Stability of pesticide residues in stored analytical samples

The additional storage stability data listed as desirable were not provided.

NATIONAL MAXIMUM RESIDUE LIMITS

The Spanish MRLs listed below were reported to the Meeting.

Crop	MRL (mg/kg)
Citrus	2
Cotton seed	0.1
Hops	2
Maize	0.1
Maize fodder	2
Melons	0.1
Pome fruit	1
Potatoes	0.2
Sorghum	0.1
Sorghum fodder	2
Sugar beet	0.1
Water melon	0.1

APPRAISAL

Carbosulfan was first reviewed for residues by the 1984 JMPR. Although extensive data were reviewed for a number of commodities, the Meeting recommended only a temporary limit for citrus fruits pending the submission of required and desirable information, in particular critical supporting studies and GAP information.

The 1991 Meeting recommended that limits for carbosulfan should be for carbosulfan *per se* and that separate limits should be set for the sum of carbofuran and 3-hydroxy-carbofuran to accommodate residues resulting from the use of carbosulfan or carbofuran. Keto-carbofuran was deleted from the definition. The 1991 JMPR decided not to propose a new limit for citrus fruits, pending the review of additional data received too late for consideration. Additional requirements were added to those listed by the 1984 Meeting.

The Meeting received a discussion paper, summary comments and selected pages and summary tables from a number of reports on the identification and fate of residues in animal products in response to the 1984 JMPR requirements. The Meeting did not review these documents in the absence of the complete reports from which the summaries came. The required information on brassica vegetables was not provided. GAP information and

trials data on a number of commodities were also provided, much but not all of which had been reviewed by the 1984 JMPR.

Citrus. The 1984 JMPR recommended a 2 mg/kg temporary limit for citrus fruits, pending the submission of required information. The definition of the residue was revised to separate the limits for carbosulfan from those of its metabolites by the 1991 JMPR, resulting in separate limits at 2 mg/kg for carbosulfan *per se* and 2 mg/kg for the sum of carbofuran and 3-hydroxy-carbofuran. No change in the carbosulfan numerical level was proposed, pending the review of additional information. Clarification of apparent discrepancies in Spanish GAP was also requested but the GAP has not been fully clarified. One Codex delegation provided written comments explaining its view that a 2 mg/kg limit was not justified for carbosulfan or carbofuran, citing the general lack of data reflecting GAP and the low residues of carbosulfan in particular. In order to address the issues the Meeting considered newly submitted information on GAP and new and re-submitted residue trials data, and took into account data reviewed by the 1984 JMPR as well as the Codex comments.

The most extensive data (1984 JMPR) are those from trials in the USA. However, the use is not yet GAP in the United States and the Meeting could not with confidence relate the data to the GAP of other countries. Further, new data on peel and pulp residues in Spanish trials did not include important information, precluding a determination of whether the data reflected Spanish GAP (which is itself still in question). If future submissions show that all of the Spanish data reflect GAP, maximum whole fruit residues of <0.05 mg/kg carbosulfan and 0.3 mg/kg carbofuran plus 3-hydroxy-carbofuran would be indicated, assuming a peel to pulp ratio of 30:70. Re-submitted Brazilian data (<0.05 mg/kg carbosulfan, carbofuran, or 3-hydroxy-carbofuran) could not be related to the Brazilian GAP information provided.

The only data that could be related to GAP with some confidence were from Italian trials reviewed in 1984 which could be compared to Spanish GAP and Israeli data which could be compared to Israeli GAP. The Italian trials according to Spanish GAP resulted in maximum residues of 0.7 mg/kg carbosulfan and 1.7 mg/kg for the sum of carbofuran and 3-hydroxy-carbofuran. Maximum residues reflecting Israeli GAP were 0.05 mg/kg carbosulfan and 0.4 mg/kg for the sum of carbofuran and 3-hydroxy-carbofuran, although requested information on the interval from sampling to analysis to give greater confidence in the results was not provided.

Because the requested clarification of GAP and the sampling-to-analysis intervals still have not been supplied, because only a relatively small data base is available which can be compared to GAP, and because of a large discrepancy between the two sets of results which were comparable to GAP, the Meeting concluded that insufficient information had been provided to support a citrus limit for carbosulfan or carbofuran. The Meeting was informed that additional supervised citrus trials would be conducted in Spain, Brazil and Mexico.

Hops. German data for green and dry hops, spent hops and beer that were reviewed by the 1984 JMPR were re-submitted to the Meeting, together with GAP for Spain and Germany. No GAP information was provided to the 1984 Meeting. The data indicate that dry hop residues are unlikely to exceed 3 mg/kg for carbosulfan and 7 mg/kg for combined residues of carbofuran and 3-hydroxy-carbofuran after the 28-day Spanish PHI from the 37.5 g ai/hl used in the supervised trials. No data were available for the maximum 75 g ai/hl reported to be Spanish GAP. Maximum residues 21 days (German GAP PHI) after 6 applications at 37.5 ai/hl resulted in carbosulfan residues of 0.04-3.4 mg/g in dry hops and 0-2.2 mg/kg in green hops. Corresponding residues of carbofuran plus 3-hydroxy-carbofuran were 1.4-11.3 mg/kg in dry hops and 1-9.9 mg/kg in green hops. Because 6 applications were used, compared to GAP of one application, the Meeting was unable to estimate a limit for hops.

Melons. Data (indoor) were available from only two trials in one country (two results at the GAP PHI) with only one trial at the maximum application rate. No analyses were conducted for 3-hydroxy-carbofuran and critical

supporting information on sample storage conditions and the interval from sampling to analysis were lacking. It was therefore concluded that the data were insufficient to support an MRL for melons. The Meeting was informed that supervised trials on melons are being conducted in Spain.

Pome fruit. The 1984 JMPR reviewed data on apples and pears from three countries, but did not estimate a limit for pome fruit because the data base could not be related to available GAP and critical supporting information (e.g. storage conditions and intervals) was not provided. Summaries of some of the data reviewed by the 1984 JMPR were provided to the present Meeting as well as additional summary data not previously reviewed. Because only summary information was provided, because most of the old and new data could not be compared to the available Spanish GAP, and because the desirable critical supporting information still had not been provided, the Meeting concluded that the data were insufficient to support limits for pome fruit.

Potatoes. Fairly extensive data from Italy, France and the UK were reviewed by the 1984 JMPR. Summaries of these data were provided to the Meeting, but 11 of the 13 trials did not reflect the (Spanish) GAP PHI of 28 days. A discussion document and selected pages from a sugar beet metabolism study were provided in response to the 1984 JMPR requirement for a root metabolism study from both foliar and soil treatments. The Meeting concluded that data reflecting GAP were not adequate to recommend an MRL and that submission of the complete metabolism study would be needed before that requirement could be regarded as satisfied.

Stone fruit. Summary data from a substantial number of trials were available, but were not adequately reported and many of the trials did not reflect GAP PHIs for use in recommending MRLs. The trials most adequately reported were based on an 83-day PHI, whereas GAP is 28 days. The Meeting concluded that data reflecting GAP were inadequate and inadequately reported to recommend MRLs.

Sugar beet. Extensive data were reviewed by the 1984 JMPR but critical supporting information was sketchy or in some cases missing, data were not relevant to available GAP information and a root metabolism study was required. Information provided to the Meeting indicated that Spanish GAP was similar to that used in the French and UK trials reviewed in 1984, except that the Spanish PHI is 60 days whereas most of the trials results were at $\geq$104 days. Summary discussion information and selected pages from a sugar beet root metabolism study provided to the Meeting did not meet a 1984 JMPR root metabolism requirement. The full reports need to be submitted. Summary Italian data from trials at application rates greater than the Spanish GAP rates and at longer intervals than the minimum Spanish PHI were also provided. The Meeting concluded that data reflecting GAP were still inadequate to support an MRL for sugar beets. When the complete metabolism study and the detailed Italian data are provided a Meeting can reconsider the position.

Metabolism. The Meeting was informed that metabolism studies on oranges, rats, and goats and an animal transfer study on cows are being conducted.

RECOMMENDATIONS

On the basis of information provided to the Meeting, and in view of the lack of complete critical supporting studies requested since 1984 and of adequate data reflecting GAP, the Meeting recommended that the temporary limit for carbosulfan in citrus fruits should be withdrawn.

Definition of the residue: carbosulfan.

CCN	Crop	Maximum Residue Limit (mg/kg)	
		New	Previous

FC 00001	Citrus	Withdrawn	2 T

REFERENCES

FMC. 1983. Determination of Residual Concentrations of FMC 35001 and its Metabolites, Carbofuran and 3-Hyroxy Carbofuran, in Oranges From Spain (Pulp and Peel). Unpublished FMC report FCC40B/82584, submitted by the Spanish government.

FMC. 1984. The Determination of Concentrations of FMC 35001 and its Metabolites, Carbofuran and 3-Hydroxycarbofuran in Oranges From Israel. Project No. FCC 63. 1984. Submitted to the 1991 and 1993 JMPRs. Unpublished report.

FMC. 1993. Volumes 1 and 2 submitted to the 1993 JMPR by FMC Corporation.

Volume 1 - Carbosulfan/Citrus Residue Data, Supplemental Information, JMPR-1993. Tab 10 - 1985 Brazilian supervised trials data. Tab 11 - Determination of Concentrations of FMC 35001 and its Metabolites, Carbofuran and 3-Hydroxy Carbofuran in Oranges From Israel. Unpublished FMC report FCC 63, 1984.

Volume 2 -JMPR Response Carbosulfan. Includes FMC discussion and comments and selected tables and text from various reports in response to 1984 and 1991 requirements and desirables:

Cold studies - Cow tissues (Tilka, 1982 Report RAN-007); in milk (Leppert, 1982, Report RAN-0058); carbosulfan phenolic residues in cow milk and tissue (Witkonton, 1982a, Report P-0496); residues of dibutylamine residue in cow milk and tissues (Witkonton, 1982b, Report P-0516) and methods of analysis for dibutylamine in milk and tissues (Witkonton, 1982c, Report P-0501).

Label studies: ruminant tissues and milk (Wu, 1982a, Report M-4854) and carbosulfan (Wu, 1982b, Report M4875); eggs (Markle,1982, Report RAN-0050).
Metabolism Study on Sugar Beets (Robinson, 1982, Report P-0498).

Spain. 1993. Spanish GAP and Residues Studies in Apples (8), Oranges (2), Nectarines (4), Peaches (12), Melon (2), Potatoes (13), Sugar Beet (3), Hops (26).

	Report Numbers
apple	73/39; 73/25; 73/6; 73/26
oranges	FCC40A/82583;
peaches	E88-312, 314
melons	Facultad de Ciencias, Universidad de Murcia
potatoes	FCC24/28/42 821050(G); FCC 26; FCC 24/1;
Beer/hops	73/40; 73/36; 73/32B; 73/32A; 73/32; 73/21; 73/22; 73/31; 73/12
	73/12

30 June 1993 submission of the Spanish government. Most of the information was also submitted, but not reviewed, in 1991.

CHLOROTHALONIL (081)

EXPLANATION

Chlorothalonil was first evaluated in 1974. Further data were assessed in 1977, 1978, 1979, 1981, 1983, 1985, 1987, 1988 and 1990. The ADI of 0.03 mg/kg was confirmed in 1992.

This evaluation has been prepared as part of the periodic review programme of the CCPR. Information on current GAP and residue trials data have been provided by the manufacturer. A number of countries have also supplied information on GAP.

USE PATTERN

Chlorothalonil is a non-systemic protectant fungicide. Products containing chlorothalonil are used as surface contact fungicides on a range of agricultural and horticultural crops.

Registered uses in various countries are summarized in Table 1, to which the following notes apply (see also the footnotes at the end of Table 1).

1. Crops are listed alphabetically.

2. Only commodities for which data from supervised trials are available are included in the list.

Table 1. Summary of GAP in the use of chlorothalonil in various countries.

Crop	Country	Application			PHI, days
		No.	kg ai/ha	kg ai/hl	
Banana	Australia	-	1.1-2.15	-	1
	USA	-	0.875-1.625	-	0
Barley	Belgium	1	1.0	0.17-0.34	42
	Denmark	1	1.0-1.4	0.33-0.47	(GS 45)[1]
	France	2	1.1	-	-
	Ireland	1-2	0.9-1.35	0.45-0.68	(GS 59)[1]
	Luxembourg	1	1.0	0.17-0.33	42
	UK	1-2	0.9-1.35	0.45-0.68	(GS 59)[1]

Crop	Country	Application			PHI, days
		No.	kg ai/ha	kg ai/hl	
Beans	Australia	-	1-1.5	-	7
	Greece	-	-	0.225	42
	Ireland	2	1.5	-	14
	Italy	-	1.0	-	14
Beans contd.	Spain	-	1.5	0.15	15
	UK	2	1.5	-	14
	USA	4	1.2-2.5	-	7
Broccoli	Australia	-	2.5	-	3
	Canada	-	1.25-2.45	-	7
	UK	2	1.5	-	7
	USA	-	1.7	-	7
Brussels sprouts	Australia	-	2.5	-	3
	Canada	-	1.25-2.15	-	7
	Ireland	-	1.5	-	7
	Netherlands	-	1.5	-	14
	UK	2	1.5	-	7
	USA	-	1.7	-	7
Bulb onion	Australia	-	1.7	-	14
	Denmark	4-5	2.0	0.2-0.7	14
	France	-	1.5	-	-
	Greece	-	-	0.144	10
	Ireland	6	1.0	-	14
	Italy	-	1.5	-	14
	Netherlands	-	1.5	-	14
	Spain	-	1.5	-	15
	UK	6	1.0	-	14
	USA	-	0.8-1.7	-	7
Cabbage	Australia	-	2.5	-	3
	Canada	-	1.25-2.45	-	7
	France	-	1.5	-	-[2]
	Greece	-	1.4	-	10

Crop	Country	Application			PHI, days
		No.	kg ai/ha	kg ai/hl	
	Ireland	2	1.5	-	7
	UK	2	1.5	-	7
	USA	-	1.3	-	7
Carrot	Australia	-	1.3	-	7
	Canada	-	0.8-2.06	-	7
	Spain	-	-	0.12-0.15	15
	USA	-	1.3-1.7	-	0
Cauliflower	Australia	-	2.5	-	3
	Canada	-	1.25-2.45	-	7
	Greece	-	-	0.225	10
	Ireland	-	1.5	-	7
	UK	2	1.5	-	7
	USA	-	1.3	-	7
Celery	Australia	-	1.3	-	2
	Canada	-	0.8-2.06	-	7
	Greece	-	-	0.15	10 outdoors
	Italy	-	1.5	-	14
	Netherlands	-	1.875	-	28
	Spain	-	-	0.125-0.15	15
	UK	-	1.5	0.14-0.15	14
	USA	-	0.8-2.5	-	7
Cherry	Australia	-	2.3	0.12	7
	USA	4	2.6-4.6	-	shuck fall[3]
Cranberry	USA	3	3.4-5.9	-	50
Cucumber	Australia	-	1.8		1
	Canada	-	2.4	-	1
	Denmark	-	1.25 outdoor	0.15 glasshouse	3
	France	-	1.5	-	-
	Ireland	-	1.1	0.11	28
	Italy	-	1.5	-	14
	Netherlands	-	-	0.15	3

Crop	Country	Application			PHI, days
		No.	kg ai/ha	kg ai/hl	
	UK	-	-	0.11	28
	USA	-	1.3-2.5	-	0
Grapes	Australia	-	1.6		7
	France	-	0.4	-	30
	Greece	-	-	0.225	10
Melons	Australia	-	2.5	-	3
	Canada	-	2.4	-	1
	France	-	1-1.6	0.11-0.15	7
	Greece	-		0.15	10
	Italy	-	1.5	-	14
	Netherlands	-	-	0.15	3
	USA	-	1.3-2.5	-	0
Peach	Australia	-	2.3	0.12	7
	Greece	-	1.5	-	14
	Italy	-	1.0	-	21
	Spain	-	1.5	-	15
	USA	4	2.6-4.6	-	shuck fall[3]
Peanut	Australia		1.3		0
	USA		0.8-1.3		14
Potato	Australia	-	1.3	-	-
	Belgium	-	1.1-1.5	0.19-0.36	7
	Canada	-	0.6-1.3	-	1
	Denmark	10	1.25-1.75	0.3-0.4	14
	France	-	1-1.5	0.15-0.44	-
	Greece	-	-	0.15	10
	Ireland	-	1.5	-	7
	Italy	-	1.5	-	14
	Luxembourg	-	1.5	-	7
	Netherlands	-	0.6-2.2	-	3
	Portugal	-	1.5	-	7
	Spain	-	1.5	0.12-0.15	15

Crop	Country	Application			PHI, days
		No.	kg ai/ha	kg ai/hl	
	UK	-	1.5	-	0
Potato cont.	USA	8	0.6-1.3	-	7
Sugar beet	Greece	-	1.5	-	14
Summer & winter squash	Australia	-	1.8	-	1
	Canada	-	2.4	-	1
	Greece	-	-	0.15	10
	USA	-	1.3-2.5	-	0
Sweet corn	USA	-	0.6-1.7	-	14
Tomato	Australia	-	1.7	-	1 outdoors
	Belgium	-	1.4-2	-	3
	Canada	-	2.4	-	1
	France	-	1-1.6	0.11-0.15	7
	Greece	-	-	0.15	10
	Ireland	-	1.1	-	3 glasshouse
	Italy	-	1.5	-	14
	Luxembourg	-	1.5-2	-	3
	Netherlands	2	1.9-3.8	0.15	3
	Portugal	-	-	0.125-0.15	7
	Spain	-	1.5	0.125-0.15	15
	UK	-	1.1	0.11	12 hours
	USA	-	1.2-2.5	-	0

Wheat	Belgium	2	1-1.25	0.17-0.42	42
	Canada (proposed)	1	0.75-1.25	-	30
	Denmark	1	1-1.25	0.33-0.42	30
	France	2-3	1.1-1.2	-	-
	Germany	1	0.7-1.1	0.18-0.28	35-42
	Ireland	1	1.0	-	(GS 59)[1,2]
	Luxembourg	-	1.25	-	42
	Netherlands	2	1.0-1.2	-	42
	Spain	1	1.5	0.15	15
	UK	1	1.0	-	(GS 59)[1]

[1] Zadoks Growth Stage: GS 59 = ear emergence complete
GS 45 = boot swollen
[2] Only used before transplanting
[3] Latest time of treatment

RESIDUES RESULTING FROM SUPERVISED TRIALS

Extensive data were submitted for a range of crops. The trials were carried out in the USA, Australia and Europe.

The residue data are summarized in Tables 2-26.

Table 2 Cherries
Table 3 Peaches
Table 4 Cranberries
Table 5 Grapes
Table 6 Bananas
Table 7 Bulb onions
Table 8 Cabbages
Table 9 Broccoli
Table 10 Brussels sprouts
Table 11 Cauliflower
Table 12 Melons
Table 13 Cucumbers
Table 14 Summer and winter squash, Sweet corn
Table 15 Tomatoes
Table 16 Beans
Table 17 Carrots
Table 18 Potatoes
Table 19 Sugar beet
Table 20 Celery
Table 21 Barley
Table 22 Wheat
Table 23 Oats and Rye
Table 24 Peanuts

In the Tables each location listed in the left hand column represents a different site or site year. Where two or more figures appear in the 'residue' column for a particular location they represent results for separate field samples. Where reports listed replicate analytical results these are represented in the Table by their mean.

Reported residues are not corrected for recovery.

Underlined results in these Tables are those referred to in the text which are from trials where treatment regimes most closely reflected the GAP that was likely to lead to the highest residue.

Several samples were also analysed for 4-hydroxy-2,5,6-trichloroisophthalonitrile (SDS-3701, DAC-3701), 3-carboxy-2,5,6-trichlorobenzamide (SDS-46851, DAC-46851), hexachlorobenzene (HCB), and pentachlorobenzonitrile (PCBN). Residues were not found or were very low.

Cherries (Table 2). Residue data were available from a series of trials in the USA, where the maximum permitted application is 4.6 kg ai/ha at shuck fall. Chlorothalonil residues of <0.03-0.52 mg/kg were found where the use was within US GAP.

Table 2. Residues of chlorothalonil in cherries treated with an SC formulation from supervised residue trials in the USA. Last treatments were made at shuck (cot) fall

Crop Location/Year	Application		PHI, days	Residues (mg/kg)	Ref.
	kg ai/ha	No.			
Sweet Cherry Oregon/1981	3.5 4.7	4 4	76 76	0.25,0.27 0.44	1
Tart Cherry Oregon/1981	3.5 4.7	4 4	73 72	0.04, <0.03 0.04	1
Sweet Cherry N York/1981	3.5 4.7	2 2	62 62	0.03, <0.03(2) <0.03 (3)	1
Tart Cherry N York./1981	4.7 5.9	3 3	55 55	0.03 0.03	1
Sweet Cherry Michigan/1982	4.7	4	45	0.52 0.38,0.11	2
Sweet Cherry N York 1982	4.7	5	54	0.06, 0.05, 0.09	2
	9.4	5	54	0.12, 0.22, 0.09	2
Tart Cherry Michigan/1984	3.5	4	50	0.06	3

Underlined results reflect maximum permitted use in the USA.

Peaches (Table 3). In supervised trials from the USA residues in peaches were in the range <0.05-0.12 mg/kg when the treatment regime reflected maximum use within US GAP, which is a last application of 4.6 kg ai/ha at shuck (cot) fall. Residues of 0.57 and 0.98 mg/kg were reported in a trial in Italy under conditions within the official GAP of Spain and Greece (1.5 kg ai/ha and 14-15 days PHI). One of these results (0.98 mg/kg) was reported for a crop treated in accordance with the maximum permitted use in Italy (1.0 kg ai/ha and 21-day PHI).

Table 3. Residues of chlorothalonil in peaches from supervised trials in Italy, Spain and the USA.

Location/ year	Form.	Application		No.	Date of last treatment (day/month)	PHI (days)	Residue (mg/kg)	Ref
		kg ai/ha	kg ai/hl					
Italy, 1990	WP	0.84	0.1	2	-	21	0.18	10
Italy, 1990	WP	1.7 0.82	0.2 0.1	2 3	- -	21 21	0.57 0.14	11 11
Italy, 1990	SC	1.0 2.0	0.04 0.09	4 4	8/7 8/7	21 21	0.98 1.32	12 12
Italy, 1990	WP	1.5	0.1	3	shuck fall (15/4)	64	<0.01(2)	13
Italy, 1990	DG	1.25	0.09	3	shuck fall 15/4	64	<0.01(2)	14

Location/ year	Form.	Application		No.	Date of last treatment (day/month)	PHI (days)	Residue (mg/kg)	Ref
		kg ai/ha	kg ai/hl					
Spain, 1990	WP	2.0 +2.6	0.11 0.15	1	 25/4	 61	 0.16	 15
Spain 1990	WP	2.0 +2.6	0.11 0.15	1	25/4	83	0.01	16
Spain 1990	WP	2.0 +2.6	0.11 0.15	1 1	25/4	155	0.02	17
Spain 1990	SC	0.5 0.75 1.25	- - -	4 4 4	shuck fall (1/4) shuck fall (1/4) shuck fall (1/4)	82 82 82	<0.01 <0.01 <0.01	18 18 18
Spain 1991	SC	0.5 0.75 1.25	- - -	4 4 4	shuck fall (23/3) shuck fall (23./3) shuck fall (23/3)	69 69 69	<0.01 <0.01 0.01	19 19 19
USA, California 1979 USA, California 1979 USA, Oregon 1978/9	SC SC SC 	5.0 9.4 5.0 9.4 5.3 10.5 4.7 9.4 3.5 4.7	- - - - - - - - -- -	2 2 2 2 1 1 2 2 3 3	23/3 23/3 23/3 23/3 4/10 4/10 15/1 15/1 18/4 18/4	136 136 158 158 299 299 197 197 131 131	<0.05(3) <0.05(3) <0.05(3) <0.05(3) <0.05 <0.05 <0.05 <0.05 <0.05 <0.05	4 4 4 4 4 4 4 4 4 4
USA, Washington 1989 USA, Louisiana 1979 USA, Louisiana 1978 USA, California 1980 USA, California 1980 USA, Washington 1980	SC SC SC SC SC SC 	5.9 2.2 3.5 2.2 3.5 0.9 1.4 1.8 9.4 9.4 5.9 11.7	- - - - - - - - - - - -	3 1 1 3 3 12 12 12 2 3 3 3	20/4 16/6 16/6 16/6 6/3 10/3 13/5 13/5	117 81 81 58 58 12 12 12 152 133 89 89	0.90,0.08 0.06 <0.05 <0.05 <0.05 <0.05 0.28 0.76 1.00 <0.03(2), 0.03 <0.03(6) 0.23,0.80 0.37 0.19,0.20 0.28	4 4 4 4 4 4 4 4 5 5 5 5
USA, California 1980	SC 	4.7 9.4	- -	2 2	12/3 12/3	106 106	<0.03(3) <0.03(3)	5 5
USA, California 1980	SC 	4.7 9.4	- -	2 2	5/3 5/3	146 146	<0.03 (4) <0.03(4)	5 5
USA, California 1981	SC 	3.5 4.7 4.7	- - -	1 1 1	26/2 26/2 26/2	183 183 177	<0.03(3) <0.03(3) <0.03(3)	6 6 6
USA, Oregon 1981	SC 	2.7 3.5	- -	5 5	14/4 14/4	147 147	<0.03(2) <0.03(2)	6 6
USA, California 1981	SC 	3.5 4.7	-	4 4	9/3 9/3	139 139	<0.03(3) <0.03(3)	6 6
USA, California 1981	SC 	4.7		1 2 1 3	4/2 4/2 20/2 20/2	149 149 133 133	<0.03 <0.03 <0.03 <0.03	6 6 6 6
USA, Washington 1981	SC 	5.9 11.7	- -	3 3	9/5 9/5	86 86	<0.03, 0.06 0.03 0.14, 0.04,0.09	6 6

Location/ year	Form.	Application		No.	Date of last treatment (day/month)	PHI (days)	Residue (mg/kg)	Ref
		kg ai/ha	kg ai/hl					
USA, California 1983	SC	3.5*	-	3	petal fall (28/2)	100	<0.03	7
USA California 1983	SC	3.5 3.5	- -	5 5	- -	122 122	<0.03 <0.03	7 7
USA Louisiana 1985	SC	2.7 + 7.0	-	2 1	shuck fall (9/4)	84	0.03,0.02	8
USA, Florida 1985	SC	3.0 + 9.1	-	4 1	shuck fall (18/3)	84	0.03(2)	8
USA, N Carolina 1986	SC	1.8 + 4.7	-	4 1	shuck fall (8/4)	91	0.01,0.02	8
USA, Ohio 1987	SC	2.4 2.4 + 4.7 2.4 +4.7	- - -	6 4 +1 5 +1	first cover shuck fall first cover	63 73 63	0.09 0.08 0.16	9 9 9
USA, Virginia 1987	SC	2.4 +4.7 2.4 +4.7	- -	4 +1 3 +1	first cover shuck fall	88 96	0.27 0.12	0 9
USA, Louisiana 1988	SC	2.4 +4.7	-	2 +1	shuck fall	70	0.02	9
USA N Carolina 1987	SC	2.4 +4.7	-	1 +2	shuck fall	113	0.02	9
USA, N Carolina 1987	SC	2.4 +4.7	-	1 +1	shuck fall	84	<0.01	9

Results underlined once reflect maximum permitted use in the USA.
Results underlined twice reflect use within GAP in Spain and Greece; two of these results reflect maximum permitted use in Italy.

Cranberries (Table 4). GAP has only been reported for the USA, where a number of residue trials have been carried out. Although only three trials combined the highest allowed application rate with approximately the minimum PHI, the data base included other relevant trials which used 80% of the maximum rate and the minimum PHI. Residues in mature berries sampled from relevant trials were 0.67-4.1 mg/kg.

Table 4. Residues of chlorothalonil in cranberries in supervised residue trials in the USA. All products used were SCs.

Location, year	Application		PHI, days	Residue (mg/kg)	Reference
	kg ai/ha	No.			
Washington, 1982	2.35 2.35 4.7 4.7	2 2 2 2	56 70 56 70	0.38 0.11 2.9 0.69	20 20 20 20
Washington, 1983	5.9	4	54	4.1	20
N Jersey, 1984	5.9 5.9	3 3	49 49	1.3 0.67	20
Wisconsin, 1988	3.5	3	83	0.12	21
Wisconsin, 1988	3.5	3	91	0.04	21

Location, year	Application		PHI, days	Residue (mg/kg)	Reference
	kg ai/ha	No.			
Massachusetts, 1988	4.6	3	50	0.77	21
Washington, 1985	5.9	3	60	3.67	21

Underlined results are from uses similar to the maximum permitted use in the USA.

Grapes (Table 5). Products containing chlorothalonil are registered for use on grapes in Australia and a number of European countries. Maximum application rates range from 0.4 to 2.2 kg ai/ha and minimum PHIs are between 7 and 30 days. Table 5 includes new data and data previously considered by the 1983 JMPR.

In supervised trials carried out in Australia residues were 0.3-5.6 mg/kg for treatments at 0.11-0.15 kg ai//hl after PHIs of 7-28 days. The maximum permitted use in Australia is 1.6 kg ai/ha (equivalent to 0.16 kg ai/hl) with a PHI of 7 days.

Residue levels were 0.01-0.11 mg/kg in trials carried out in France which reflected the maximum permitted use in France (0.4 kg ai/ha and a PHI of 30 days).

Table 5. Residues of chlorothalonil in grapes from supervised trials in Australia, Canada, France, Germany and South Africa.

Location/year	Form.	Application		No.	PHI, days	Residue, mg/kg	Ref
		kg ai/ha	kg ai/hl				
Australia Hunter Valley 1973/4	WP	-	0.11	7	-1 0 10	3.9 6.1, 7.1 5.6 (8.6)[1]	1983 JMPR
	WP	-	0.22	7	-1 0 10	6.8 10.7 8.7 (13.4)[1]	1983 JMPR
S. Australia 1973/4	WP	-	0.13	6	1 7 18 26	1.4 0.6 1.6 (2.9)[1] 0.6, 0.3	1983 JMPR
S. Australia 1973/4	WP	-	0.26	6	1 7 18 26	2.3 3.1 2.7 (4.9)[1] 0.8	1983 JMPR
N. Australia 1991/2	SC	-	0.15 0.15 0.15	7 5 3	28 77 113	0.6 0.04 <0.01	22
Australia Hunter Valley 1990/1	SC		0.15	7 6 4	15 30 66	1.4 0.50 0.20	23
Australia Langhorne Creek 1991	SC	-	0.15 0.15 0.15	6 5 3	19 63 111	2.3 <0.02 <0.02	24
Canada Ontario 1979	WP	1.65	5	5	40	0.28, 0.26 <0.01 (2)	1983 JMPR
	WP	1.65	-	4	30	1.9, 1.6	1983 JMPR
	WP	1.65	-	3 2 1	30 30 30	3.8, 4.1 0.54 0.63, 1.0, 1.2	1983 JMPR

Location/year	Form.	Application		No.	PHI, days	Residue, mg/kg	Ref
		kg ai/ha	kg ai/hl				
France 1983	SC	0.35	-	7	61	0.02	25
	SC	0.175 0.35 0.7	- - -	7 7 7	61 61 61	<0.01 <0.01 <0.01	25
France 1984	SC	0.7	-	6	24	0.08	
France 1984	SC	0.175 0.35 0.46		9 9 9	29 29 29	0.01 0.01 0.03	
France 1986	SC	0.75	-	5	40	0.02 (2) 0.01 (2)	
France, 1987	SC	0.35	-	1	0 7 14 21 29	0.44 0.22 0.10 0.03 0.02	30
France, 1987	SC	0.35	-	1	0 7 14 21 28 42	0.50 0.28 0.09 0.06 0.02 <0.01	31
France, 1987	SC	0.35	-	1	0 7 14 21 30	0.50 0.32 0.18 0.05 0.11	32
France, 1987	SC	0.35	-	8	34	0.02	33
France, 1987	SC	0.35	-	7	27	0.01	34
France, 1987	SC	0.46	-	3	21	0.39	35
France, 1987	SC	0.46	-	4	22	0.43	36
France, 1987	SC	0.46	-	4	15	0.44	37
France, 1987	SC	0.35	-	5	44	0.04	38
France, 1987	SC	0.35	-	10	8	0.11,0.02	39
France, 1987	SC	0.35	-	8	15	0.85	40
France, 1987	SC	0.35	-	8	34	0.02	41
France, 1987	SC	0.35	-	7	27	0.02	
Germany, 1973	-	1.5	-	8	2 51	2.7 0.38	1983 JMPR
Germany, 1974	-	2.2 + 2.9 2.9	- -	6+2 8	0 21 28 35 42 0 21 28 35 42	26 17 8.0 7.0 4.2 28 14 8.0 4.8 6.7	1983 JMPR 1983 JMPR
Germany 1974	- -	1.75 2.33	- -	6 6	0 21 28 35 42 0 21 28 35 42	3.7 1.1 0.55 0.4 0.23 5.2 2.2 1.1 0.72 0.21	1983 JMPR 1983 JMPR
Germany 1975	-	-	0.15 + 0.2	8+2	0 21 28 35	3.8 0.27 0.62 0.63	1983 JMPR

Location/year	Form.	Application		No.	PHI, days	Residue, mg/kg	Ref
		kg ai/ha	kg ai/hl				
					42	0.59	
Germany 1975	-	-	0.15 + 0.18	8+2	0 21 27 35 42	1.9 0.49 0.76 0.52 0.76	1983 JMPR
Germany, 1975	-	-	0.15 + 0.2	6+4	0 21 35 43	2.4 1.1 1.8 2.0	1983 JMPR
S Africa, 1979/80	SC	1.5	-	8	1 2 4 16 32	18,21 12,13 3.6,3.5 2.7,2.5 2.6,2.3	

(Notes on next page)

[1]Figures in parentheses are corrected for recovery (64% Hunter Valley, 55% S Australia)
Results underlined once are from maximum permitted use in Australia.
Results underlined twice are from maximum permitted use in France.

Bananas (Table 6). Supervised trials carried out in Latin America and Australia included application rates similar to the maximum approved in the USA and Australia. Residue levels in treated bananas sampled 0-3 days after treatment were <0.01-2 mg/kg.

Table 6. Residues of chlorothalnil in bananas from supervised trials in Australia, Colombia, Costa Rica, Mexico and Panama.

Location/ year	Form.	Application kg ai/ha	No.	PHI, days	Residue (mg/kg)	Ref
N. Australia, 1978	SC	1.1	10	1 14 28	0.6 0.44 0.03	45
		2.2	10	1 14 28	2.0 0.10 0.09	45
Colombia, 1985	SC	1.5	11	3	<0.01(6)	44
Costa Rica, 1985	SC	1.75	10	6	0.02,0.03(2) 0.11,0.12,0.10	44
Mexico, 1984/5	WP	1.1-1.5	13	2	<0.01(6)	44
Panama, 1978	SC	1.3	8	0	<0.01(4)	43

Underlined results reflect maximum permitted use in Australia.

Bulb onions (Table 7). Products containing chlorothalonil are registered for use in the USA, Australia and several European countries. Maximum application rates range from 1.0 to 2.25 kg ai/ha and minimum pre-harvest intervals are from 7 to 28 days.

In UK trials where treatments were within Danish GAP, up to 2.0 kg ai/ha and a minimum 14-day PHI, residues were <0.01-0.1 mg/kg. In US trials where treatments reflected US GAP at 1.7 kg

ai/ha and a minimum 7-day PHI, residues were 0.02-0.06 mg/kg.

Table 7. Residues in bulb onions following treatment with chlorothalonil.

Location/ year	Form.	Application		PHI, days	Residue (mg/kg)	Ref
		kg ai/ha	no.			
Australia, 1982	SC	1.5	3 6	41 27	0.1 <0.1	48 38
Canada, 1987	SC	1.6-1.8	3	0 1 3 7 10 14	0.16 0.07 0.04 0.02 <0.01 <0.01	58
Denmark, 1981	SC	2.0	5	14	0.04	50
Denmark, 1981	SC	1.25	1	14 21	<0.01 <0.01	51
Italy, 1990	SC	1.5 3.0	2 2	14 14	<0.01 <0.01	52 52
Italy, 1990	SC	1.5 3.0	2 2	14 14	<0.01 <0.01	53 53
Netherlands, 1981	WP	1.5	7 6 5 4	7 14 21 28	0.34 0.25 0.19 0.10	54
Netherlands, 1981	WP	1.5	7 6 5 4	7 14 21 28	0.57 0.52 0.52 0.28	55
UK, 1990	SC	1.0 1.5 2.0 3.0	6 6 6 6	2 2 2 2	0.02(2), 0.03(2) 0.05, 0.06, 0.03(2) 0.03, 0.06, 0.07, 0.05 0.11(2), 0.10, 0.08	6 6 6 6
	SC	0.9	6	2	0.01	56
UK, 1990	SC	1.0 1.5 2.0 3.0	6 6 6 6	20 20 20 20	0.01 (4) <0.01 (4) <0.01 (3), 0.02 <0.01 (2), 0.03 (2)	56 56 56 56
UK, 1990	SC	1.0 1.5 2.0	6 6 6	27 27 27	<0.01 (7), 0.02, 0.03 0.05 <0.01, 0.01 (3), 0.02(5), 0.03(2), 0.04, 0.03(2) 0.07, 0.06, 0.02, 0.03,	56 56 56 56 56

Location/ year	Form.	Application		PHI, days	Residue (mg/kg)	Ref
		kg ai/ha	no.			
		3.0	6	27	0.04, 0.12	56
	SC	0.9	6	27	0.02(3)	56
UK, 1991	SC	1.0	5	14	<0.01 (2), 0.01 (3), 0.02(2), 0.03	57
		2.0	5	14	<0.01, 0.01, 0.02 (4), 0.03 (2)	57
	DG	1.0	5	14	<0.01 (2), 0.01 (4) 0.02, 0.05	57
		2.0	5	14	0.01 (3), 0.02, 0.03 (4)	57
UK, 1991	SC	1.0 2.0	5 5	14 14	<0.01, 0.01 0.06, 0.02	57 57
UK, 1991	DG	1.0 2.0	5 5	14 14	0.02, 0.01 0.05, 0.04	57 57
UK, 1991	SC	1.0 2.0	5 5	14 14	0.02, 0.05 0.05	57
	DG	1.0 2.0	5 5	14 14	0.02 (2) 0.10, 0.06	57 57
USA, California, 1985	SC	1.75	12	7	0.04	46
USA, Texas, 1985	SC	1.75	7 12	7 7	0.06 0.12	46 46
USA, Michigan, 1984	SC	1.15 + 1.75	3+6	12	<0.01	47
USA, New York 1986	SC	1.75	12	7	0.02	47

Results underlined once reflect Danish GAP.
Results underlined twice reflect US GAP.

Cabbages (Table 8). The USA, Australia, Canada and a number of European countries have reported GAP for chlorothalonil on cabbage, with maximum application rates of 1.25-2.45 kg ai/ha and minimum PHIs of 0-10 days.

Residues in crops treated at 1.3 kg ai/ha and harvested 7 days after the last treatment (within US GAP) at two sites in the USA contained residues at <0.01 or <0.03 mg/kg.

Results from trials at 3 UK sites where treatment regimes were within UK and Irish GAP (1.5 kg ai/ha, 7-day PHI) were

0.04-0.7 mg/kg.

Table 8. Residues of chlorothalonil in cabbages from supervised trials carried out in the UK and the USA.

Location/ year	Form.	Application		PHI, days	Residue (mg/kg)	Ref.
		kg ai/ha	No.			
UK 1990	SC	1.5	2	39	0.09,0.10, 0.14,0.15, 0.16 ,0.21, 0.27,0.41,0.46, 0.53	62
		3.0	3	39	0.91,1.37,1.47, 1.46	62
UK, 1991	SC	1.5	2	7	0.18,0.17	63
	DG	1.5	2	7	0.07,0.04	63
UK, 1991	DG	1.5	2	8	0.16,0.13	63
	SC	1.5	2	8	0.28,0.61	63
UK, 1991	SC	1.5	2	8	0.16,0.19, 0.20(2), 0..22,0.24, 0.28,0.53	63
UK, 1991		3.0	2	8	0.14,0.17,0.25, 0.33, 0.49,0.50, 0.69, 0.74	63
	DG	1.5	2	8	0.29,0.30,0.35, 0.38,0.42,0.55, 0.64,0.70	63
		3.0	2	8	0.24(2), 0.26, 0.28,0.34,0.60, 0.64,0.81	63
USA, N York, 1986	SC	1.3	11	0	5.0	59
USA, Georgia, 1984	SC	1.3	9	7	<0.03	60
USA, N York, 1985 USA, Florida, 1986	SC SC SC	1.3 1.3 1.3	8 11 11 11	0 0 1 7 1 7	6.4 5.9 0.23 <0.01 0.03 <0.01	60 60 61 61 61 61

Results underlined once reflect US GAP.
Results underlined twice reflect UK GAP.

Broccoli (Table 9). GAP for applications to broccoli has been reported for Australia, Canada, the UK and the USA. Maximum application rates are 1.0-2.5 kg ai/ha and minimum harvest intervals 3 or 7 days.

In two trials carried out in the USA the treatment regimes were within US and Canadian GAP. Residues in these trials were 2.2 and 2.6 mg/kg.

Table 9. Residues of chlorothalonil in broccoli from supervised trials carried out in the USA using SC formulations.

Location/ year	Form.	Application	PHI, days	Residue (mg/kg)	Ref.

		kg ai/ha	No.			
California, 1986		1.6	9	0	1.7	64
New York, 1985		1.3	6	0	5.8	65
Michigan, 1985		1.3	4	6	2.2	66
New York, 1986		1.3	8	0	11	66
New York, 1987		1.3	8	7	2.6	67

Underlined results reflect GAP in the USA and Canada

Brussels sprouts (Table 10). Products containing chlorothalonil are registered for use on Brussels sprouts in Australia, Canada, the USA and several European countries. Maximum applications rates are 1-2.5 kg ai/ha and minimum pre-harvest intervals are 3, 7 or 14 days.

A residue level of 2.3 mg/kg was reported from an Australian trial where the treatment regime was within Australian GAP (2.5 kg ai/ha, 3-day PHI). Two results (<0.01 and 4.3 mg/kg) were available from US trials reflecting US and Canadian GAP (1.7 and 1.25-2.15 kg ai/ha respectively, 7-day PHI). Residues were 0.09-0.92 mg/kg in six trials carried out in The Netherlands and the UK using treatment regimes within UK and Irish GAP (1.5 kg ai/ha, 7-day PHI).

Table 10. Residues of chlorothalonil in Brussels sprouts from supervised trials carried out in Australia, The Netherlands, the UK and the USA. All products used were SCs.

Location/ year	Application		PHI, days	Residue (mg/kg)	Ref.
	kg ai/ha	No.			
S.Australia 1977	1.3	5	0 1 6	1.3 1.0 2.2	70 70 70
	2.5	5	0 1 6	2.6 2.3 2.3*	70 70 70
Netherlands 1989	1.5	7	7 14 21	0.16,0.09,0.13,0.57 0.49,0.31,0.45,0.66 0.23,0.17,.0.14,0.29	71 71 71
Netherlands 1989	1.5	6	7 14 21	0.35,0.28,0.32,0.32 0.48,0.33,0.36,0.35 0.20,0.19,0.13,0.18	72 72 72
Netherlands 1989	1.5	6	7 14 21	0.14,0.13,0.09,0.36 0.06,0.08,0.06,0.21 0.05,0.04,0.15,0.19	73 73 73
UK, 1989	1.5	2	13	0.14,0.12	74
UK, 1989	1.5	2	12	0.09,0.12	75
UK,1989	1.5	2	12	0.09,0.05	76
UK, 1990	1.5	2	0 3 5 7 12 13	0.19.,0.29,0.18 0.28,0.19,0.19 0.14,0.14,0.21 0.35,0.37,0.18 0.19,0.33,0.28 0.26,0.19,0.23 0.23,0.24,0.17 0.28,0.21,0.27 0.15(2),0.17,0.12, 0.11,0.14 0.24,(2),0.09 0.15,0.18(3)	77 77 77 77 77 77

Location/ year	Application		PHI, days	Residue (mg/kg)	Ref.
	kg ai/ha	No.			
				0.16(2),0.12, 0.20,0.08	
	3.0	2	13	0.15,0.22,0.36,0.61	77
UK,1990	1.5 3.0	2 2	7 7	0.45,0.53,0.92,0.31 1.27,0.68,1.18,1.24	77 77
UK, 1990	1.5 3.0	2 2	7 7	0.15,0.13,0.47,0.29 0.23,0.76,0.49,0.33	77 77
USA, N York 1986	1.3	13	0 7	4.4 4.3	68 68
USA, N York 1987	1.3	9	7	<0.01	69

Results underlined once reflect US GAP.
Results underlined twice reflect UK and Irish GAP.
The asterisked result reflects Australian GAP.

Cauliflower (Table 11). Cauliflower crops may be treated with chlorothalonil in Australia, Canada, the USA and a number of European countries. Maximum treatment rates range from 1.0 to 2.45 kg ai/ha and minimum pre-harvest intervals are 0, 3 or 7 days.

Supervised trials have been carried out in the USA and the UK. The US GAP of 1.3 kg ai/ha and a 7-day PHI was used in two US trials. Residue levels in these trials were 0.12 and 0.04 mg/kg. In UK trials, samples harvested 7 days after treatment at 1.3-1.5 kg ai/ha (within GAP in the UK and Ireland) contained residues in the range 0.04-0.47 mg/kg.

Table 11. Residues of chlorothalonil in cauliflower from supervised trials in the UK and the USA.

Location/ year	Form.	Application		PHI, days	Residue (mg/kg)	Ref.
		kg ai/ha	No.			
UK, 1990	SC	1.5 3.0	2 2	32 32	<0.01(3), 0.01(4), 0.02(5) <0.01(2), 0.01(2)	80 80
UK,1991	SC DG	1.5 1.5	2 2	6 6	0.28(2) 0.45,0.42	81 81
UK, 1991	SC DG	1.5 1.5	2 2	6 6	0.19,0.25 0.32,0.47	81 81
UK,1991	SC DG	1.5 3.0	2 2	42 42	0.01,0.02(3) 0.02(4)	81 81
UK,1991	SC DG	1.5 3.0 1.5 3.0	2 2 2 2	42 42 42 42	0.01(3),0.02 0.01(2),0.02(2) 0.01(5),0.02(3) 0.01(2),0.02(2),0.03(4)	81 81 81 81
USA, Oregon 1985	SC	1.3*	8	7	0.12	78
USA, Oregon 1985	SC	1.3	8	7	0.04	78
USA, N York 1985	SC	1.3	8	0	0.41*	78
USA, Florida 1985	SC	1.3	9	0	1.8	78
USA, N York 1985	SC	1.3	6	0	4.5	79

* high apparent residues in controls suggests mislabelling.
Results underlined once reflect GAP in the USA.

Results underlined twice reflect GAP in the UK and Ireland.

Melons (Table 12). Products containing chlorothalonil are registered for use on melons in Australia, Canada, the USA and several European countries. Registered maximum treatment rates are 1.0-2.5 kg ai/ha and minimum pre-harvest intervals are 0-15 days.

Supervised trials have been carried out in the USA, France and Italy. A number of results were available for samples which had been treated at around 2.5 kg ai/ha and harvested on the day of treatment (this is US GAP); residues ranged from 0.18 to 1.45 mg/kg. Residues were <0.01-0.03 mg/kg in samples from French trials which had been treated in accordance with French GAP.

Table 12. Residues of chlorothalonil in melons from supervised trials in France, Italy and the USA.

Location/ year	Form.	Application		PHI, days	Residue (mg/kg)	Ref
		kg ai/ha	No.			
France, 1982	SC	1.1	4	6	<0.01	83
	WP	1.5	4	6	0.01	84
France, 1982	SC	1.5`	4	6	<0.01	85
France, 1989	SC	1.5	4	3	0.02	86
				7	0.03	86
Italy, 1990	SC	1.5 3.0	3 3	14 14	0.10 0.56	87 87
USA, Texas, 1980	SC WP	2.5 2.6	5 5	0 0	0.22,0.26,0.21 1.45,0.80(2) 0.30,0.24,0.18, 0.58,0.36,0.84	82 82

(Notes on next page)

Results underlined once reflect US GAP
Results underlined twice reflect French GAP.

Cucumbers (Table 13). GAP for cucumbers has been reported for Australia, Canada, the USA and several European countries. Maximum treatment rates are 1.0-2.5 kg ai/ha (or 0.08-0.19 kg ai/hl) and minimum PHIs are from 0 to 28 days.

Data were available from supervised trials carried out in the USA, France, Italy and Spain. None of these data are from crops grown under protection. Samples treated at 2.5 kg ai/ha and harvested on the day of treatment (this is US GAP) had residue levels in the range 0.43-4.3 mg/kg.

Table 13. Residues of chlorothalonil in cucumbers from supervised trials in France, Italy, Spain and the USA. All trials appear to have been carried out in the field (i.e. without protection).

Location/ year	Form.	Application			PHI, days	Residue (mg/kg)	Ref.
		kg ai/ha	kg ai/hl	No.			
France, 1989	SC	1.5	-	7	3 7	0.05 0.05	92
France, 1989	SC	-	0.15	1	0 3	0.17 0.14	93

Location/ year	Form.	Application		PHI, days	Residue (mg/kg)	Ref.
		kg ai/ha	kg ai/hl / No.			
				5 7 14 21	0.09 0.08 0.10 <0.01	
France, 1989	SC	-	0.15 / 1	0 3 5 7 14 21	0.19 0.21 0.33 0.11 0.07 0.01	94
France, 1989	SC	-	0.15 / 1	0 3 5 7 14 21	0.07 0.12 0.28 0.11 0.03 <0.01	95
France, 1989	SC	2.4	- / 1	0 1 5 10	1.2 1.0 0.26 0.15	96
		4.8	- / 1	0 1 5 10	1.5 1.7 0.61 0.42	96
Italy, 1990	SC DG	0.75 0.66	- / 2	15 15	0.02(2) 0.01	97 97
Italy, 1990	SC DG	1.5 1.3	- / 2 - / 2	15 15	0.01,0.02 0.01	98 98
Italy, 1991	SC	1.5	- / 3	0 14	0.41,0.20,0.34 <0.01(2),0.01	99
Italy, 1991	SC	1.5	- / 3	0 14	1.26,1.42,0.87 <0.01,0.01,0.02	100
Italy, 1991	DG	1.5	- / 3	0 14	0.23,0.83,0.46 0.02,0.01,<0.01	101
Italy, 1991	DG	1.5	- / 3	0 14	1.1(2),1.7 0.01,0.02(2)	102
Italy,1991	DG	1.5	- / 3	0 14	0.10,0.35,0.41 <0.01(2),0.03	103
Italy,1991	DG	1.5	- / 3	0 14	0.70,1.1,1.6 0.02(2),0.05	104
Italy,1991	SC	1.5	- / 3	0 14	0.31,0.36,0.40 0.01(3)	105
Italy,1991	SC	1.0	- / 3	0 14	0.23,0.57,0.74 <0.01(3)	106
Spain, 1990	SC WP DG	1.25 1.5 1.5 1.6	- / 3 3 3 3	7 7 7 7	0.21 0.17 0.22 0.05	107 107 107 105
USA, Michigan, 1979	SC	1.3	- / 6	0 7	0.07,0.06,0.06 0.06,0.04,0.06	88
USA, Texas, 1986	SC SC	2.5 2.5 2.5 +6.8	- / 9 - / 9 - / 8 - / 1	0 0 0 0	4.3 2.6 2.9	89 89 89
USA, S Carolina 1986	SC SC	2.5 2.5 2.5 +6.9	- / 9 - / 9 - / 8 - / 1	0 0 5	2.8 1.4 0.97	89 89 89
USA, Florida, 1986	SC SC	2.5 2.5 2.5 +6.8	- / 9 - / 9 - / 8 - / 1	0 0 0	2.3 0.43 4.0	90 90 90
USA, California 1986	SC	2.5	- / 8	0	1.7	91

Underlined results reflect US GAP.

Summer and winter squash, Sweet corn (Table 14). Products containing chlorothalonil are registered for use on summer and winter squash crops in Canada, the USA, Greece and Italy. Maximum application rates range from 1.1 to 2.5 kg ai/ha and minimum pre-harvest intervals from 0 to 10 days.

Supervised trials have been carried out in the USA. Crops harvested on the day of the last of ten treatments at 2.5 kg ai/ha (US GAP) contained residues in the range 0.59-3.6 mg/kg.

Chlorothalonil residues were not found (<0.01 mg/kg) in sweet corn when chrorothalonil was used according to GAP in supervised trials carried out at one site in the USA.

Table 14. Residues of chlorothalonil in summer squash, winter squash and sweet corn from supervised trials in the USA using SC formulations. All trials accord with US GAP.

Crop	Location/ year	Application		PHI, days	Residue (mg/kg)	Ref.
		kg ai/ha	No.			
Summer squash	Florida, 1985	2.5	10	0	0.97	108
	California, 1985	2.5	10	0	1.8,3.6	108
Winter squash	California, 1985	2.5	10	0	2.6	109
	Florida, 1985	2.5	10	0	0.59	109
	Texas, 1985	2.5	10	0	1.3	109
Sweet corn	Illinois, 1985	1.6	8	14	<0.01(2)	110

Tomatoes (Table 15). Products containing chlorothalonil are approved for use on tomatoes in Australia, Canada, the USA and several European countries. Maximum treatment rates range from 1.1-2.5 kg ai/ha and minimum pre-harvest intervals from 0 to 15 days.

Supervised trials have been carried out in the USA, Italy and Spain. When crops were treated in accordance with the maximum GAP, 2.5 kg ai/ha, and sampled on the day of treatment, residues were in the range 0.15-4.6 mg/kg.

Table 15. Residues of chlorothalonil in tomatoes from supervised trials in
a) the USA and b) Italy and Spain.

a) Trials carried out in the USA.

Location/ year	Application			PHI, days	Residue (mg/kg)	Ref
	Form.	kg ai/ha	No.			
N.Carolina, 1984	SC	1.5	16	1	0.12	111
Florida, 1984	SC	2.4	11	0	0.15	111
Florida, 1984	SC	2.4	16	0	0.18	111

Location/ year	Application			PHI, days	Residue (mg/kg)	Ref
	Form.	kg ai/ha	No.			
Florida, 1984	SC	2.4	11	0	0.49	111
Florida, 1984	SC	2.4	12	1	0.84	111
California, 1984	WP	1.6	8	0	2.4	112
Virginia, 1984	WP	1.6	6	5	0.22,0.17,0.08	112
Ohio, 1984	WP	1.6	5	5	0.64,0.23,0.36	112
California, 1984	WP	1.6	3 6	12 12	0.07,0.06 0.08,0.11	112 112
California, 1984	DG	1.8	6	1	0.36,0.97,1.1, 1.3	112
California, 1984	DG	1.8	7	1	0.37,0.41,1.5, 2.5	112
Ohio, 1985	DG	1.8	7	1	0.18,0.44,0.70	112
California, 1984	DG	1.8	5	1	0.37,0.72,1.6, 1.7	112
Florida, 1985	SC	2.5	9	-	1.4,2.7,2.8,4.6	113

b) Decline trials carried out in Europe

Location	Form.	Application			Residues (mg/kg) after PHI (days)							Ref
		kg ai/ha	kg ai/hl	No.	0	3	5	7	14	21	28	
Italy, 1989	SC	-	0.26 0.52	1 1	0.38 1.0	0.47 1.1	1.2 3.3	1.0 1.8	0.61 1.6	0.40 0.89	0.13 0.63	114
Italy, 1989	SC	-	0.26 0.52	1 1	0.61 0.88	0.73 2.2	1.1 1.9	0.61 1.9	0.50 1.8	0.51 1.4	0.19 0.43	115
Italy, 1989	DG	-	0.26 0.52	1 1	1.1 1.2	1.5 3.5	0.88 1.6	0.68 1.1	0.53 1.0	0.33 0.80	0.26 0.24	116
Italy, 1989	SC	-	0.13	3	1.2 0.13[1]	0.89 0.19[1]	0.64 0.22[1]	0.54 0.13[1]	0.29 0.15[1]	0.24 0.08[1]		117

Location	Form.	Application			Residues (mg/kg) after PHI (days)							
		kg ai/ha	kg ai/hl	No.	0	3	5	7	10	14		
Italy, 1989	SC	-	0.13	3	1.2	1.1	0.89	0.72	0.37	0.39		118
Italy, 1989	DG	-	0.13	3	1.2	1.1	0.87	0.36	0.25	0.27		119
Italy, 1990	SC	1.5	0.15	3	0.32	0.27		0.23	0.23	0.15		120
Italy, 1990	SC	1.5	0.15	3	0.55	0.33		0.24	0.21	0.17		121
Italy, 1990	SC	3.0	0.3	3	0.98	0.42		0.35	0.23	0.29		122
Italy, 1990	SC	1.5	0.15	3	0.25	0.35		0.29	0.28	0.26		123
Italy, 1990	SC	3.0	0.3	3	0.61	0.94		0.40	0.56	0.33		124
Italy, 1990	DG	1.5	0.15	3	0.13	0.27		0.28	0.20	0.11		125
Italy, 1990	DG	3.0	0.3	3	0.66	0.86		0.75	0.47	0.23		126
Italy, 1990	SC	1.1	0.11	3	0.18	0.21		0.15	0.10	0.08		127
Italy, 1990	SC	1.1	0.11	3	0.09	0.04		0.04	0.07	0.07		128
Italy, 1990	SC	3.0	0.3	3	0.32	0.15		0.16	0.17	0.16		129
Italy, 1990	SC	1.5	0.15	3	0.22	0.07		0.14	0.08	0.03		130
Italy, 1990	SC	3.0	0.3	3	0.24	0.21		0.20	0.28	0.17		131
Italy, 1990	DG	1.5	0.15	3	0.21	0.12		0.07	0.10	0.16		132
Italy, 1990	DG	3.0	0.3	3	0.49	0.40		0.31	0.19	0.30		133
Italy, 1990	SC	0.64-0.78	-	5						0.34[2]		134

Location	Form.	Application			Residues (mg/kg) after PHI (days)							Ref
		kg ai/ha	kg ai/hl	No.	0	3	5	7	14	21	28	
		1.4-1.7	-	5						0.62[2]		134
Italy, 1990	SC	0.6-0.75	-	5						0.13[2]		135
		1.1-1.6	-	5						0.32[2]		135
Italy, 1990	SC	1.5	-	3	0.08 0.12 0.23 0.25					0.09 0.15 0.08 0.11		136

					Residues (mg/kg) after PHI, days							
					0	3	5	7	14			
Italy, 1991	DG	1.5	-	3	0.15 0.24 0.30 0.37				0.10 0.13(2) 0.14			137
Italy, 1991	DG	1.5	-	3	0.16 0.24 0.28 0.29				0.06 0.09(2) 0.10			138
Spain, 1990	WP	3.4	-	4	1.3	1.0		0.71	0.49			139
Spain, 1990	SC	1.7	-	4	0.33[2]	0.32[2,3]		0.38[2]				140
Spain, 1990	SC	1.7	-	4	0.38[2]	0.73[2,3]		0.58[2]				141
Spain, 1990	SC	1.8	-	3	1.02[2]	0.69[2,3]		0.46[2,3]	0.20[2,4]			142
Spain, 1990	SC	3.5	-	3	1.02	1.2		0.71	0.33[4]			

[1] After washing
[2] Greenhouse experiment
[3] 4-day PHI
[4] 11-day-PHI

Underlined results reflect US GAP

Beans (Table 16). GAP for beans has been reported for Australia, the USA and several European countries. Maximum treatment rates are 1.5-2.5 kg ai/ha and minimum PHIs are 7-15 days.

In four supervised trials carried out on common beans in the USA and reflecting GAP at 2.5 kg ai/ha and a 7-day PHI residues levels were 0.4-3.1 mg/kg.

Table 16. Residues of chlorothalonil in beans from supervised trials in France, Italy, the UK and the USA.

a) Common beans

Location, year	Form.	Application		PHI, days	Residue (mg/kg)	Ref.
		kg ai/ha	No.			
France, 1988	SC	0.75 0.75	2 2	12 13	<0.01 0.02	146 146
France, 1988	SC	0.75	2	21	0.01	147
Italy, 1989	SC	1.5	2	0 3 5 7 10 14	1.81 0.50 0.39 0.42 0.22 0.03	148
Italy, 1989	SC	1.5	2	0 3 5 7 10 14	1.71 0.53 0.25 0.22 0.06 0.04	149

Location, year	Form.	Application		PHI, days	Residue (mg/kg)	Ref.
		kg ai/ha	No.			
Italy, 1989	DG	1.5	2	0 3 5 7 10 14	1.3 0.50 0.38 0.35 0.15 0.04	150
Italy, 1990	SC DG	1.5 1.5	1 1	16 16	0.02(2) 0.04	151
Italy, 1990	SC DG	3.0 3.0	1 1	16 16	0.28,0.02 0.13	152
UK, 1985	SC	0.9	2	76	<0.01*	153
UK, 1986	SC	1.0	2	51	<0.01*(4)	154
UK, 1986	SC	1.0	2	71	<0.01*(2)	155
UK, 1988	SC	1.5 3.0	1 1	19 19	0.78, 0.39 1.5,0.60	156
UK,1988	SC SC	0.75 1.0	1 1	19 19	0.29 0.45	157
UK, 1988	SC	1.5 3.0	1 1	7 10 14 7 10 14	0.37 0.25 0.12 1.2 0.50 0.38	158 158
UK, 1990	SC	1.5 3.0	2 2	84 84	<0.01*(3) <0.01*(3)	159 159
UK, 1991	SC	1.5 3.0	2 2	62 62	<0.01*(3) 0.14*, 0.09*,0.02*	159 159
UK, 1991	SC	1.5 3.0	2 2	49 49	0.02*(2), <0.01* 0.02*(2), 0.03*	160 160
	DG	1.5 3.0	2	49 49	0.02*,0.07* 0.04*,<u>0.02</u>*	160 160
UK, 1991	SC	1.5 3.0	2 2	60 60	<0.01* ,0.01*(3),0.02*(4) 0.02*(3),0.03*(2),0.08* 0.04*,0.05*,<0.01*,0.01*(3)	160 160
	DG	1.5 3.0	2 2	60 60	<0.01*(4),0.01*(3),0.03* 0.02*(4),0.03*(4)	160 160
UK, 1991	SC DG	1.5 3.0 1.5 3.0	2 2 2 2	57 57 57 57	<0.01*(3) <0.01*(3) <0.01*(2) <0.01*(2)	160 160 160 160
USA, Florida, 1986	SC	2.5	3	7	<u>0.40</u>	144
USA, Oregon, 1986	SC	2.4	3	7	<u>3.0</u>	144
USA, N York, 1986	SC	2.5	3	7	<u>3.1</u>	145
USA, Wisconsin, 1986	SC	2.5	4	7	<u>0.78</u>	145

b) Broad beans

Location/ year	Form.	Application		PHI, days	Residue (mg/kg)	Ref.
		kg ai/ha	No.			
UK, 1991	SC	1.5 3.0	2 2	10 10	0.98,1.0,0.77,1.4,0.86, 0.96,0.32,0.58,0.61 1.7,4.3,1.4	159 159

Underlined results reflect US GAP.

<u>Carrots</u> (Table 17). Products containing chlorothalonil are registered for use on carrots in Australia, Canada, Spain and the USA at treatment rates up to 2 kg ai/ha and with minimum pre-harvest intervals of 0 to 21 days.

Residues were in the range 0.02-0.96 mg/kg in trials in

which crops were treated in accordance with the US GAP of 1.7 kg ai/ha and a 0-day PHI.

Table 17. Residues of chlorothalonil in carrots from supervised trials in Australia and the USA according to US GAP.

Location/ year	Form.	Application		PHI, days	Residue (mg/kg)	Ref.
		kg ai/ha	No.			
S Australia, 1976	WP	1.3 2.6	15 15	0 6 0 6	2.0 2.6 1.7 3.6	165 165 165 165
USA, Texas, 1979	SC WP	1.6 1.6	6 6	0 0	0.20,0.26,0.19 0.34,0.33,0.50	161 161
USA, California, 1979	SC WP	1.6 1.6	5 5	0 0	0.30,0.10,0.07 0.46,0.64,0.96	161 161
USA, California, 1979	SC	1.6	5	0	0.88,0.82(2)	162
USA, Texas, 1979	WP SC	1.6 1.6	6 6	0 0	0.21,0.19,0.15 0.12,0.06,0.09	162 162
USA, California, 1986	SC	1.6	10	0	0.09	163
USA, Texas, 1986	SC	1.6* 1.6	10	0	0.08* 0.10	164 164
USA, Washington, 1986	SC	1.6	10	0	0.02	164
USA, Wisconsin, 1986	SC	1.6	8	0	0.03	164

Potatoes(Table 18). Products containing chlorothalonil are authorised for use on potatoes in Australia, Canada, the USA and several European countries. Maximum application rates are between 0.8 and 2.2 kg ai/ha and minimum pre-harvest intervals are 0 to 15 days.

Supervised trials data were available from the USA and several European countries. Results were available from a number of trials carried out in the USA where samples were taken 7 days after treatment, and application rates were around 1.3 kg ai/ha (US GAP is 1.3 kg ai/ha, PHI 7 days). Residues of chlorothalonil were not found (<0.03 mg/kg). Where treatment regimes reflected maximum UK GAP residue levels were 0.01-0.18 mg/kg.

Table 18. Residues in potatoes following treatment with chlorothalonil.

Location/ year	Form.	Application		PHI, days	Residue (mg/kg)	Ref
		kg ai/ha	No.			
Belgium, 1980	SC	1.5-2.2	6	7 14 29	0.01 <0.01 <0.01	170
Belgium, 1980	SC	1.5-1.8	5	14 28 41	<0.01 <0.01 <0.01	171
Greece, 1987	WP	0.9	5 6	8 8	<0.01 <0.01	172 172
Italy, 1990	SC	1.5 3.0	2 2	21 21	<0.01 <0.01	173

Location/ year	Form.	Application		PHI, days	Residue (mg/kg)	Ref
		kg ai/ha	No.			
Italy, 1990	SC WP	1.5 3.0	2 2	14 14	<0.01 <0.01	174
UK, 1989	SC WP	3.0 3.0	6 6	49 49	<0.01 <0.01	175 175
UK, 1989	WP DG	1.0-1.5 1.0-1.5	7 7	38 38	<0.01 <0.01	176 176
UK, 1989	SC WP	3.0 3.0	6 6	49 49	<0.01 <0.01	177 177
UK, 1989	WP DG	1.0-1.5 1.0-1.5	7 7	34 34	<0.01 <0.01	178 178
UK, 1990	SC	0.75 1.0 3.0	12 12 12	28 28 28	<0.01(2) <0.01(2) <0.01(2)	179 179 179
UK, 1990	SC	0.75 1.0 3.0	13 13 13	17 17 17	<0.01(2) <0.01(2) <0.01(2)	179 179 179
UK, 1990	SC	0.75 1.0 3.0	11 11 11	35 35 35	<0.01(2) <0.01(2) <0.01(2)	179 179 179
UK, 1991	SC	1.5 3.0	13 13	8 8	<0.01(7), 0.01, 0.01(5),0.02(2),0.04	180 180
	WP DG	1.5 3.0 1.5 3.0	13 13 13 13	8 8 8 8	<0.01(3), 0.01 0.01,0.02,0.03,0.04 <0.01(4),0.01(3),0.02 0.01(4),0.02(4)	180 180 180 180
UK, 1991	SC WP DG	1.5 1.5 1.5	11 11 11	20 20 20	<0.01,0.01 <0.01 <0.01,0.01	180 180 180
UK, 1991	SC WP DG	1.5 1.5 1.5	11 11 11	6 6 6	0.01,0.03 0.01 0.02,0.04	180 180 180
USA, California, 1979 USA, Nebraska, 1979	SC SC	1.5 1.5 1.5	3 6 6	0 0 0	0.01,0.02(2),0.07 0.05 0.04	166 166 166
USA, Florida, 1979	SC WP	1.3 1.3	9 9	0 0	0.08,0.01 0.06,0.09(2) 0.12,0.18,0.06	167 167
USA, Florida, 1979	SC WP	1.3 1.3	11 11	0 0	0.02,0.04(3),0.06(2) 0.02,0.04,0.10	167 167
USA, Idaho, 1979	SC	1.3	2	0	0.1,0.0.2(2)	167
USA, New York, 1980	SC WP	1.3 1.2	9 9	0 0	0.01(3) 0.01(2),.0.02	167 167
USA, Maine, 1984	SC	0.7-1.2	7	7	<0.03	168
USA, Washington, 1984	SC	0.7-1.2	11	7	<0.03	168
USA, Ohio, 1985	SC	0.9-1.2	10	6	<0.03	168
USA, Florida, 1984	SC	0.6-1.2	6	7	<0.03	168
USA, Michigan, 1984	SC	0.6-1.2	11	20	<0.03	168
USA, Idaho, 1986	SC	1.3	6	25	<0.03	169
USA, California, 1986	SC	0.9-1.3	7	12	<0.03	169
USA, Oregon, 1986	SC	1.3	11	7	<0.03	169
USA, Colorado, 1986	SC	0.9	7	27	<0.03	169

Results underlined once reflect UK GAP.
Results underlined twice reflect US GAP.

Sugar beet (Table 19). GAP for sugar beet was reported for Greece. Supervised trials were carried out in 1990 in France and Italy. In four trials the treatment regime reflected the maximum GAP. Residue levels in these trials were <0.01-0.1 mg/kg in roots and 0.33-14 mg/kg in leaves.

Table 19. Residues of chlorothalonil in sugar beet from supervised trials in France and Italy. All treatments were with SC formulations.

Location/ year	Application kg ai/ha	No.	PHI, days	Residue(mg/kg) roots	leaves	Reference
France, 1990	1.5	2	64 65	<0.01,0.02 <0.01,0.09	0.1,8.2 2.2,8.4	181
Italy, 1990	1.0 1.5`	3 3	16 16	<0.01 <u><0.01,0.02</u>	1.4 <u>9,8,4.7</u>	182 182
Italy, 1990	1.5	2	0 7 14 21 28	0.05 0.07 <u>0.02</u> <u>0.05</u> 0.05	39 22 <u>14</u> <u>8.3</u> 6.4	184
Italy, 1990	1.5	2	0 7 14 21 28	0.05 0.03 <u>0.05</u> <u>0.16</u> 0.03	34 23 <u>14</u> <u>5.4</u> 4.6	184
Italy, 1990	1.0 1.5	3 3	18 18	0.02 <u>0.07,0.10</u>	0.33 <u>0.33,5.6</u>	185 185
Italy, 1990	0.6	2	14 21	0.01 <0.01	1.1 0.41	186
Italy, 1990	0.6	2	14 21	<0.01 <0.01	0.30 0.11	187

Underlined results reflect GAP in Greece.

Celery (Table 20). Products containing chlorothalonil are registered for use on celery in Australia, Canada, the USA and several European countries. Maximum treatment rates are in the range 1-2.5 kg ai/ha and minimum PHIs are 1-28 days.

Supervised trials have been carried out in the USA, France and the UK. The US GAP of 2.5 kg ai/ha with a PHI of 7 days was used in a number of trials. Residue levels in samples treated in this way were 0.03-9.8 mg/kg.

Table 20. Residues of chlorothalonil in celery from supervised trials in France, the UK and the USA. Products used were SC formulations.

Location/ year	Application kg ai/ha	No.	PHI, days	Residue (mg/kg)	Ref.
France, 1981	1.5	4	0 7 14 21	8.6 6.5 6.2 2.7	192
France, 1980	1.5	4	0 14 21 28	36 25 10 5.3	193
France, 1981	1.1	4	0 7 14 21	7.0 5.5 4.5 4.5	194
UK, 1980	1.5	1	7 14	0.34 0.41	191
	3.0	1	7 14	<u>0.23</u> <u>1.1</u>	191
USA, Michigan, 1980	2.5	8	0 7	6.9, 4.2, 2.4 <u>3.2,1.4,2.5</u>	188

Location/ year	Application kg ai/ha	No.	PHI, days	Residue (mg/kg)	Ref.
France, 1981	1.5	4	0 7 14 21	8.6 6.5 6.2 2.7	192
USA, California, 1980	2.5	7	7	0.03,0.04(2) 1.3(2), 1.1	188
USA, California, 1986	2.5*	10	7	4.3	189
USA, Michigan, 1986	2.5	15	1	9.8	190
USA, Florida, 1987	2.5	16	7	2.9	190

Underlined results reflect US GAP.

Barley (Table 21). GAP for barley has been reported for several European countries. Maximum application rates are 1.0-1.4 kg ai/ha and minimum PHIs are 42 days or expressed as the latest time of treatment to be complete at ear emergence or earlier.

Supervised trials have been carried out in Germany and the UK. Residue levels in trials where treatment was within 50 days of harvest using rates and numbers of applications which are GAP in at least one country were <0.01-1.4 mg/kg in grain and 0.36-8.1 mg/kg in straw.

Table 21. Residues of chlorothalonil in barley grain and straw from supervised trials in Germany and the UK.

Location, year	Form	Application		PHI, days	Residue (mg/kg)		Ref
		kg ai/ha	No.		grain	straw	
Germany, 1983	SC	1.0	1	42	<0.01	5.2	208
Germany, 1983	SC	1.0	2	42	ND*	1.7	209
Germany, 1983	SC	1.0	2	53	ND*	2.9	210
Germany, 1983	SC	1.0	2	59	ND*	0.83	211
Germany, 1983	SC	1.0	2	30	0.02	1.0	213
UK, 1988	SC	0.75	1	74	<0.01	<0.01	219
UK, 1988	SC	0.75	1	53	<0.01	0.02	220
UK, 1981	SC	1.1	2	93	<0.01	1.0	221
			1	122	<0.01(2)	0.70,0.16	221
UK, 1981	SC	1.1	2 1	67 119	<0.01 <0.01(2)	0.05 <0.01,0.03	222
UK, 1981	SC	1.1	2 1	61 118	<0.01 <0.01(2)	0.13 <0.01(2)	223
UK, 1988	SC	0.75	2 1 1	56 56 93	<0.01 <0.01 <0.01	0.05 0.04 0.02	224
UK, 1988	SC	0.75	2 1 1	75 75 104	<0.01 <0.01 <0.01	0.15 0.09 0.13	225
UK, 1988	SC	0.75	2 1 1	71 71 104	<0.01 <0.01 <0.01	0.02 0.02 <0.01	226

Location, year	Form	Application		PHI, days	Residue (mg/kg) grain	straw	Ref
		kg ai/ha	No.				
UK, 1988	SC	0.75	1	91	<0.01	0.04	227
UK, 1990	SC	0.9 1.8	2 2	50 50	0.05 0.05	4.1 11	228
	SC	0.75 1.5	2 2	50 50	0.02 0.05	2.1 8.1	228
	SC	0.9 1.8	2 2	49 49	0.03 0.08	0.36 0.94	228
	SC	0.75 1.5	2 2	49 49	0.03 0.49	0.97 4.9	228
	SC	0.9 1.8	2 2	49 49	0.04 0.29	2.4 6.2	228
	SC	0.75 1.5	2 2	49 49	0.08 1.4	3.7 16	228

*limit of determination not reported
Results underlined once reflect use at 1.4 kg ai/ha, 49-day PHI.
Results underlined twice reflect use at 1.0 kg ai/ha, 49-day PHI.

Wheat (Table 22). GAP for wheat has been reported for Canada (a proposed use) and several European countries. Maximum application rates are 0.5-1.5 kg ai/ha and minimum PHIs are 15-42 days. In some countries the latest time of treatment is specified as ear emergence complete.

Supervised trials have been carried out in a number of European countries. Residue levels in samples where treatment was within 49 days of harvest using rates and numbers of applications which are GAP in at least one country were <0.01-0.09 mg/kg in grain and 0.09-3.1 mg/kg in straw. Average residues in these data sets (one result, the mean, taken for each location, n = 20) were 0.01 mg/kg in grain and 0.8 mg/kg in straw.

Table 22. Residues of chlorothalonil in wheat grain and straw from supervised trials in Denmark, France, Germany and the UK.

Location/ year	Form	Application kg ai/ha	No.	PHI, days	Residue (mg/kg) grain	straw	Ref.
Denmark, 1980	SC SC SC SC	1.4 1.4 0.75 0.75	1 1 1 1	35 54 35 54	<0.01 <0.01 <0.01 <0.01	0.09 0.44 0.11 0.17	195 195 195 195
Denmark, 1980	SC SC SC SC	1.4 1.4 0.75 0.75	1 1 1 1	35 54 35 54	<0.01 <0.01 <0.01 <0.01	0.23 0.53 0.10 0.12	196 196 196 196
Denmark, 1980	SC SC SC SC	1.4 1.4 0.75 0.75	1 1 1 1	35 54 35 54	<0.01 <0.01 <0.01 <0.01	0.11 0.18 0.18 0.16	197 197 197 197
France, 1990	SC	1.1	2	57	<0.01(4)	0.40,0.41, 0.26,0.37	198

Location/ year	Form	Application kg ai/ha	No.	PHI, days	Residue (mg/kg) grain	straw	Ref.
	DG	1.1	2	57	<0.01(2), 0.01(2)	0.21,0.37	198
France, 1990	SC	1.1	2	81	<0.01(4)	0.75,0.57, 0.49,0.63	199
	DG	1.1	2	81	<0.01, 0.01	0.97,0.53	199
France, 1990	SC	1.1	2	43	<0.01(2) 0.01(2)	0.55,1.2, 1.0,1.8	200
	DG	1.1	2	43	<0.01(2)	0.62,1.1	
France, 1990	SC	1.1	2	62	<0.01(4)	0.32,0.40, 0.66,1.2	201
	DG	1.1	2	62	<0.01(2)	0.27,0.44	
France, 1990	SC	1.1	1	57	<0.01(2)	0.18,0.75	202
France, 1990	SC	1.1	1	58	<0.01(2)	0.19,0.07	203
France, 1990	SC	1.1	1	55	<0.01(2)	1.0,1.2	204
	DG	1.1	1	55	<0.01	0.70	
France, 1991	SC DG	1.1 1.1	2 2	45 45	<0.01(2) <0.01(2)	0.54,0.94 0.74,0.70	205
France, 1991	SC DG	1.1 1.1	2 2	46 46	<0.01(2) <0.01(2)	1.4,1.8 1.6,1.9	206
France, 1991	SC	1.1	1	81	<0.01(8)	0.09,0.16, 0.30,0.31, 0.43,0.45, 0.73,1.39	207
	DG	1.1	1	81	<0.01(8)	0.12, 0.13(2), 0.14,0.19, 0.43,0.49, 0.61	207
Germany, 1982	SC	1.1	1	48	<0.01	0.29	216
Germany, 1982	SC	1.1	1	35	<0.01	0.18	217
Netherlands, 1980	SC	1.0	1	68	<0.01	0.38	218
UK, 1988	SC	0.75	1	86	<0.02	0.13	229
UK, 1988	SC	0.75	1	79	<0.01	0.04	230
UK, 1988	SC	0.75	1	68	<0.01	0.02	231
UK, 1989	SC	1.0	4	49	<0.01	1.7	232
UK, 1989	SC	1.0	4	59	<0.01	2.4	233
UK, 1989	SC	1.0	4	43	<0.01	0.27	234
UK, 1989	SC	0.5+1.0	2	49	<0.01	0.33	235
		0.5+1.0	2	63	<0.01	0.11	235
		0.5+1.0	2	82	<0.01	0.19	235
UK, 1989	SC	0.5+1.0 0.5+1.0 0.5+1.0	2 2 2	59 73 89	<0.01 <0.01 <0.01	0.14 0.15 0.05	236 236 236
UK, 1989	SC	0.5+1.0 0.5+1.0 0.5+1.0	2 2 2	43 57 76	<0.01 <0.01 <0.01	0.15 0.09 0.05	237 237 237
UK, 1989	SC	1.0	1	61	<0.01(2)	0.19,0.17	238
UK, 1989	SC	1.0	1	43	<0.01(2)	0.14,0.13	239
UK, 1988	SC	1.0	1	50	<0.01	0.29	240
UK, 1988	SC	0.9	1	73	<0.01	0.02	241
UK, 1988	SC	0.9	1	86	<0.01	0.05	242
UK, 1988	SC	0.9	1	79	<0.01	0.02	243

Location/ year	Form	Application kg ai/ha	No.	PHI, days	Residue (mg/kg) grain	straw	Ref.
UK, 1990	SC	0.5+1.0	2	41	0.02	0.61	244
		0.5+1.0	2	70	0.02	1.2	244
	SC	1.0+2.0	2	41	0.05	3.7	244
		1.0+2.0	2	70	0.03	8.3	244
UK, 1990	SC	0.5+1.0	2	41	<0.01	0.58	244
				60	<0.01	0.92	244
		1.0+2.0	2	41	0.04	7.9	244
				60	0.02	9.2	244
UK, 1990	SC	0.5+1.0	2	45	<0.01	0.74	244
				74	<0.01	1.2	244
		1.0+2.0	2	45	0.04	7.3	244
				74	<0.01	5.5	244
UK, 1990	SC	0.5+1.0	2	41	<0.01	2.3	245
				70	<0.01	1.2	245
		1.0+2.0	2	41	0.04	12	245
				70	0.05	20	245
UK, 1990	SC	0.5+1.0	2	41	<0.01	0.70	245
				60	<0.01	0.13	245
	SC	1.0+2.0	2	41	0.02	5.9	245
				60	0.05	8.9	245
	SC	0.5+1.0	2	45	0.01	1.4	245
				74	<0.01	0.09	245
	SC	1.0+2.0	2	45	0.03	5.1	245
				74	0.03	7.3	245
UK, 1990	DG	1.3	1	84	<0.01(2)	2.1,2.2	246
		3.0	1	84	0.04, 0.08	3.2,7.0	246
UK, 1990	DG	1.3	1	77	<0.01(2)	1.9,1.6	246
		3.0	1	77	<0.01(2)	3.7,2.2	246
UK, 1990	DG	1.3	1	77	<0.01(2)	1.3,0.58	246
		3.0	1	77	0.06,0.07	4.4,9.9	246
UK, 1990	SC	0.6	1	83	<0.01	0.46	247
		1.2	1	83	0.02	4.7	247
		0.75	1	83	<0.01	0.06	247
		1.5	1	83	<0.01	0.15	247
UK, 1990	SC	0.6	1	74	0.02	0.05	247
		1.2	1	74	<0.01	0.98	247
		0.75	1	74	<0.01	0.24	247
		1.5	1	74	<0.01	1.5	247
UK, 1990	SC	0.6	1	88	<0.01	0.62	247
		1.2	1	88	<0.01	1.2	247
		0.75	1	88	<0.01	0.22	247
		1.5	1	88	<0.01	0.41	247
UK, 1991	SC	0.5+1.0	2	37	0.04,0.05	1.5, 1.3	248
		1.0+2.0	2	37	0.16,0.14	7.1,8.5	248
	DG	0.5+1.0	2	37	0.07,0.09	1.5(2)	248
		1.0 +2.0			0.19,0.24	6.8,7.9	248
UK, 1991	SC	0.5+1.0	2	37	<0.01(2)	1.3,3.1	248
		1.0+2.0	2	37	0.02,0.03	10,12	248
	DG	0.5+1.0	2	37	<0.01,0.01	0.9, 1.3	248
		1.0+2.0	2	37	0.02(2)	6.0,5.7	248
UK, 1991	SC	0.5+1.0	2	37	0.01,0.02	2.2,0.8	248
		1.0+2.0	2	37	0.10,0.18	15,10	248
	DG	0.5+1.0	2	37	0.01,0.03	1.1,1.4	248
		1.0+2.0	2	37	0.08,0.15	6.5,5.5	248

Underlined results reflect use up to 1.5 kg ai/ha, PHI 35-49 days.

Oats and rye (Table 23). A single trial on oats and two trials on rye were reported from Germany. Residues in the grain were ≤0.02 mg/kg and in the straw 0.04-0.41 mg/kg.

Table 23. Residues of chlorothalonil in oat and rye grain and

straw from supervised trials carried out in Germany in 1983 with an SC formulation.

Crop	Application kg ai/ha	No.	PHI, days	Residue (mg/kg) grain	straw	Ref.
Oats	1.0	2	51	0.02	0.04	212
Rye	1.0	2	76	ND*	0.12	214
Rye	1.0	2	69	ND*	0.41	215

*Limit of determination not reported

Peanuts (Table 24). In supervised trials carried out in the USA and using treatment regimes within the maximum USA GAP residues were <0.01-0.03 mg/kg in nut-meat and <0.01-0.18 mg/kg in hulls.

Table 24. Residues of chlorothalonil in peanuts from supervised trials carried out in the USA using SC formulations.

Location, year	Application kg ai/ha	No.	PHI, days	Residue (mg/kg) nut-meat	hull	Ref.
Florida, 1986	1.3	7	12	<0.01	0.02	249
Texas, 1986	1.3	6	13	0.01	0.06	249
Alabama, 1986	1.3	8	17	<0.01	0.08	249
Alabama, 1986	1.3	8	17	<0.01	0.10	249
Texas, 1986	1.3	6	13	<0.01	0.04	249
Texas, 1986	1.3	6	13	<0.01	0.07	249
Texas, 1986	1.3	7	17	<0.01	0.08	249
Georgia, 1986	1.3	7	22	0.03	0.18	249
Virgina, 1986	1.3	7	27	<0.01	0.11	249
S Carolina, 1986	1.3	8	32	<0.01	0.03	249
N Carolina, 1986	1.3	5	35	<0.01	0.09	249
Oklahoma, 1986	1.3	6	43	<0.01	<0.01	249

Underlined results reflect use within US GAP.

FATE OF RESIDUES

In plants

The metabolism of chlorothalonil has been investigated in lettuce, tomato, carrot and celery.

Lettuce. Lettuce plants growing in an environmental chamber were treated four times with [^{14}C]chlorothalonil at a rate equivalent to 1.8 kg ai/ha (Nelsen *et al.*, 1985). Concentrations of radioactivity in samples harvested 1-21 days after the last treatment were 100-200 mg chlorothalonil equivalent/kg. Nearly all (>80%) of the radioactivity was

associated with chlorothalonil. The rest was characterized as 4-hydroxy-2,5,6-trichloroisophthalonitrile (SDS-3701, DAC-3701) (up to 2%) or was associated with uncharacterized water-soluble (up to 7%) or unextracted (up to 5%) material.

Tomato. Applications of [^{14}C]chlorothalonil at a rate equivalent to 2.3 kg ai/ha to tomato plants growing in an environmental chamber resulted 1, 7 and 14 days later in total residues in fruit of 2.6, 0.6 and 0.6 mg chlorothalonil equivalent/kg respectively (Nelsen and Duane, 1988). The major component of the residue was chlorothalonil which accounted for 76% of the residue at day 1 and 58% at day 14. The metabolite SDS-3701 was found in fruit, contributing 2-5% of the total residue. The balance of radioactivity was associated with polar water-soluble material (up to 32%) which appeared to contain predominantly conjugated derivatives of chlorothalonil, as well as small amounts (up to 5%) of unextractable material.

Carrot. Carrot plants were treated three times at weekly intervals at 1.6 kg ai/ha (Nelsen *et al.*, 1987). Samples of roots taken 1,7, 14 and 21 days after the last treatment contained total radioactive residues equivalent to 0.07, 0.02, 0.01 and 0.03 mg parent/kg. The residue in the 21-day sample was made up as follows: 40% chlorothalonil, 3% SDS-3701, 15% uncharacterized organosoluble, 20% uncharacterized water-soluble and 30% unextracted material.

Celery. Total radioactive residues in celery stalks harvested 7 and 21 days after the last of 12 applications at rates equivalent to 2.4 kg ai/ha were 1.0-4.6 and 0.7-1.4 mg/kg respectively (Huhtanen, 1992). Chlorothalonil was a significant component (10-56%, average 43%) of the total residue in all stalk samples. SDS-3701 and 3-carboxy-2,5,6-trichlorobenzamide (SDS-46851, DAC-46851) were not found. The balance of radioactivity was associated with a large number of minor components which could not be characterized by comparison with standard materials and did not generate characterizable materials on acid or enzyme hydrolysis.

In storage and processing

A number of studies have been conducted to investigate the fate of chlorothalonil residues during storage and handling or processing of crops prior to consumption.

In several of these studies levels of SDS-3701, SDS-46851, hexachlorobenzene (HCB) and pentachlorobenzonitrile (PCBN) were also measured in processing fractions. Levels of these metabolites and of formulation impurities were low in all samples.

Cherry (Anon, undated). Levels of chlorothalonil in treated cherries (6 x 1.8 kg ai/ha, 7-day PHI) and washed and processed cherries were as follows.

	Residue	
	mg/kg	% of initial
whole unwashed	2.7	100
whole washed	0.52	19
pitted washed	0.38	14
canned (with water)	0.03	1
canned (with syrup)	0.03	1

Chlorothalonil was found in the waste liquid from the pitter (2.6 mg/kg), in cherry stones (0.06 mg/kg) and in wash water (0.03-0.27 mg/kg). No SDS-3701 (<0.01 mg/kg) was found in any sample.

Peach (Anon, undated). Levels of chlorothalonil in treated peaches (6 x 0.9 kg ai/ha, 7-day PHI) and their processed products were as follows:

	Residue	
	mg/kg	% of initial
whole unwashed	13	100
whole washed (water)	5.9	45
whole peeled (caustic)	0.21	2
canned peach puree	<0.01	-

Chlorothalonil was not found (<0.01 mg/kg) in waste pulp. SDS-3701 was not found (<0.01 mg/kg) in waste pulp or peach puree but was found (0.13 mg/kg) in whole peeled peaches.

Grapes (Table 5). In trials carried out in 1987 in France residues were not found (<0.0025 mg/kg) in wine prepared from grapes containing low residues (0.02 mg/kg) of chlorothalonil.

Cabbage (King *et al.*, 1986) Residue levels in treated cabbages were determined at 3 points in the distribution chain: at the farm gate, on leaving the packing house and at retail outlets. Samples were taken and traced from four locations through 3 packing houses to a total of 37 grocery stores.

Residue levels at retail outlets were 8.5% of those present at harvest.

Cucumber. The effect of commercial processing on residue levels in treated cucumbers (2 x 6.54 l/ha Bravo 500, 12-hour PHI) has been investigated (King and Ballee, 1987). Chlorothalonil concentrations were 1.3 mg/kg in unwashed cucumbers, 0.71 mg/kg after washing and 0.52 mg/kg following additional rinsing. After slicing and soaking in brine for 1 hour the residue level was 0.38 mg/kg. After a boiling vinegar/water/sugar solution was added and pickle slices were allowed to cool the residue level was 0.11 mg/kg. After canning, including heating at 210^{o}F for 10 minutes, residue levels in hot canned pickle slices were 0.01 mg/kg. Thus 98% of the residue was lost during commercial processing.

In a separate study (Marks, 1987), treated cucumbers (3 or

4 x 1.8 kg ai/ha, 0- or 1-day PHI) were sampled at 3 points in the distribution chain: at the farm gate (4 sites), at the exits from packing houses (4 houses) and at retail outlets (a total of 40 stores or restaurants/delicatessens). The ranges of residue levels found were 0.02-0.79, <0.01-0.05 and <0.01-0.04 mg/kg at the respective locations. Residue levels decreased to 8-14% of initial levels at the packing houses, where samples were washed, brushed and waxed.

Squash (King and Prince, 1990a). Levels of chlorothalonil in treated squash (11 x 2.6 kg ai/ha, 0-day PHI) and sequential fractions of processed squash were as follows.

	Residue	
	mg/kg	% of initial
whole unwashed	3.2	100
peeled, deseeded	<0.01	-
milled	<0.01	-
partially cooked	<0.01	-
baby food	<0.01	-

Chlorothalonil was not found (<0.01 mg/kg) in finisher waste and 0.15 mg/kg was found in squash waste. SDS-3701 was not found except at very low levels in whole unwashed squash (0.02 mg/kg) and finisher waste (0.01 mg/kg). SDS 46851 was not found except for low levels in whole unwashed fruit (0.06 mg/kg), peeled squash (0.04 mg/kg) and squash waste (0.03 mg/kg). HCB and PCBN were only found in waste (0.003 or 0.005 mg/kg and 0.008 mg/kg, respectively).

Tomato. Levels of chlorothalonil have been measured in treated tomatoes (5, 7 or 9 x 1.8 or 1.6 kg ai/ha 4,7 or 12-day PHI) and processed tomatoes (Anon, undated). Two processing procedures were used; both simulated commercial practice. In one the peel was removed and in the other it was retained. Results were as follows.

	Residue, mg/kg	
Peel removed		
whole unwashed	i) 3.2	ii) 1.8
whole washed	0.2	0.02
whole washed and rinsed	0.2	-
pomace	0.02	0.03
juice	<0.01	<0.01
Peel retained		
whole unwashed	4.0	
pomace	0.62	
canned tomatoes	<0.01	
canned tomato paste	<0.01	
canned tomato juice	<0.01	

In a second study tomatoes were treated 7 times at 2.5 kg ai/ha or double that rate (Szalkowski *et al.*, 1980). Samples (400 lb from each plot) were harvested one day after the last treatment. Tomatoes were processed using a commercial method

which does not remove skins during the early stages of processing. Fruits were power-washed with cold water, passed through a disintegrator and heated to 140-150◦F before passing through a cyclone separator with 0.093 and 0.060 inch finisher screens in place. Juice was hot-filled at 150◦F and air-cooled. Paste (36-40% solids) was prepared by vacuum distillation at 140-150◦F and hot-filled at 150◦F. Chlorothalonil concentrations in processing fractions (mg/kg) were as follows:

	2.5 kg/ha		5 kg/ha
whole unwashed	2.5		4.7
whole washed	0.65		1.2
pomace	2.2	3.8	
juice	0.02		0.78
paste	<0.01		0.02

Chlorothalonil was found in wash water (0.4 or 0.2 mg/kg) but not in condensate (<0.0003 mg/kg). Levels of SDS-3701 were low in all fractions.

In another study (Dillon, 1986a) treated tomatoes were sampled at 3 points in the distribution chain: in the field, at the packing house and at the point of retail sale. Crops had been treated 2-15 times at 0.2-2.3 kg ai/ha and were harvested one day after the last treatment. Four crop locations, four packing houses and 40 retail outlets were investigated. Fruit were washed, dried and waxed in the packing house. Residue levels were 0.12-2.9 mg/kg in field samples, <0.01-0.07mg/kg in packing house samples and <0.01-0.03 mg/kg in retail samples. The residue loss at the packing house was 98%.

Snap beans (Ballee *et al.*, 1980). Treated samples (no treatment details given) were mechanically harvested, air-cleaned, then washed with water, blanched and canned or washed twice, sliced and blanched for freezing. Chlorothalonil concentrations (mg/kg) in the processed beans were as follows.

Canning		Freezing	
at harvest	0.84	at harvest	0.78
after air cleaning	0.54	after first wash	0.16
washed	<0.01	after second wash	0.09
blanched	<0.01	after slicing	0.10
canned	<0.01	after blanching	<0.01

Concentrations of chlorothalonil in canning waste were 29 mg/kg in field trash, 5.2 mg/kg in air cleaner trash, 0.02 mg/kg in solid waste, 0.05 mg/l in wash water and 0.05 mg/l in water discharged from the plant. In freezing waste they were 0.11 mg/kg in bean waste, 0.005 mg/l in first wash water, 1 mg/l in second wash water and 0.004 mg/l in water discharged from the plant.

Carrots (King and Prince, 1990b). Carrot crops were treated 11 times at 2.3 or 23 kg ai/ha and harvested on the day of the last treatment. Root samples (400 lbs) were peeled, cooked, and pureed, then canned. Concentrations of chlorothalonil (mg/kg) were as follows.

	Lower rate	Higher rate
whole, unwashed	0.04	2.2
whole, peeled	<0.01	<0.01
pureed	<0.01	<0.01
cooked	<0.01	<0.01
canned baby food	<0.01	<0.01

Potatoes (Dillon *et al.*, 1986). Potato crops were treated 8 times at 0.6 or 1.2 kg ai/ha. Vines were killed 14 days after the last treatment. Eight days later crops were treated at 0, 2, 8 or 16 pints/acre. Residues of chlorothalonil were not found (<0.01 mg/kg) except in samples treated at the highest rate. Potato samples were washed and peeled, then either sliced and crisped or diced, cooked, dehydrated to granular potato and/or powdered. Residue levels (mg chlorothalonil/kg) were as follows.

whole, unwashed	0.01		
peeled, washed	<0.01		
crisps, sliced	<0.01	dried diced	<0.01
crisps	<0.01	cooked	<0.01
		dried, granular	<0.01
		dried, powdered	<0.01

Chlorothalonil levels were 2.8 mg/kg in the wash water and 0.04 mg/kg in the peel.

Celery (Dillon, 1986b). Samples of treated celery were taken for analysis at 3 points in the distribution chain: in the field, at the packing house and at the point of retail sale (grocery or restaurant). Crops had been treated 2-11 times at 1.1-2.4 kg ai/ha and were harvested 7 or 8 days after the last treatment. Four treatment locations, 4 packing houses and 40 retail outlets were included. Concentrations of chlorothalonil were 0.12-7.3 mg/kg in the field, 0.06-6.5 mg/kg at the packing house, 0.06-1.6 mg/kg at grocery stores and <0.01-0.82 mg/kg at restaurants. Samples taken at restaurants were sliced or diced using normal procedures. The loss of residues was 49% in the packing house and end users received 25% of field residues in grocery stores and 3% in restaurants.

Peanuts (Kenyon and Ballee, 1987). Plants were either treated 11 times at 1.2 kg ai/ha and combined 16 days after the last application or 13 times at 1.2 kg ai/ha and combined 6 days after the last treatment. Residues were found only when the latter regime was used. Samples (40 lbs) were shelled and pressed; crude oil was refined. Residue levels (mg/kg) were as

follows:

field sample, nut-meat	0.01
nut-meat before processing	0.02
crude oil (after pressing)	<0.01
crude oil (solvent extracted from presscake)	0.01
refined oil	<0.01

Chlorothalonil was not found (<0.01 mg/kg) in the presscake following solvent extraction or in soapstock but was present in hulls (0.19-0.40 mg/kg) and trash (0.42 mg/kg).

Stability of pesticide residues in stored analytical samples

Cherries (King *et al.*, 1990a). Residues of chlorothalonil in sour cherries were stable during freezer storage for one year; two samples were taken for analysis after 0, 1, 7, 29, 85, 194, 271 and 362 days storage. Samples for storage were harvested two hours after the last of 10 treatments at 3.4 kg ai/ha and had field-incurred residues of 10 mg/kg.

Cucumbers (Wiedmann and Ballee, 1990). Samples were taken one hour after the last of four treatments at 5 or 7.5 kg ai/ha and had field-incurred residues of 1 mg chlorothalonil/kg. Residues of chlorothalonil were stable during freezer storage for one year; four samples were taken for analysis after 0, 1, 7, 28, 91, 182, 276 and 360 days storage.

Tomatoes (Kenyon *et al.*, 1990a). Residues of chlorothalonil in tomatoes were stable during freezer storage for one year; four samples were taken for analysis after 0, 1, 7, 30, 92, 174, 274 and 363 days storage. Samples for storage were harvested 1½ hours after the last of nine treatments at 2.5 or 7.5 kg ai/ha and had field-incurred residues of 10 mg chlorothalonil/kg.

Carrots (Rose *et al.*, 1990a). Samples were taken five hours after the last of 11 treatments at 17 kg ai/ha and had field-incurred residues of 1.5 mg chlorothalonil/kg. Residues of chlorothalonil were stable during freezer storage for one year; four samples were taken for analysis after 0, 1, 6, 33, 90, 180, 270 and 363 days storage.

Potatoes (Rose *et al.*, 1990b). Residues of chlorothalonil in potatoes were stable during freezer storage for one year; four samples were taken for analysis after 0, 1, 7, 30, 90, 180, 270 and 363 days storage. Samples for storage were harvested on the day of the last of 15 applications at 12 kg ai/ha and had field-incurred residues of 2 mg chlorothalonil/kg.

Celery (King *et al.*, 1990b). Samples were taken one hour after the last of 16 treatments at 2.5 kg ai/ha and had field-incurred residues of 5 mg chlorothalonil/kg. Residues of chlorothalonil were stable during freezer storage for one year; four samples were taken for analysis after 0, 1, 7, 28,

91, 181, 280 and 364 days storage.

Wheat grain (Kenyon *et al.*, 1990b). Residues of chlorothalonil in wheat grain were stable during freezer storage for one year; four samples were taken for analysis after 0, 1, 7, 30, 91, 179, 273 and 362 days storage. Samples for storage were harvested on the day of the last of seven treatments at 12 kg ai/ha and had field-incurred residues of 40 mg chlorothalonil/kg.

Peanuts (King *et al.*, 1991). Samples of nut-meat were taken on the day of the last of 11 treatments at 10-12 kg ai/ha and had field-incurred residues of 13 mg/kg. Four samples were taken for analysis after 0, 1, 7, 28, 80, 171, 266, 300, 328, 363, 425, 485, 544, 601, 663 and 726 days storage. There was a 22% per year decline in chlorothalonil residues. This was not, however, a clear linear decline, rather a 'shift' at 266 days preceded and followed by apparent stability.

APPRAISAL

Chlorothalonil was first evaluated in 1974. This evaluation has been prepared as part of the programme of periodic reviews agreed by the CCPR.

Information on current GAP and residue trials data were made available to the Meeting by one of the manufacturers; GAP information was also provided by Australia, Canada and the EC.

At the initiation of this review there were 35 MRLs for chlorothalonil; all were CXLs except the MRL for grapes which was at step 7B.

The fate of chlorothalonil has been studied in lettuce, tomato, carrot and celery. Chlorothalonil was the major characterised component of the residue in all cases; small amounts of 4-hydroxy-2, 5, 6-trichloroisophthalonitrile (SDS-3701) were also found.

Data from supervised residue trials carried out in a number of countries and on a range of crops were available.

No GAP was reported for citrus fruit, so the Meeting recommended that the CXL of 5mg/kg should be withdrawn.

The CXL of 10 mg/kg for cherries was proposed in 1974; it was based on US GAP with a 7-day pre-harvest interval and residue data from trials carried out in the USA. Since use so close to harvest is no longer GAP in the USA the CXL is obsolete and the Meeting considered that it should be withdrawn. Results from a series of trials carried out in accordance with current GAP in the USA were available at the Meeting. Residues up to 0.5 mg/kg were found. The Meeting recommended that an MRL of 0.5 mg/kg was appropriate for this use.

For peaches, the CXL of 25 mg/kg was again based on US GAP permitting use up to 7 days before harvest and residue trials data from the USA. This US GAP is now obsolete and therefore the CXL was not acceptable. Supervised trial data on peaches from Italy, Spain and the USA were made available to the Meeting. Residues up to 0.12 mg/kg were found when chlorothalonil was used according to current US GAP, and up to 0.98 mg/kg in Italian trials within Spanish and Greek (1.5kg ai/ha and 14-15 days PHI) and Italian (1.0 kg ai/ha and 21-day PHI) GAP. The Meeting recommended an MRL of 1 mg/kg for peaches.

Chlorothalonil residues up to 4.1 mg/kg were found in cranberries harvested 50-70 days after treatment at 5.9 kg ai/ha (within US GAP) in a series of trials in the USA in the 1980s. The Meeting confirmed the MRL of 5 mg/kg for cranberry.

The CXLs of 25 mg/kg for raspberries (red and black) and currants (black, red and white) and 10 mg/kg for blackberries were based on GAP and trials in the USA. Since this GAP is no longer current the Meeting recommended that these CXLs should be withdrawn.

For grapes, the draft MRL, at step 7B, is 10 mg/kg. This proposal was based on Austrian GAP of 0.11 kg ai/ha with a PHI of 7 days and on data from supervised trials carried out in Germany; this GAP is no longer current. A 1.6 kg ai/ha, 7-day PHI GAP has been reported for Australia and in one trial in 1973/4 chlorothalonil residues up to 5.6 mg/kg were found in supervised trials after treatment within this GAP. However, GAP in France (0.4 kg ai/ha, 30 days PHI) yielded much more recent data that were consistent and were deemed more suitable as the basis for a recommendation. The Meeting therefore recommended an MRL of 0.5 mg/kg, based on the data from France.

For banana the GAP on which the CXL of 0.2 mg/kg was based is not clearly described in the 1973 evaluations. The data base considered by the present Meeting was not sufficient to support a soundly based MRL and the Meeting recommended that the CXL should be withdrawn.

The CXL of 5 mg/kg for bulb onions was based on trials data for green onions; the Meeting therefore concluded that it needed revision. Chlorothalonil residues up to 0.57 mg/kg were found in bulb onions harvested 7 days after treatment at 1.5 - 1.75 kg ai/ha (within US GAP) and up to 0.52 mg/kg 14 days after treatment at 1.5 kg ai/ha (within other countries' GAP), although most results were lower than these. The Meeting recommended an MRL of 0.5 mg/kg.

The CXL of 5 mg/kg for cabbages was based on residue data from US trials where crops were harvested on the day of the last treatment. Since current US GAP specifies a minimum PHI of 7 days the CXL should be revised. Chlorothalonil residues

up to 0.7 mg/kg were reported from trials using treatment regimes within US, UK and Irish GAP. The Meeting recommended an MRL of 1 mg/kg.

For broccoli, the CXL of 5 mg/kg is based on a 7-day PHI and results from US trials. This GAP is still current in the USA and Canada but although results were reported from two further US trials where treatments were within GAP, the Meeting considered the data were inadequate and recommended that the CXL of 5 mg/kg should be withdrawn.

The CXL of 5 mg/kg for Brussels sprouts was based on a PHI of 7 days and data from the USA. Chlorothalonil residues up to 4.3 mg/kg were reported for samples harvested 6-7 days after treatment at 1.3-2.5 kg ai/ha. The Meeting recommended that the CXL should be maintained.

For cauliflower, the CXL of 5 mg/kg was based on a PHI of 7 days and residue data from the USA. Chlorothalonil residues up to 0.47 mg/kg were reported from trials where treatments were within current GAP in the USA, UK and Ireland. The Meeting recommended an MRL of 1 mg/kg.

For kale, the CXL of 10 mg/kg was based on US GAP and residue data. Since this GAP is no longer current the Meeting recommended withdrawal of the CXL.

The CXL of 5 mg/kg for melons except watermelon was based on US trials data and a 1-day PHI. Chlorothalonil residues up to 1.45 mg/kg were found in samples treated in accordance with US GAP. The Meeting recommended an MRL of 2 mg/kg but recognised that additional data on residues on different types of melons would be desirable.

For cucumbers, the CXL of 5 mg/kg is based on a 1-day PHI. Chlorothalonil residues up to 4.3 mg/kg were reported from trials where treatments were in accordance with US GAP and the Meeting recommended that the CXL should be maintained.

The CXLs of 5 mg/kg for summer and winter squash and pumpkins were based on a 1-day PHI. Chlorothalonil residues up to 3.6 mg/kg were found in samples of summer and winter squash treated in accordance with current US GAP. The Meeting recommended that MRLs of 5 mg/kg were appropriate for summer and winter squash. No residue data were presented for pumpkins and therefore that CXL should be withdrawn, although pumpkins appear to be covered in the Codex Classification by the MRL for winter squash.

For sweet corn, the CXL of 1 mg/kg was based on a 1-day PHI which is no longer GAP. Residue data reflecting current US GAP were available from only one trial; these were not sufficient to estimate a maximum residue level. The Meeting recommended withdrawal of the CXL.

The CXL for tomato is 5 mg/kg, based on US data and GAP.

Chlorothalonil residues up to 4.6 mg/kg were found in trials where treatments were within GAP. The Meeting recommended that the CXL should be maintained.

The CXL of 10 mg/kg for peppers was based on US GAP and residue data. Since use on peppers is no longer GAP in the USA the Meeting recommended withdrawal of this recommendation.

The CXLs for endive, lettuce and witloof chicory (sprouts) were based on US GAP and residue data. Since use on these crops is no longer GAP in the USA the Meeting recommended withdrawal of these CXLs.

The CXL of 5 mg/kg for common bean (pods and/or immature seeds) was based on US GAP and residue data. In supervised trials residue levels in crops treated in accordance with GAP were up to 3.1 mg/kg. The Meeting recommended that the CXL should be maintained.

The CXL for lima beans (dry) was based on US GAP and residue data. Since this use is no longer GAP in the USA the Meeting recommended withdrawal of the CXL.

The CXL of 1 mg/kg for carrots was based on GAP and residue data from the USA. Residues up to 0.96 mg/kg were reported from trials where treatments were within GAP. The Meeting recommended that the CXL should be maintained.

The CXL of 0.1 mg/kg for potato was based on a 0-day PHI. Residues up to 0.18 mg/kg were reported from trials where treatments were within GAP although only one result exceeded 0.1 mg/kg. The Meeting recommended an MRL of 0.2 mg/kg.

The CXL of 1 mg/kg for sugar beet was based on a 1-day PHI; this is no longer GAP. Residues reflecting current GAP were up to 0.1 mg/kg in the root. The Meeting recommended an MRL of 0.2 mg/kg for sugar beet root. Corresponding residues in the leaves reached 14 mg/kg. The Meeting recommended an MRL of 20 mg/kg for sugar beet leaves or tops but realised that appropriate animal transfer studies were lacking.

The CXL of 15 mg/kg for celery was based on a 7-day PHI. Chlorothalonil residues up to 9.8 mg/kg were found in trials where treatments reflected current GAP. The Meeting recommended an MRL of 10 mg/kg.

Barley grain from crops treated in accordance with GAP contained up to 1.4 mg chlorothalonil/kg. Most results however were much lower than this. The Meeting decided that the data reflecting use up to 1.4 kg ai/ha were not sufficient to support a soundly based MRL and recommended an MRL of 0.1 mg/kg for grain, based on application rates up to 1.0 kg ai/ha. The Meeting also recommended an MRL of 20 mg/kg for barley straw; animal transfer studies are desirable.

Wheat grain from crops treated in accordance with GAP

contained up to 0.09 mg chlorothalonil/kg. The Meeting recommended that the MRL should be established at 0.1 mg/kg for grain and 20 mg/kg for wheat straw, recognising that animal transfer studies were desirable.

GAP was not reported for any other cereal grain. The Meeting recommended that the CXL for cereal grains should be withdrawn.

The CXLs for whole peanut and peanut kernels were based on a 1-day PHI; this is no longer GAP. Chlorothalonil residues up to 0.03 mg/kg were found in crops treated in accordance with current GAP. The Meeting recommended an MRL of 0.05 mg/kg for peanut and withdrawal of the CXL for whole peanuts.

Information on residue distribution between the inedible and edible portions of the commodity was available for banana; chlorothalonil is essentially a surface residue and transfer to pulp was insignificant.

Processing studies are available for cherry, peach, grape, cabbage, cucumber, squash, tomato, snap bean, carrot, potato, celery and peanut.

Washing cherries, peaches, cucumbers, tomatoes and snap beans removed 45-95% of the residue. Residue reductions of 75-98% occurred in cabbages, cucumbers, tomatoes and celery during distribution from the farm gate to retail outlets. Residue levels in canned cherries, canned pickled cucumber and tomato juice made from treated crops were very low (1-2% of initial residues). Residues were not found in canned peach puree, wine, squash-based baby food, tomato paste, canned or frozen snap beans, carrot-based baby food, potato crisps, dried potato or refined peanut oil prepared from crops with incurred residues.

Chlorothalonil residues were stable during freezer storage for one year in cherries, cucumbers, tomatoes, carrots, potatoes, celery and wheat grain.

RECOMMENDATIONS

On the basis of the data on residues from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits.

Definition of the residue: chlorothalonil

Commodity		Recommended MRL (mg/kg)		PHI on which based, days
CCN	Name	New	Previous	
FI 0327	Banana	W	0.2	-
GC 0640	Barley	0.1	0.2 (cereals)	49

Commodity		Recommended MRL (mg/kg)		PHI on which based, days
CCN	Name	New	Previous	
AS 0640	Barley straw and fodder, dry	20	-	49
FB 0264	Blackberries	W	10	-
VB 0400	Broccoli	W	5	7
VB 0402	Brussels sprouts	5	5	7
VB 0041	Cabbages, Head	1	5	7
VR 0577	Carrot	1	1	0
VB 0404	Cauliflower	1	5	7
VS 0404	Celery	10	15	7
GC 0080	Cereal grains	W	0.2	-
FS 0013	Cherries	0.5	10	*
FC 0001	Citrus fruits	W	5	-
VP 0526	Common bean (pods and/or immature seeds)	5	5	7
FB 9265	Cranberry	5	5	50
VC 0424	Cucumber	5	5	0
FB 0021	Currants (Black, Red, White)	W	25	-
VL 0476	Endive	W	10	-
FB 0269	Grapes	0.5	10	30
VL 0480	Kale	W	10	-
VL 0482	Lettuce, Head	W	10	-
VD 0534	Lima bean (dry)	W	0.5	-
VC 0046	Melons, except watermelon	2	5	0
VA 0385	Onion, Bulb	0.5	5	14
FS 0247	Peach	1	25	14-21
SO 0703	Peanut, whole	W	0.5	-
SD 0697	Peanut	0.05	0.1	14
VO 00051	Peppers	W	10	-
VR 0589	Potato	0.2	0.1	0
VC 0429	Pumpkins	W	5	-
FB 0272	Raspberries (Red, Black)	W	10	-
VC 0431	Squash, summer	5	5	0
VR 0596	Sugar beet	0.2	1	14
AV 0596	Sugar beet leaves or tops	20	-	14
VO 0447	Sweet corn	W	1	
VO 0448	Tomato	5	5	0
GC 0654	Wheat	0.1	0.2 (cereals)	41-45
AS 0654	Wheat straw and fodder, dry	20	-	41-45
VC 0433	Winter squash	5	5	0
VS 0469	Witloof chicory (sprouts)	W	10	-

* last use at shuck (cot) fall.

FURTHER WORK OR INFORMATION

Desirable

1. Additional residue data from supervised trials on different types of melons.

2. Animal transfer studies assuming a residue equivalent to the recommended MRL of 20 mg/kg in sugar beet leaves or tops, barley straw and wheat straw.

3. Additional residue data on grapes treated according to GAP in Australia.

REFERENCES

References to supervised trials are cited by number in the Tables. References to the fate of residues are cited by authors' names in the text.

Reference List 1 (numerical) gives complete references to all the citations in the monograph. List 2 (alphabetical) comprises the references to the fate of residues. Each reference gives only the author(s), the year, and the number in List 1 under which the complete reference will be found.

All references are unpublished.

1. Numerical list

1. Stallard, D.E., Marks, A.F. and Ignatoski, J.A. 1982. Residues of chlorothalonil, SDS-3701, HCB and PCBN on cherries - early season application of Bravo 500. ISK Biotech. Document Number: 532-3CR-81-0204-00.

2. Nelsen, T.R., Ballee, D.L., Marks, A.F. and Ignatoski, J.A. 1982. Residues of chlorothalonil, SDS-3701, HCB and PCBN on sweet cherries - early season application, 1982. ISK Biotech. Document Number: 604-3CR-82-0166-002.

3. Rose, C.A. and Ballee, D.L., 1987. Residues of chlorothalonil, SDS-3701, SDS-46851, HCB and PCBN on mechanically harvested tart cherries - 1984. ISK Biotech. Document Number; 1382-86-0075-CR-001.

4. Szalkowski, M.B., Ballee, D.L., Stallard, D.E. and Ignatoski, J.A. 1980. Residues of chlorothalonil and SDS 3701 on peaches. ISK Biotech. Document Number: 126-3CR-80-0107-001.

5. Ballee, D.E., Stallard, D.E. and Ignatoski, J.A. 1980. Residues of chlorothalonil, SDS-3701, HCB and PCBN on peaches. ISK Biotech. Document Number: 126-3CR-80-0173-001.

6. Stallard, D.E., Marks, A.F. and Ignatoski, J.A. 1982. Residues of chlorothalonil, SDS-3701, HCB and PCBN on peaches. ISK Biotech. Document Number: 532-3CR-80-0173-005.

7. Dillon, K.A., Ballee, D.L., Marks A.F. and Ignatoski, J.A. Residues of chlorothalonil, SDS-3701, HCB and PCBN on peaches following aerial application. ISK Biotech. Document Number: 656-3CR-083-0124-001.

8. Kenyon R.G. and Ballee, D.E. Residues of chlorothalonil, SDS-3701, SDS-46851 HCB and PCBN on peaches. ISK Biotech. Document Number: 1441-86-0097-CR-001.

9. Macgregor, D.C. 1989. Determination of residues of chlorothalonil, SDS-3701, SDS-46851, HCB and PCBN in crops: peaches and nectarines. ISK Biotech. Document Number: HLA-6012-241B.

10. Nangniot, P. 1990a. Residues of chlorothalonil and carbendazim on peaches. ISK Biotech. Document Number: 90/457.

11. Nangniot, P. 1990b. Residues of chlorothalonil and carbendazim on peaches. ISK Biotech. Document Number: 90/458.

12. Anon. 1991a. Residues of chlorothalonil on peaches. ISK Biotech. Document Number: CRP/91/585A.

13. Anon. 1991b. Residues of chlorothalonil on peaches. ISK Biotech. Document Number: CRP/91/604A.

14. Anon. 1991c. Residues of chlorothalonil on peaches. ISK Biotech. Document Number: CRP/91/605A.

15. Nangniot, P. 1990c. Residues of chlorothalonil on peaches. ISK Biotech. Document Number: 90/444.

16. Nangniot, P. 1990d. Residues of chlorothalonil on peaches. ISK Biotech. Document Number 90/445.

17. Nangniot, P. 1990e. Residues of chlorothalonil on peaches. ISK Biotech. Document Number 90/470.

18. Anon. 1992a. Residues of chlorothalonil on peaches. ISK Biotech. Document Number CRP/92/945A

19. Anon. 1992b. Residues of chlorothalonil on peaches. ISK Biotech. Document Number CRP/92/946A.

20 Dillon, K.A. 1985. Residues of chlorothalonil, SDS-3701, HCB and PCBN on cranberries - 1982, 1983, 1984. ISK Biotech. Document Number 698-3CR-84-0070-001.

21. Macgregor, D.C. 1990. Residues of chlorothalonil, SDS-3701, SDS-46851, HCB and PCBN in crops: cranberries. ISK Biotech. Document Number HCA-6012-2416.

22. Anon. 1992. Residues of chlorothalonil in grapes. ISK Biotech. Document Number: 92/1208.

23. Anon. 1992. Residues of chlorothalonil in grapes. ISK Biotech. Document Number: 91/0792.

24. Anon. 1992. Residues of chlorothalonil in grapes. ISK Biotech. Document Number: 92/1272.

25. Anon. 1983. Residues of chlorothalonil in grapes. ISK Biotech. Document Number: 83/136.

26. Anon. 1983. Residues of chlorothalonil in grapes. ISK Biotech. Document Number: 83/137.

27 Anon. Undated. Pesticide residues. ISK Biotech. Document Number: 10307.

28. Anon. 1985. Chlorothalonil and folpet on grapes. ISK Biotech. Document Number: 85/178.

29. Anon. 1987. Chlorothalonil and folpet on grapes. ISK Biotech. Document Number: 87/248.

30. Anon. 1988. Decline curve for chlorothalonil on grapes. ISk Biotech. Document Number: 88/288.

31. Anon. 1988. Decline curve for chlorothalonil on grapes. ISK Biotech. Document number: 88/289.

32. Anon 1988. Decline curve for chlorothalonil on grapes. ISk Biotech.

Document number: 88/290.

33 Anon. 1988. Residues of chlorothalonil, DAC 3701 and folpet on grapes. ISK Biotech. Document number: 88/291.

34. Anon. 1988. Residues of chlorothalonil, DAC 3701 and folpet on grapes. ISK
Biotech. Document Number: 88/292.

35 Anon. 1988. Residues of chlorothalonil, DAC 3701 and folpet on grapes. ISK biotech. Document Number: 88/293.

36. Anon. 1988. Residues of chlorothalonil, DAC 3701 and folpet on grapes. ISK Biotech. Document Number: 88/294.

37. Anon. 1988. Residues of chlorothalonil, DAC 3701 and folpet on grapes. ISK Biotech. Document Number 88/295.

38. Anon. 1988. Residues of chlorothalonil, DAC 3701 and folpet on grapes. ISK Biotech. Document Number: 88/296.

39. Anon. 1988. Residues of chlorothalonil, DAC 3701 and folpet on grapes. ISK Biotech. Document Number: 88/297.
40 Anon. 1988. Residues of chlorothalonil, DAC 3701 and folpet on grapes. ISK Biotech. Document Number 88/298.

41. Anon. 1988. Residues of chlorothalonil, DAC 3701 and folpet in grapes, must and wine. Document Number 88/299.

42. Anon. 1988. Residues of chlorothalonil, DAC 3701 and folpet in grapes, must, lees and wine. Document Number: 88/300.

43. Ballee, D.L., Szalkowski, M.B., Stallard, D.E. and Ignatoski, J.A. 1980. A cooperative study for the determination of residues of chlorothalonil and DS-3701 on banana fruit. ISK Biotech. Document Number: 075-3CR-79-0056-001.

44. Kenyon, R.G., Ballee, D.L., Marks, A.F. and Ignatoski, J.A. 1986. Residues of chlorothalonil, SDS-3701, HCB and PCBN on bananas. ISK Biotech. Document Number: 743-3CR-85-0022-001.

45. Anon. Undated and untitled. ISK Biotech. Document Number: 1127/78.

46. King, C. 1986. Residues of chlorothalonil, SDS-3701, SDS-46851, HCB and PCBN on bulb onions - 1985 Texas and California. ISK Biotech. Document Number: 1158-86-0013-CR-001.

47. King, C. and Ballee, D.L. 1987. Residues of chlorothalonil, SDS-3701, SDS-46851, HCB and PCBN on dry bulb onions, Michigan and New York 1984-1986. ISK Biotech. Document Number: 1158-86-0013-CR-003.

48. Anon. Undated and untitled. ISK Biotech. Document Number: 297/82.

49. Anon., undated and untitled. ISK Biotech. Document Number: AY/36.

50. Nangniot, P. 1982, Chlorothalonil on onions. ISK Biotech. Document Number: 81/64.

51 Nangniot, P. 1982. Chlorothalonil on onions. ISK Biotech. Document Number: 81/65.

52 Anon. 1991. Residues of chlorothalonil on onions. ISK Biotech. Document Number: CRP/91/609A.

53. Anon. 1991. Residues of chlorothalonil on onions. ISK Biotech. Document Number: CRP/91/610.

54. Anon. Undated and untitled. ISK Biotech. Document Number: 1460/81.

55. Anon. Undated and untitled. ISK Biotech. Document Number: 1462/81.

56. McKenzie, J. 1992. Determination of concentrations of chlorothalonil in onions. Source: UK. ISK Biotech. Document Number: ISK 0391.

57. McKenzie, J. 1992. Determination of concentrations of chlorothalonil in onions. Source: UK. ISK Biotech. Document Number: ISK 0392.

58. 1988, Pesticide Research Report, Agriculture Canada.

59. King, C. and Ballee, D.L. 1987. Residues of chlorothalonil, SDS-3701, SDS-46851, HCB and PCBN on cabbage. ISK Biotech Corporation USA. Document Number: 1423-86-0095-CR-001.

60. Marks, A.F. 1986. Residues of chlorothalonil, SDS-3701, SDS-46851, HCB and PCBN on cabbage. ISK Biotech Corporation USA. Document Number: 1163-86-0017-CR-992.

61. King, C. and Ballee, D.L. 1987. Residues of chlorothalonil, SDS-3701, SDS-46851, HCB and PCBN on cabbage - Florida, 1987. ISK Biotech Corporation, USA. Document Number: 1420-86-0095-CR-002.

62. McKenzie, J. 1991. Determination of concentations of chlorothalonil in cabbages. Source: UK. ISK Biotech. Document Number ISK 0791.

63. McKenzie, J. 1992. Determination of concentrations of chlorothalonil in cabbage. Source: UK ISK Biotech. Document Number ISK 0792.

64. Higgins, W.P., Dillon, K.A., Ballee, D.L. and Marks, A.F. 1986. Residues of chlorothalonil degradaiton products and formulation impurities on 1986 broccoli samples from Soledad, California. ISK Biotech. Document Number: 1165-86-0053-CR-001.

65. Higgins, W.P., Dillon, K.A. and Ballee, D.L. 1987. Residues of chlorothalonil, SDS-3701, SDS-46851, HCB and PCBN on broccoli - 1985. ISK Biotech, Document Number: 1165-86-0027-CR-003.

66. Higgins, W.P., Dillon, K.A. and Ballee, D.L. 1987. Residues of chlorothalonil, SDS-3701, SDS-46851, HCB and PCBN on broccoli samples from Michigan and New York ,1985 and 1986. ISK Biotech. Document Number: 1165-86-0053-CR-002.

67. King, C. and Ballee, D.L. 1988. Residues of chlorothalonil, SDS-3701, SDS-46851, HCB and PCBN on broccoli 1987. ISK Biotech. Document Number: 1715-87-0122-CR-001.

68. King, C. and Ballee, D.L. 1987. Residues of chlorothalonil, SDS-3701, SDS-46851, HCB and PCBN on Brussels sprouts - 1986. ISK Biotech. Document Number: 1509-87-0022-CR-001.

69. King, C. and Ballee, D.L. 1988. Residues of chlorothalonil, SDS-3701, SDS-46851, HCB and PCBN on Brussels sprouts-1987. ISK Biotech. Document Number: 1716-88-0003-CR-001.

70. Anon. Undated and untitled. ISK Biotech. Doocument Number: 103/77

71. Anon. Undated and untitled. ISK Biotech. Doocument Number: 90/375

72. Anon. Undated and untitled. ISK Biotech. Doocument Number: 90/374

73. Anon. Undated and untitled. ISK Biotech. Doocument Number: 90/373

74. Anon. Undated and untitled. ISK Biotech. Doocument Number: 90/408

75. Anon. Undated and untitled. ISK Biotech. Doocument Number: 90/407

76. Anon. Undated and untitled. ISK Biotech. Doocument Number: 90/406

77. McKenzie, J. 1991. Determination of concentrations of chlorothalonil in Brussels sprouts. Source: UK. ISK Biotech. Document Number: ISK 0991

78. Marks, A.F. and Ignatoski, J.A. Determination of residues of chlorothalonil, SDS-3701, SDS-46851, HCB and PCBN on cauliflowers - 1985. ISK Biotech. Document Number: 1162-86-0014-CR-002

79. Kenyon, R.G., Ballee, D.L. and Marks, A.F. 1986. Residues of chlorothalonil, SDS-3701, SDS-46851, HCB and PCBN on cauliflower - 1985. ISK Biotech. Document Number: 1162-86-0014-CR-002-001.

80. McKenzie, J. 1991. Residues of chlorothalonil in cauliflower. Source UK. ISK Biotech. Document Number: ISK 0691.

81. McKenzie, J. 1991. Residues of chlorothalonil in cauliflower. Source UK. ISK Biotech. Document Number: ISK 0692.

82. Kenyon, R.G., Ballee, D.L., Marks, A.F. and Ignatoski, J.A. Residues of chlorothalonil, HCB and PCBN on honeydew melons - 1980. ISK Biotech. Document Number: 336-3CR-80-0235-001.

83. Anon. Undated and untitled. ISK Biotech. Document Number: MEL/F/1

84. Anon. Undated and untitled. ISK Biotech. Document Number: MEL/F/2

85. Anon. Undated and untitled. ISK Biotech. Document Number: MEL/F/3

86. Anon. Undated and untitled. ISK Biotech. Document Number: MEL/F/4

87. Anon. Undated and untitled. ISK Biotech. Document Number: CRP/91/578A

88. Ballee, D.L., Szalkowski, M.B., Stallard, D.E. and Ignatoski, J.A. 1980. Residues of chlorothalonil, HCB and PCBN on cucumbers following field application of Bravo 500 by sprinkler irrigation. ISK Biotech. Document Number: 336-3CR-80-0181-001.

89. Kenyon, R.G. and Ballee, D.L. 1987. Residues of chlorothalonil, SDS-3701, SDS-46851, HCB and PCBN on cucumbers - 1986. ISK Biotech. Document Number: 1474-87-0001-CR-001.

90. Kenyon, R.G., Ballee, D.L. and Marks, A.F. 1987. Residues of chlorothalonil, SDS-3701, SDS046851, HCB and PCBN on cucumbers - Florida 1987. ISK Biotech. Document Number: 1474:87-0001-CR-002.

91. Fitzgerald, T.J., Kenyon, R.G., Ballee, D.L. and Marks, A.F. Residues of chlorothalonil, SDS-3701, SDS-46851, HCB and PCBN on cucumbers - California, 1987. ISK Biotech. Document Number: 1474-87-0001-CR-003.

92. Anon. 1989. Studies on 'Daconil 500 Flow' on mildew in cucurbits. ISK Biotech. Document Number: 31-1 ONDES

93. Anon. 1989. Chlorothalonil on cucumbers. ISK Biotech. Document Number: 89/355

94. Anon. 1989. Chlorothalonil on cucumbers. ISK Biotech. Document Number: 89/354.

95. Anon. 1989. Chlorothalonil on cucumbers. ISK Biotech. Document Number: 89/353.

96. Anon. 1989. Decline for chlorothalonil on cucumbers. ISK Biotech.

Document Number: 13/10/89

97. Anon. 1991. Residues of chlorothalonil on cucumbers. ISK Biotech. Document Number: CRP/91/514A

98. Anon. 1991. Residues of chlorothalonil on cucumbers. ISK Biotech. Document Number: CRP/515A

99. Anon. 1991. Residues of chlorothalonil on cucumbers. ISK Biotech. Document Number: CRP/92/798A

100. Anon. 1992. Residues of chlorothalonil on cucumbers. ISK Biotech. Document Number: CRP/92/799A

101. Anon. 1992. Residues of chlorothalonil on cucumbers. ISK Biotech. Document Number: CRP/92/800A

102. Anon. 1992. Residues of chlorothalonil on cucumbers. ISK Biotech. Document Number: CRP/92/801A

103. Anon. 1992. Residues of chlorothalonil on cucumbers. ISK Biotech. Document Number: CRP/92/802A

104. Anon. 1992. Residues of chlorothalonil on cucumbers. ISK Biotech. Document Number: CRP/92/803A

105. Anon. 1992. Residues of chlorothalonil on cucumbers. ISK Biotech. Document Number: CRP/92/804A

106. Anon. 1992. Residues of chlorothalonil on cucumbers. ISK Biotech. Document Number: CRP/92/805A

107. Anon. 1991. Residues of chlorothalonil on cucumbers. ISK Biotech. Document Number: CRP/91/53

108. Marks, A.F. 1986. Residues of chlorothalonil, SDS-3701, SDS-46851, HCB and PCBN on summer squash - 1985. Biotech. Document Number: 1160-86-0032-CR-002

109. Marks, A.F. 1986. Residues of chlorothalonil, SDS-3701, SDS-46851, HCB and PCBN on winter squash. ISK Biotech. Document Number: 1161-86-0041-CR-002

110. Kenyon, R.G., Ballee, D.L. and Marks, A.F. 1976. Residues of chlorothalonil, SDS-3701, SDS-46851, HCB and PCBN on sweetcorn - 1985. ISK Biotech. Document Number: 1166-86-0047-CR-001.

111. Kenyon, R.G., Ballee, D.L. and Marks, A.F. 1986. Residues of chlorothalonil, SDS-3701, SDS-46851, HCB and PCBN on 'Bravo 500' treated tomatoes. ISK Biotech. Document Number: 1142-85-0063-CR-003.

112. Marks, A.F. and Ignatoski, J.A. 1986. Residues of chlorothalonil, SDS-3701, HCB and PCBN on 'SDS-63049' treated tomatoes. ISK Biotech. Document Number: 740-3CR-85-0062-002

113. Marks, A.F. and Ignatoski, J.A. 1986. Residues of chlorothalonil, SDS-3701, SDS-46851, HCB and PCBN on tomatoes. ISK Biotech. Document Number: 1156-86-0008-CR-002

114. Anon. 1989. Residues of chlorothalonil on tomatoes. ISK Biotech. Document Number: 89/350.

115. Anon. 1989. Residues of chlorothalonil on tomatoes. ISK Biotech. Document Number 89/351

116. Anon. 1989. Residues of chlorothalonil on tomatoes. ISK Biotech.

Document Number 89/352

117 Anon. 1990. Residues of chlorothalonil on tomatoes. ISK Biotech. Document Number 90/380

118 Anon. 1990. Residues of chlorothalonil on tomatoes. ISK Biotech. Document Number 90/381

119 Anon. 1990. Residues of chlorothalonil on tomatoes. ISK Biotech. Document Number 90/382

120 Anon. 1991. Residues of chlorothalonil on tomatoes. ISK Biotech. Document Number CRP/91/506

121 Anon. 1991. Residues of chlorothalonil on tomatoes. ISK Biotech. Document Number CRP/91/507

122 Anon. 1991. Residues of chlorothalonil on tomatoes. ISK Biotech. Document Number CRP/91/549A

123 Anon. 1991. Residues of chlorothalonil on tomatoes. ISK Biotech. Document Number CRP/91/550A

124 Anon. 1991. Residues of chlorothalonil on tomatoes. ISK Biotech. Document Number CRP/91/551A

125 Anon. 1991. Residues of chlorothalonil on tomatoes. ISK Biotech. Document Number CRP/91/552A

126 Anon. 1991. Residues of chlorothalonil on tomatoes. ISK Biotech. Document Number CRP/91/553A

127 Anon. 1991. Residues of chlorothalonil on tomatoes. ISK Biotech. Document number CRP/91/556A

128 Anon. 1991. Residues of chlorothalonil on tomatoes. ISK Biotech. Document Number CRP/91/558

129 Anon. 1991. Residues of chlorothalonil on tomatoes. ISK Biotech. Document Number CRP/91/559

130 Anon. 1991. Residues of chlorothalonil on tomatoes. ISK Biotech. Document Number CRP/91/560

131 Anon. 1991. Residues of chlorothalonil on tomatoes. ISK Biotech. Document Number CRP/91/561

132 Anon. 1991. Residues of chlorothalonil on tomatoes. ISK Biotech. Document Number CRP/91/562

133 Anon. 1991. Residues of chlorothalonil on tomatoes. ISK Biotech. Document Number CRP/91/563

134 Anon. 1991. Residues of chlorothalonil on tomatoes in greenhouses. ISK Biotech. Document Number CRP/91/574A

135 Anon. 1991. Residues of chlorothalonil on tomatoes in greenhouses. ISK Biotech. Document Number CRP/91/575A

136 Anon. 1991. Residues of chlorothalonil on tomatoes. ISK Biotech. Document Number CRP/91/653A.

137. Anon. 1992. Residues of chlorothalonil on tomatoes. ISK Biotech. Document Number CRP/91/655A

138 Anon. 1992. Residues of chlorothalonil on tomatoes. ISK Biotech.

Document Number CRP/91/656A.

139. Nangniot, P. 1990. Untitled. ISK Biotech. Document Number: 90/446

140. Anon. 1991. Residues of chlorothalonil on tomatoes grown under protection. ISK Biotech. Document Number: CRP/92/504A.

141. Anon. 1991. Residues of chlorothalonil on tomatoes grown under protection. ISK Biotech. Document Number: CRP/92/505.

142. Anon. 1991. Residues of chlorothalonil on tomatoes grown under protection. ISK Biotech. Document Number: CRP/91/516

143. Anon. 1991. Residues of chlorothalonil on tomatoes. ISK Biotech. Document Number: CRP/91/511.

144. King, C., Ballee, D.L. and Marks, A.F. 1986. Residues of chlorothalonil, SDS0-3701, SDS-46851, HCB and PCBN on snapbeans. ISK Biotech. Document Reference: 1321-86-0046-CR-001.

145. King, C., Ballee, D.L. and Marks, A.F. 1986. Residues of chlorothalonil, SDS0-3701, SDS-46851, HCB and PCBN on snapbeans. ISK Biotech. Document Reference: 1321-86-0046-CR-002.

146. Nangniot, P. 1989. Residues of chlorothalonil on beans. ISK Biotech. Document Number: 89/360.

147. Nangniot, P. 1989. Residues of chlorothalonil on beans. ISK Biotech. Document Number: 89/361.

148. Nangniot, P. 1990. Residues of chlorothalonil on beans. ISK Biotech. Document Number: 90/393.

149 Nangniot, P. 1990. Residues of chlorothalonil on beans. ISK Biotech. Document Number: 90/394

150 Nangniot, P. 1990. Residues of chlorothalonil on beans. ISK Biotech. Document Number: 90/395.

151. Anon. 1991. Residues of chlorothalonil on beans. ISK Biotech. Document Number: CRP/91/516A.

152. Anon. 1991. Residues of chlorothalonil on beans. ISK Biotech. Document Number: CRP/91/517A.

153. Nangniot, P. 1991. Residues of chlorothalonil and carbendazim on beans harvested dry. ISK Biotech. Document Number: 86/214

154. Nangniot P. 1986. Residues of chlorothalonil on beans. ISK Biotech. Document Number: 89/229

155. Nangniot P. 1986. Residues of chlorothalonil on beans. ISK Biotech. Document Number: 89/230

156. Nangniot P. 1989. Residues of chlorothalonil on beans. ISK Biotech. Document Number: 89/347

157. Nangniot P. 1989. Residues of chlorothalonil on beans. ISK Biotech. Document Number: 89/348

158. Nangniot P. 1990. Residues of chlorothalonil on beans. ISK Biotech. Document Number: 89/392

159. McKenzie, J. 1991, Determination of concentraiton of chlorothalonil in field beans and broad beans. Source UK ISK Biotech. Document Number: ISK 0591.

160. McKenzie, J. 1991. Determination of concentrations of chlorothalonil in field beans. Source: UK. ISK Biotech. Document Number: ISK 0592

161. Ballee, D.L., Stallard, D.E. and Ignatoski, J.A. 1981. Residues of chlorothalonil, HCB and PCBN on carrots resulting from application of formulated chorothalonil by sprinkler irrigation. ISK Biotech. Document Reference: 336-3CR-81-0156-001.

162. Kenyon, R.G., Ballee, D.L., Marks, A.F. and Ignatoski, J.A. 1985. Residues of chlorothalonil, HCB and PCBN on carrots - 1979-80. ISK Biotech. Document Reference : 334-3CR-81-0172-001.

163. Dillon, K.A., Ballee, D.L. and Marks, A.F. 1986. Residues of chlorothalonil, degradation products and formulation impurities on 1986 carrot samples from Soledad, California. ISK Biotech. Document Reference: 1319-86-0044-CR-001.

164. Higgins, W.P., Dillon, K.A. and Ballee, D.L. 1987. Residues of chlorothalonil, SDS-3701, SDS-46851, HCB and PCBN on 1986 carrot samples from Texas, Washington and Wisconsin. ISK Biotech. Document Reference: 1319-86-0044-CR-002.

165. Anon. Undated and untitled. ISK Biotech. Document Reference: 692/74.

166. Ballee, D.L., Stallard, D.E. and Ignatoski, J.A. 1981. Residues of chlorothalonil, HCB and PCBN on potatoes following field applications of 'Bravo 500' by sprinkler irrigation or by aerial application. ISK Biotech. Document Reference: 336-3CR-81-0044-001.

167. Ballee, D.L., Stallard, D.E. and Ignatoski, J.A. 1981. Residues of chlorothalonil, HCB and PCBN on potatoes. ISK Biotech. Document Reference: 334-3CR-81-0043-001.

168. Kenyon, R.G., Ballee, D.L. and Marks, A.F. 1986. Residues of chlorothalonil, SDS3701, SDS-46851, HCB and PCBN on potatoes. ISK Biotech. Document Reference:1144-85-0061-CR-003.

169. Fitzgerald, T.J., Kenyon, R.G. and Ballee, D.L. 1987. Residues of chlorothalonil, SDS-3701, SDS-46851, HCB and PCBN on potatoes. ISK Biotech. Document Reference: 1331-86-0052-CR-001.

170. Nangniot, P. 1981. Chlorothalonil in potatoes. ISK Biotech. Document Number 81/015.

171. Nangniot, P. 1981. Chlorothalonil in potatoes. ISK Biotech. Document Number 81/017.

172. Nangniot, P. 1987. Chlorothalonil in potatoes. ISK Biotech. Document Number 81/258.

173. Anon. 1981. Chlorothalonil in potatoes. ISK Biotech. Document Number CRP/91/576.

174. Anon. 1981. Chlorothalonil in potatoes. ISK Biotech. Document Number CRP/91/577.

175. Nangniot, P. 1989. Chlorothalonil in potatoes. ISK Biotech. Document Number 89/356.

176. Nangniot, P. 1989. Chlorothalonil in potatoes. ISK Biotech. Document Number 89/357

177. Nangniot, P. 1989. Chlorothalonil in potatoes. ISK Biotech. Document Number 89/358.

178. Nangniot, P. 1989. Chlorothalonil in potatoes. ISK Biotech. Document Number 89/359.

179. McKenzie, J. 1991. Determination of concentraitons of chlorothalonil in potatoes. Source UK. ISK Biotech. Document Number: ISK 0291.

180. McKenzie, J. 1991. Determination of concentraitons of chlorothalonil in potatoes. Source UK. ISK Biotech. Document Number: ISK 0292.

181. Anon. 1991. Residues of chlorothalonil in sugar beet. ISK Biotech. Document Number: CRP/91/534.

182. Anon. 1991. Residues of chlorothalonil in sugar beet. ISK Biotech. Document Number: CRP/91/535.

183. Anon. 1991. Residues of chlorothalonil in sugar beet. ISK Biotech. Document Number: CRP/91/536.

184. Anon. 1991. Residues of chlorothalonil in sugar beet. ISK Biotech. Document Number: CRP/91/537.

185. Anon. 1991. Residues of chlorothalonil in sugar beet. ISK Biotech. Document Number: CRP/91/538.

186. Anon. 1991. Residues of chlorothalonil in sugar beet. ISK Biotech. Document Number: CRP/91/588.

187. Anon. 1991. Residues of chlorothalonil in sugar beet. ISK Biotech. Document number: CRP/91/589.

188. Ballee, D.L., Stallard, D.E. and Ignatoski, J.A. 1981. Residues of chlorothalonil, HCB and PCBN on celery resulting from application of formulated chlorothalonil by sprinkler irrigation. ISK Biotech. Document Number: 336-3CR-81-0083-001.

189. Kenyon R.G., Ballee, D.L. and Marks, A.F. 1986. Residues of chlorothalonil, SDS-3701, SDS-4851, HCB and PCBN on celery - California - 1987. ISK Biotech. Document Number: 1320-86-0045-CR-001.

190. Fitzgerald, T.J., Kenyon, R.G. and Ballee, D.L. 1987. Residues of chlorothalonil, SDS-3701. SDS-46851, HCB and PCBN on celery -1986-7. ISK Biotech. Document Number: 1320-86-0045-CR-002.

191. Anon. Undated and untitled. ISK Biotech. Document Number: 80/310.

192. Anon. 1981. Chlorothalonil on celery. ISK Biotech. Document Number: 81/57

193. Nangniot, P. 1980. Chlorothalonil on celery. ISK Biotech. Document Number: 0/13

194. Nangniot, P. 1981. Chlorothalonil and carbendazim on celery. ISK Biotech. Document number: 81/58.

195. Nangniot, P. 1980. Chlorothalonil on cereals. ISK Biotech. Document Number: 008

196. Nangniot, P. 1980. Chlorothalonil on cereals. ISK Biotech. Document Number 009.

197. Nangniot, P. 1980. Chlorothalonil on cereals. ISK Biotech. Document Number 010.

198. Anon. 1991. Residues of chlorothalonil on wheat. ISK Biotech. Document Number: CRP/91/593.

199. Anon. 1991. Residues of chlorothalonil on wheat. ISK Biotech. Document Number: CRP/91/594.

200. Anon. 1991. Residues of chlorothalonil on wheat. ISK Biotech. Document Number: CRP/91/595.

201. Anon. 1991. Residues of chlorothalonil on wheat. ISK Biotech. Document Number: CRP/91/596.

202. Anon. 1991. Residues of chlorothalonil on wheat. ISK Biotech. Document Number: CRP/91/597.

203. Anon. 1991. Residues of chlorothalonil on wheat. ISK Biotech. Document Number: CRP/91/598.

204. Anon. 1991. Residues of chlorothalonil on wheat. ISK Biotech. Document Number: CRP/91/599.

205. Anon. 1991. Residues of chlorothalonil on wheat. ISK Biotech. Document Number: CRP/92/922

206. Anon. 1991. Residues of chlorothalonil on wheat. ISK Biotech. Document umber: CRP/92/923.
.
207. Anon. 1991. Residues of chlorothalonil on wheat. ISK Biotech. Document Number: CRP/92/924.

208. Anon. 1983. Untitled. ISK Biotech. Document Number: 3334

209. Anon. 1984. Untitled. ISK Biotech. Document Number: 4346.

210. Anon. 1983. Untitled. ISK Biotech. Document Number: 2883

211. Anon. 1984. Untitled. ISK Biotech. Document Number: 4345

212. Anon. 1983. Untitled. ISK Biotech. Document Number: 3377

213. Anon. 1983. Untitled. ISK Biotech. Document Number: 2983

214. Anon. 1983. Untitled. ISK Biotech. Document Number: 4347

215. Anon. 1983. Untitled. ISK Biotech. Document Number:4344

216. Anon. 1983. Residues of chlorothalonil on spring wheat, ISK Biotech. Document Number: 83/119

217. Anon. 1983. Residues of chlorothalonil on winter wheat, ISK Biotech. Document Number: 83/120.

218. Anon. 1981. Chlorothalonil on cereals. ISK Biotech Document Number: 81/034.

219. Nangniot, P. 1989. Residues of chlorothalonil and carbendazim on spring barley. ISK Biotech. Document Number: 89/340

220. Nangniot, P. 1989. Residues of chlorothalonil and carbendazim on spring barley. ISK Biotech. Document Number: 89/339

221. Nangniot, P. 1982. Chlorothalonil and carbendazim on barley. ISK Biotech. Document Number: 82/72

222. Nangniot, P. 1982. Chlorothalonil and carbendazim on barley. ISK Biotech. Document Number; 82/73

223. Nangniot, P. 1982. Chlorothalonil and carbendazim on barley. ISK Biotech. Document Number; 82/74

224. Nangniot, P. 1989. Residues of chlorothalonil and carbendazim on winter barley. ISK Biotech. Document Number: 89/335.

225. Nangniot, P. 1989. Residues of chlorothalonil and carbendazim on winter barley. ISK Biotech. Document Number: 89/336.

226. Nangniot, P. 1989. Residues of chlorothalonil and carbendazim on winter barley. ISK Biotech. Document Number: 80/337.

227. Nangniot, P. 1989. Residues of chlorothalonil and carbendazim on winter barley. ISK Biotech. Document Number: 89/338.

228. McKenzie, J. 1991. Determination of concentrations of chlorolonil in winter barley, grain and straw. Source UK. ISK Biotech. Document Number: ISK 0191/4

229. Nangniot, P. 1989. Residues of chlorothalonil and carbendazim on winter wheat. ISK Biotech. Document Number: 89/333

230 Nangniot, P. 1989. Residue of chlorothalonil and carbendazim on winter wheat. ISK Biotech. Document Number 89/333.

231. Nangniot, P. 1989. Residues of chlorothalonil and carbendazim on winter wheat. ISK Biotech. Document Number: 89/332

232. Nangniot, P. 1990. Residues of chlorothalonil on winter wheat. ISK Biotech. Document Number: 90/391.

233. Nangniot, P. 1990. Residues of chlorothalonil on winter wheat. ISK Biotech. Document Number: 90/390.

234. Nangniot, P. 1990. Residues of chlorothalonil on winter wheat. ISK Biotech. Document Number: 90/389.

235. Nangniot, P. 1990. Residues of chlorothalonil on winter wheat. ISK Biotech. Document Number: 90/388.

236. Nangniot, P. 1990. Residues of chlorothalonil on winter wheat. ISK Biotech. Document Number: 90/387.

237. Nangniot, P. 1990. Residues of chlorothalonil on winter wheat. ISK Biotech. Document Number: 90/386.

238. Nangniot, P. 1990. Residues of chlorothalonil on winter wheat. ISK Biotech. Document Number: 90/385.

239. Nangniot, P. 1990. Residues of chlorothalonil on winter wheat. ISK Biotech. Document Number: 90/384.

240. Nangniot, P. 1990. Residues of chlorothalonil on winter wheat. ISK Biotech. Document Number: 90/383.

241. Nangniot, P. 1989. Residues of chlorothalonil on winter wheat, ISK Biotech. Document Number: 89/343

242. Nangniot, P. 1989. Residues of chlorothalonil on winter wheat, ISK Biotech. Document Number: 89/342

243. Nangniot, P. 1989. Residues of chlorothalonil on winter wheat, ISK Biotech. Document Number: 89/341

244. Brown, P.C. 1991. Determination of concentrations of chlorothalonil in winter wheat grain and straw. Source UK. ISK Biotech. Document Number: ISK 0191/1.

245. Brown, P.C. 1991. Determination of concentrations of chlorothalonil in winter wheat grain and straw. Source UK. ISK Biotech. Document Number: ISK 0191/2.

246. Brown, P.C. 1991. Determination of concentrations of chlorothalonil in winter wheat grain and straw. Source UK. ISK Biotech. Document Number: ISK 0191/3.

247. McKenzie, J. 1991. Determination of concentrations of chlorothalonil in winter wheat grain and straw. Source UK. ISK Biotech. Document Number: ISK 0191/5.

248. McKenzie, J. 1992. Determination of concentrations of chlorothalonil in winter wheat, grain and straw. Source UK. ISK Biotech. Document Number: ISK 0192/1

249. King, C. and Ballee, D.L. 1986. Residues of chlorothalonil, SDS-3701, SDS-46851, HCB and PCBN on peanut nut-meats and hulls- 1986. ISK Biotech. Document Number: 1424-86-0094-CR-001.

250. Nelsen, T.R., Marks, A.F. and Ignatoski, J.A. 1985. A plant metabolism study with 14C-chlorothalonil on lettuce. ISK Biotech. Document Number: 672-3EF-84-0014-001.

251. Nelsen, T.R. and Duane, W.C. 1988. A plant metabolism study with 14C-chlorothalonil on tomatoes. ISK Biotech. Document Number: 1184-85-0052-EF-001.

252. Nelsen, T.R., Marks A.F. and Baxter, R.A. 1987. A plant metabolism study with 14C-chlorothalonil on carrots. ISK Biotech. Document Number: 1186-86-0026-EF-001.

253. Huhtanen, K.L. 1992. A plant metabolism study with 14C-chlorothalonil on celery. ISK Biotech. Document Number: 3503-90-0184-EF-001.

254. Anon. Undated. Fate of chlorothalonil in the processing of cherries. ISK Biotech. Document Number: 000-3CR-77-2108-001.

255. Anon. Undated. Fate of chlorothalonil in the processing of peaches. ISK Biotech Document Number: 000-3CR-77-2109-001

256. King, C, Ballee, D.L., Marks, A.F. and Ignatoski, J.A. 1986. Residues of chlorothalonil on cabbage - dietary exposure study - 1985. ISK Biotech. Document Number: 728-3CR-85-0011-001.

257. King, C. and Ballee, D.L. 1987. Residues of chlorothalonil, SDS-3701, SDS-46851, HCB and PCBN on cucumbers - processing study 1985 and 1986. ISK Biotech. Document Number: 1351-86-0059-CR-001.

258. Marks, A.F. 1987. Determination of residues of chlorothalonil on cucumbers - dietary exposure study. ISK Biotech. Document number: 1137-850-0010-CR-002.

259. King, C. and Prince, P.M. 1990a. Residues of chlorothalonil, SDS-3701, SDS-46851, HCB and PCBN on winter squash-processing study- 1988. ISK Biotech. Document Number: 3185-89-0287-CR-001.

260 Anon. Undated. Fate of chlorothalonil residues in the processing of tomatoes. ISK Biotech. Document Number 000-3CR-77-2110-001

261. Szalkowski, M.B., Ballee D.L., Stallard, P.E. and Ignatoski, J.A. 1980. Effect of commercial processing on the residue of chlorothalonil on tomatoes. ISK Biotech. Document Number: 411-3CR-80-0054-001.

262. Dillon, K.A. 1986a. Residues of chlorothalonil on fresh market tomatoes-dietary exposure study. ISK Biotech. Document Number: 728-3CR-85-

0008-001.

263. Ballee, D.L., Szalkowski, M.B., Stallard, D.E. and Ignatoski, J.A. 1980. Residues of chlorothalonil and SDS-3701 among the products of snapbean processing. ISK Biotech. Document Number: 117-3CR-79-0136-001.

264. King, C. and Prince, P.M. 1990b. Residues of chlorothalonil, SDS-3701, SDS-46851, HCB and PCBN on carrots. ISK Biotech. Document Number: 3186-89-0287-CR-001.

265 Dillon, K.A., Ballee, D.L. and Marks, A.F. 1986. Residues of chlorothalonil, degradation products and formulation impurities on potatoes - processing study - 1985. ISK Biotech. Document Number: 1154-86-0004-CR-001.

266. Dillon K.A. 1986b. Residues of chlorothalonil on celery - dietary exposure study. ISK Biotech. Document Number: 728-3CR-85-0009-001.

267. Kenyon, R.G. and Ballee, D.L. 1987. Residues of chlorothalonil, SDS-3701, SDS-46851, HCB and PCBN on peanuts - processing study. ISK Biotech. Document Number: 1155-86-0006-CR-001.

268. King, C., Wiedmann, J.L. and Ballee, D.L. 1990a. Residues of chlorothalonil, SDS-3701, SDS-46851, HCB and PCBN in cherries from a stability study (field-incurred) - 1988 - one year interim report. ISK Biotech. Document Number: 3064-88-0068-CR-001.

269. Wiedmann, J.L. and Ballee, D.L. 1990. Residues of chlorothalonil, SDS-3701, SDS-46851, HCB and PCBN in cucumbers from a stability study (field-incurred) - 1988 - one year interim report. ISK Biotech. Document Number: 3064-88-0093-CR-001.

270. Kenyon, R. G., Wiedmann, J.L. and Ballee, D.L. 1990a. Residues of chlorothalonil, SDS-3701, SDS-46851, HCB and PCBN in tomatoes from a stability study (field-incurred) - 1988 - one year interim report. ISK Biotech. Document Number: 3064-88-0083-CR-001.

271. Rose, C., Dillon, K.A. and Ballee, D.L. 1990a. Residues of chlorothalonil, SDS-3701, SDS-46851, HCB and PCBN in carrots from a stability study (field-incurred) - 1988 - one year interim report. ISK Biotech. Document Number: 3064-88-0096-CR-001.

272. Rose, C., Dillon, K.A. and Ballee, D.L. 1990b. Residues of chlorothalonil, SDS-3701, SDS-46851, HCB and PCBN in potatoes from a stability study (field-incurred) - 1988 - one year interim report. ISK Biotech. Document Number 3064-88-0095-CR-001.

273. King C., Wiedman, J.L. and Ballee, D.L. 1990b. Residues of chlorothalonil, SDS-3701, SDS-46851, HCB and PCBN in celery from a stability study (field-incurred) - 1988 - one year interim report. ISK Biotech. Document Number: 3064-88-0136-CR-001.

274. Kenyon, R.G., Wiedmann, J.L. and Ballee, D.L. 1990b. Residues of chlorothalonil, SDS-3701, SDS-46851, HCB and PCBN in wheat grain from a stability study (field-incurred) - 1988 - one year interim report. ISK Biotech. Document Number: 3064-88-0070-CR-001.

275. King C., Wiedmann, J.L. and Ballee, D.L. 1991. Residues of chlorothalonil, SDS-3701, SDS-46851, HCB and PCBN in peanuts from a stability study (field-incurred) - 1991 - two year interim report. ISK Biotech. Document Number: 3064-88-0160-CR-001.

2. Alphabetical list

CHLORPYRIFOS-METHYL (090)

EXPLANATION

Chlorpyrifos-methyl was evaluated by the JMPR in 1975 and has been reviewed several times since, most recently in 1991. Information from the producer reviewed in 1991 showed that residues of chlorpyrifos-methyl in crude and refined oil from maize containing 3.8 mg/kg were as as high as approximately 100 mg/kg. For this reason the 1991 Meeting required further information on the influence of commercial refining processes on residues in oil from maize treated with chlorpyrifos-methyl and full details of the processes used. A TMRL for chlorpyrifos-methyl at 10 mg/kg is also under consideration for another crop containing oil, rape seed, and it would be expected that residues in oil produced from post-harvest-treated rape seed would also be very high. Information was therefore required on residues in crude and refined oil from treated rape seed with full details of commercial processes.

The Meeting has received two studies on the fate of chlorpyrifos-methyl in maize oil but no information was available on the presence of residues in rape seed oil.

The Meeting has received supplementary information on approved uses of chlorpyrifos-methyl in Spain and on residues from trials on mandarins, lemons and oranges.

USE PATTERN

Registered uses of chlorpyrifos-methyl in Spain are listed in Table 1.

RESIDUES RESULTING FROM SUPERVISED TRIALS

Residue data were available from trials in Spain on lemons, mandarins and oranges. In 9 trials on lemons with the application concentration of 0.08 kg ai/hl residues after 14 days were 0.06-0.13 mg/kg. In 3 trials on mandarins at the application rate of 1.4 kg ai/ha and 0.09 kg ai/hl residues after 14 days were 0.02-0.05 mg/kg, but the registered use in Spain on citrus fruit is 2.0-3.0 kg ai/ha and 0.08-0.1 kg ai/hl. In 6 trials on oranges at a rate of 2.5-2.9 kg ai/ha residues after 14 days were 0.01-0.05 mg/kg. In all

Table 1. Registered uses of chlorpyrifos-methyl in Spain.

Crop	Application		PHI, days
	kg ai /ha	kg ai/hl	
Citrus fruit	2.0-3.0	0.08-0.1	15
Pome fruit and stone fruit	1.1-1.5	0.08-0.1	15
Grape	0.75-1.0	0.08-0.1	15
Strawberry	1.1-1.5	0.08-0.1	5
Tomato, pepper, aubergine and artichoke	1.1-1.5	0.08-0.1	5
Other vegetables	1.1-1.5	0.08-0.1	15
Maize	0.75-1.0		15
Cotton	0.45-0.60	0.08-0.1	15
Potato	0.3-0.5	0.03-0.05	15

trials on lemons, mandarins and oranges residues were considerably lower than the MRL proposed by the JMPR in 1991 (Table 2).

Table 2. Residues of chlorpyrifos-methyl in citrus fruit from supervised trials in Spain in 1992. Residues are underlined when the application used is in accordance with or approximately in accordance with registered use in Spain.

Crop (Region)	Application		PHI days	Residue, mg/kg			Report
	kg ai /ha	kg ai/hl					
Lemon		0.08	0	2.2	2.2	2.1	No information
			7	0.30	0.26	0.23	
(Alicante)			14	0.10	0.10	0.06	
			21	0.01			
(Alicante)		0.08	0	2.3	2.3	2.3	
			7	0.39	0.37	0.37	
			14	0.12	0.13	0.10	
(Marcia)		0.08	0	2.1	2.1	2.2	
			7	0.26	0.31	0.27	
			14	0.11	0.12	0.09	
Mandarine/clementine	1.44	0.09	0	0.39	0.40	1.1	
			7	0.20	0.20	0.23	
			14	0.05	0.02	0.04	
			21		0.01		
			28	0.02	0.04	0.09	
Orange (Valencia)	2.5	0.09	0	0.64	0.49	0.26	
			7	0.19	0.17	0.17	
			14	0.02	0.03	0.01	
			21		0.02		
			28	0.01			
(Villalonga)	2.9	0.09	0	0.48	0.62	0.70	
			7	0.22	0.24	0.19	
			14	0.05	0.05	0.04	
			21	0.04	0.04	0.03	
			28	0.03	0.04	0.03	

FATE OF RESIDUES

In storage and processing

In response to the requirement for details of commercial processes used in producing and refining maize oil the Meeting has received a paper in which is described the commercial processing of maize generally used in the USA. The oil is produced by either a dry or a wet milling process. Maize oil is produced from dry-milled germ either by direct solvent extraction or by continuous expelling. Wet-milled germ is best processed by a combination of expelling and solvent extraction.

The crude oil is treated with NaOH at 65-75◦C to neutralize free fatty acids, hydrolize phosphatides and remove pigments and unsaponifiable material. After cooling these impurities are removed as soapstock, leaving refined oil. The pigments in the refined oil are next removed by bleaching with activated earth at 120◦ C and filtration.

Deodorization, which removes all oxidative cleavage products, is effected by processing the oil at a vacuum of 1-6 mm Hg at 175-230◦C with 1-5% steam (Petersen, 1986).

The fate of chlorpyrifos-methyl was followed during the production of maize oil from maize grain treated with approximately 9 mg/kg. Half of the grain was processed into fractions using the wet-milling process, and the other half by the dry-milling process. The grain and its fractions were analyzed for chlorpyrifos-methyl and the metabolite 3,5,6-trichloro-2-pyridinol.

The residues in the grain and the fractions are shown in Table 3. Residues of chlorpyrifos-methyl in the refined oil from dry- and wet-processed grain were very high and of the order of 100 mg/kg, which is comparable to the residues reported to the 1991 JMPR. The residues disappeared however during the deodorization process and were <0.02 mg/kg in the oil from both wet and dry processing (McKellar, 1986).

The fate of ^{14}C-labelled chlorpyrifos-methyl residues in refined maize oil during the deodorization process was examined in a laboratory experiment. Refined oil was fortified with the ^{14}C-labelled compound to 84 mg/kg and was subjected to the deodorization process, in which the oil was heated to 220◦C for one hour under vacuum (1-5 mm Hg). More than 95% of the radoactivity distilled out of the oil and was trapped and found to be unchanged chlorpyrifos-methyl. Less than 3% of the radioactivity remained in the oil and was shown to be mainly unchanged chlorpyrifos-methyl (Yackowich, 1986).

Table 3. Residues of chlorpyrifos-methyl in fractions from processed maize.

Substrate	Chlorpyrifos-methyl (mg/kg)	3,5,6-Trichloro-2-pyridinol (mg/kg)
Wet processing		
grain	8.7	--
Steeped maize	0.4	1
Defatted germ	0.2	0.3
Gluten	3	6.7
Gluten/starch	3.5	2.7
Starch	0.1	0.2
Refined oil	86	16
Soapstock	0.05	36
Deodorized oil	<0.02	<0.05
Dry fiber/hull	7.2	2
Steepwater	0.1	0.2
Dry processing		
Whole grain	8.8	--
Defatted germ	0.6	4.5
Meal		1.3
Flour		2.2
Grits		0.6
Hull		14
Refined oil	110	<0.05
Soapstock	0.25	28
Deodorized oil	<0.02	<0.05

APPRAISAL

Data evaluated by the 1991 JMPR showed residues of chlorpyrifos-methyl in crude and refined maize oil as high as approximately 100 mg/kg, produced from maize containing only 3.8 mg/kg. The 1991 JMPR therefore required further information on the influence of commercial refining processes on residues of chlorpyrifos-methyl in oil from maize and a full description of the processes used.

As a maximum residue limit of 10 mg/kg is established for chlorpyrifos-methyl in rape seed, and it would be expected that

residue levels in oil produced from treated rape seed would also be high, information was required on the levels of residues occurring in rape seed oil.

In response to the requirement for information about commercial processes used for producing and refining maize oil a detailed description of the milling, refining and deodorization procedures generally used in the USA was received. Two studies of the fate of residues of chlorpyrifos-methyl during the processes of milling, refining and deodorization were also supplied. The compound is concentrated in the oil produced from maize grain and it does not disappear during the process of refining, but it disappears almost completely when the oil is deodorized. The procedure used for deodorizing is to heat the oil to 175-230° C in a vacuum. In this process chlorpyrifos-methyl is volatilized, and in one of the experiments the vapours were trapped and 95% of the chlorpyrifos-methyl originally present in the oil was collected as the unchanged compound.

No information was available to the Meeting on the levels of chlorpyrifos-methyl in rape seed oil either from trials or from monitoring. The Meeting therefore recommends withdrawal of the existing temporary MRL of 10 mg/kg for chlorpyrifos-methyl in rape seed.

Information was received from Spain on registered uses of chlorpyrifos-methyl and summarized residue data from trials on lemons, mandarins and oranges. The applications in the trials were in accordance with registered uses in Spain, except those on mandarins where the dosage was a little lower in the trials. All residues were low, between 0.01 and 0.13 mg/kg after 14 days, and lower than the proposed limit of 0.5 mg/kg for oranges. The proposed residue limit for oranges was confirmed, but the Meeting was unable to propose a residue limit for the whole group of citrus fruits as details from the trials in Spain were not available.

RECOMMENDATIONS

On the basis of the data on residues from supervised trials the Meeting concluded that the residue level shown below is suitable for establishing a maximum residue limit. The Meeting also recommends withdrawal of the TMRL for rape seed.

Definition of the residue: chlorpyrifos-methyl

Commodity		Recommended MRL (mg/kg)	
CNN	Name	New	Previous
SO 0495	Rape seed	withdrawn	10 Po T

FURTHER WORK OR INFORMATION

Desirable

Submission of details from trials on citrus fruits in Spain and further information on GAP for citrus fruits in Spain.

REFERENCES

McKeller, R.L. 1986. Residues of chlorpyrifos-methyl and 3,5,6-trichloro-2-pyridinol in corn process fractions obtained from corn grain treated with Reldan 4E insecticide. Dow Chemicals GH-C 1825. Unpublished.

Petersen, B.J. 1986. Commercial Processing of Corn Oil. Prepared by Technical Assessment Systems, INC for Dow Chemicals.

Yackowich, P.R. 1986. A laboratory experiment to determine the fate of residues of chlorpyrifos-methyl in refined corn oil upon processing to deodorized corn oil. Dow Chemicals. Unpublished.

CYCLOXYDIM (179)

EXPLANATION

Cycloxydim was reviewed for the first time by the 1992 JMPR but the time available did not allow adequate evaluation of the extensive residue data provided. This monograph addendum therefore deals only with the evaluation of these data.

RESIDUES RESULTING FROM SUPERVISED TRIALS

Supervised trials have been carried out in 15 countries on over 40 commodities. However, many were single trials providing only limited data. MRLs have been established in several countries, ranging from 0.05 to 5 mg/kg according to crop (FAO/WHO, 1993a).

Summaries of data from the trials, which were designed to be according to recommended use patterns, are given in Tables 1 to 4.

Fruits - See Table 1.

Citrus fruits. Trials in South Africa on grapefruit showed no residues above 0.05 mg/kg after 102 days. On lemons, residues of 0.12 and 0.13 mg/kg were found after application at either 0.4 or 0.8 kg ai/ha; however, untreated crops showed a similar level of cycloxydim in these limited trials.

Pome fruits. Following treatment in South Africa at the maximum surface application rate of 0.4 kg ai/ha, residues of <0.05 and 0.15 mg/kg were found in apples and pears respectively. When sprayed on to the fruits instead of using the recommended surface application, residues of 0.08 and 1.3 mg/kg were found on apples; residues in pears remained below 0.05 mg/kg under these conditions.

Stone fruits. In one trial on peaches in South Africa a residue of 0.1 mg/kg was found after 27 days. Trials on plums showed no residue above 0.05 mg/kg. However, residues of 2.5 and 3.1 mg/kg were found on peaches when the spray was applied to the fruits rather than using the recommended surface application.

Berries and other small fruits. On grapes, trials in South Africa yielded residues up to 0.58 mg/kg after 29 days. In France, residues were less than 0.05 mg/kg after 80 to 141 days.

In trials on strawberries in The Netherlands, residues ranged from 0.13 to 0.45 mg/kg (mean 0.33 mg/kg) at 21 days following treatment at 0.2 kg ai/ha. At three times this rate of application, residues ranged from 0.85 to 1.51 mg/kg (mean 1.20 mg/kg). In later trials in the UK residues were up to 0.21 mg/kg after 32 days but were below 0.05 mg/kg after 42 to 70 days.

Tropical fruits with inedible peel. All trials were carried out in South Africa. Residues in avocado were below 0.05 mg/kg after 43 days. On mangoes, residues were below 0.05 mg/kg 43 days after treatment at either 0.4 or 0.8 kg ai/ha. Residues in papaya were below 0.05 mg/kg after 40 days in two trials.

Following treatment of pineapples at the recommended rate of 0.4 kg ai/ha, residues were below 0.05 mg/kg from 22 to 104 days later. Treatment at 0.8 kg ai/ha showed residues of 0.09 mg/kg at 22 days but 0.05 mg/kg or less from 46 to 104 days after treatment.

Table 1. Residues of cycloxydim in fruit from supervised trials. All trials used EC; mostly 200 g/l, a few 100 g/l.

Crop	Country /'Year	Appl. kg/ha	No. of trials	Residues (mg/kg) at intervals (days) after application	Ref.
Grapefruit	S Afr. '86	0.4	1	<0.05 (102)	87/119E
		0.8	1	<0.05 (102)	87/120E
Lemon	S Afr. '86	0.4	1	<0.05 (102) High blanks	87/29E
		0.8	1	<0.05 (102) High blanks	87/30E
Apple	S Afr. '87	0.4	2	<0.05 (27), <0.05 (49)	87/9E,11E
		0.8	2	<0.05 (27), <0.05 (49)	87/10E,13E
Pear	S Afr. '87	0.4	2	0.06 (35), 0.15 (27)	87/3E,6E
		0.8	2	<0.05 (27,35)	87/4E,8E
Peach	S Afr. '87	0.4	1	0.10 (27)	87/94E
		0.8	1	<0.05 (27)	87/96E
Plum	S Afr. '87	0.4	1	<0.05 (27)	87/137E
		0.8	1	<0.05 (27)	87/138E
Grape	France '86	0.4	3	<0.05 (80, 105, 141)	86/82-84E
		0.6	4	<0.05 (80, 94, 112, 119)	86/78-81E
	'87	0.6	2	<0.05 (71, 95)	87/150,151E
	S Afr. '85	0.4	1	0.14(7),0.2(15),0.14(22),0.13(29)	86/53A
		0.8	1	0.55(7),0.42(15),0.44(22), 0.58(29)	86/54A
	'87	0.4	1	0.37 (27)	87/1E
		0.8	1	0.27 (27)	87/2E
Strawberry	N'lands'88	0.2	1	0.13 to 0.45, mean[8] 0.33 (21)	88/17-20,25-28E
		0.6	1	0.85 to 1.51, mean[8] 1.2 (21)	88/21-24,29-32E
	UK '89	0.2	2	0.10 (65), <0.05 (70)	89/42,48A
		0.45	2	0.20 (65), <0.05 (70)	89/43,49A
		0.45+0.2	2	0.40 (35), <0.05 (42)	89/44,50A
		0.45+0.2	2	0.20 (22), 0.11, 0.15, 0.16, 0.21 (32)	89/46,47A
		0.9+0.2	2	0.60 (35), <0.05 (42)	89/45,51A
Avocado	S Afr. '87	0.4	1	<0.05 (43)	87/72E
		0.8	1	<0.05 (43)	87/73E
Mango	S Afr. '86	0.4	1	<0.05 (43)	87/152E
		0.8	1	<0.05 (143)	87/153E
Papaya	S Afr. '86	0.4	1	<0.05 (40)	87/15E
		0.8	1	<0.05 (40)	87/16E
Pineapple	S Afr. '86	0.4	1	<0.05 (22 to 104)	86/55A
		0.5	1	0.09(22),0.05(46),<0.05(75), 0.05 (104)	86/56A

Vegetables - see Table 2.

Bulb vegetables. Trials in France on leeks gave residues of about 0.1 mg/kg at 55 and 60 days after treatment at recommended levels. Similarly, residues from trials in The Netherlands reached 0.20 mg/kg at 42 days. In one trial in Norway residues were below 0.05 mg/kg after 76 days.

Trials on bulb onions were carried out in Italy, Norway, The Netherlands, South Africa and the UK. Residues at harvest were generally below the high limit of determination of 0.5 mg/kg. Trials on salad onions in the UK showed residues around 0.1 mg/kg or lower.

Brassica vegetables. Trials were carried out on Brussels sprouts, cabbage (white and Savoy) and cauliflower. Residues were very variable, ranging from less than 0.05 to over 1 mg/kg at harvest.

Fruiting vegetables, Cucurbits. Residues on melons in Italy were below 0.05 mg/kg 52 days after application. In a second trial, residues reached 0.40 mg/kg at 30 days. Similar treatment of watermelons gave residues of 0.11 and 0.17 mg/kg after 29 days. Pumpkins treated in South Africa showed no residues above 0.05 mg/kg after 55 to 74 days.

Fruiting vegetables other than Cucurbits. Sweet peppers treated in Italy yielded residues ranging from 0.15 to 0.56 mg/kg from 13 to 35 days after application at recommended rates. Tomatoes in trials in South Africa and Italy showed no residues above 0.05 mg/kg from 53 to 76 days after treatment.

Leafy vegetables. Residues on endive and lettuce treated in Italy were below 0.05 mg/kg; trials on lettuce in France showed up to 0.18 mg/kg after 15 days. One trial on radicchio in Italy gave a residue of 0.67 mg/kg after 11 days.

Legume vegetables. Trials on dwarf beans in the UK showed residues up to 0.37 mg/kg in the pods after 34 days. Green beans were also treated in France, Italy, The Netherlands and South Africa, residues in pods reaching up to 1.4 mg/kg after 27 days.

Treatment of field beans in Great Britain gave rather variable results; residues of 2.5, 0.19, 0.31 and <0.05 mg/kg being found in the dry beans 69, 104, 129 and 162 days, respectively, after application at recommended rates.

Trials on peas were carried out in 5 countries. Maximum residues in the pods and/or seeds at harvest were also variable, ranging up to 6 mg/kg, though most residues were below 1 mg/kg in the whole pods.

Soya bean trials in France, Italy and South Africa showed residues ranging from 0.05 to 0.96 mg/kg after about 100 days. However, trials in Brazil gave residues up to 10 mg/kg after about 50 days.

Root and tuber vegetables. In residue trials on carrots in 4

countries, residues at harvest, 42 to 97 days after treatment, ranged from <0.05 to 0.34 mg/kg. Three trials on parsnips were carried out in the UK. At harvest, 97 to 110 days after treatment, residues were below 0.5 mg/kg.

Extensive residue data were collected from treatments of potatoes in 9 countries. At PHIs of 42 to 120 days, maximum residue levels ranged from <0.05 to 1.6 mg/kg but the majority of results were below 0.5 mg/kg/

Trials on sugar beet in 8 countries provided a lot of data which showed that residues at harvest were at or about the limit of determination, 0.05 mg/kg, with only one or two results reaching the maximum of 0.07 mg/kg. Some trials showed that the tops could yield over 1 mg/kg.

In trials on swedes in Sweden, residues did not exceed 0.05 mg/kg in the seed. Trials on turnips in Norway, Sweden and the UK showed residues in the roots under 0.1 mg/kg at harvest (60 to 108 days) in most cases, although two samples from Sweden showed 0.54 and 0.56 mg/kg at 93 and 101 days after application respectively.

Stalk and stem vegetables. Residues in artichokes and asparagus from trials in Italy were below 0.05 mg/kg. Treatment of celery in France yielded residues up to 0.28 mg/kg.

Table 2. Residues of cycloxydim in vegetables from supervised trials. All trials used EC; mostly 200 g/l, a few 100 g/l.

Crop	Country/ year	Appl., kg/ha	No. of trials	Residues (mg/kg) at intervals (days) after application	Ref.
Leek	France '86	0.3	1	0.07 (60)	86/76E
		0.6	1	0.07 (60)	86/77E
	'87	0.3	1	0.14 (55)	87/90E
		0.6	1	0.10 (55)	87/91E
	N'lands '89	0.6	4	0.07, 0.12, 0.17, 0.20 (42)	89/30,31,75,76
	Norway '87	0.6+0.4	1	<0.05 (76)	87/136E
Onion	Italy '88	0.3	1	<0.5 (80)	88/130E
(bulb)		0.5	1	<0.5 (80)	88/131E
	'89	0.2	1	<0.5 (43)	89/37E
		0.3	1	<0.5 (23-45)	89/23A
		0.5	2	<0.5 (23-45)	89/24A,38E
	'90	0.6	1	<0.5 (13-59)	90/3,4A
	N'lands '88	0.6	1	0.59, 0.60, 0.63, 0.75 (21)	88/51-54E
		0.6	1	<0.5 (21)	88/55-58E
	Norway '86	0.6+0.4	2	<0.5 (38-56)	87/131,132E
	S Afr. '88	0.2	1	<0.5 (32-65)	88/35A
		0.4	1	<0.5 (32-65)	88/36A
	UK '86	1+0.6	1	<0.5 (21-49)	86/64-65A
Onion	UK '86	0.25	1	<0.05 (50)	88/37A

Crop	Country/ year	Appl., kg/ha	No. of trials	Residues (mg/kg) at intervals (days) after application	Ref.
(salad)		0.45+0.2	1	<0.05 (36)	88/40A
		0.5+0.2	1	0.08 (30)	88/38A
		0.9+0.4	1	0.11 (30)	88/39A
Brussels	Norway '86	0.4	1	1.1 (118)	86/90E
sprouts		0.6	1	1.3 (118)	86/91E
	UK '86	0.5+0.3	1	0.98, 1.0, 1.0, 1.0 (129)	86/60A
		0.45+0.2	1	<0.25 (65)	87/40A
	'87	0.45+0.2	1	<0.25, <0.25, 0.30 (78)	88/34A
Cabbage	Italy '88	0.3	2	<0.05 (77)	88/128,133E
		0.5	2	<0.05 (77)	88/129,132E
	Norway '87	0.4	1	0.39 (50)	87/141E
		0.6	1	0.39 (50)	87/142E
	UK '86	0.5+0.3	1	<0.05, <0.05, 0.15 (29)	86/63A
	'87	0.45+0.2	1	0.09, 0.23, 0.37 (29)	87/38A
	'88	0.25	1	0.06 (46)	88/28A
Cabbage	UK 88	0.45+0.2	1	0.41 (21)	88/29A
		0.9+0.4	1	1.1, 1.2 (21)	88/30A
Cauliflower	Italy '88	0.3	1	<0.05 (64)	88/126E
		0.5	1	<0.05 (64)	88/127E
	S Afr. '88	0.2	2	<0.5 (15-60)	88/46,47A
	UK '86	0.5+0.3	1	<0.05, <0.05, 0.48 (29)	86/59A
	'87	0.45+0.2	1	0.3 (27)	87/41A
	'88	0.25	1	<0.05 (55)	88/31A
		0.45+0.2	1	0.41 (30)	88/32A
		0.9+0.4	1	0.67 (30)	88/33A
Melon	Italy '88	0.3	1	<0.05 (52)	88/88E
		0.5	1	<0.05 (52)	88/89E
	'90	0.3	1	0.62 (7), 0.16 (15), 0.05 (30)	90/15A
		0.6	1	1.45 (7), 0.66 (15), 0.40 (30)	90/16A
Pumpkin	S Afr. '86	0.12	1	<0.05 (55-74)	86/57A
		0.24	1	<0.05 (55-74)	86/58A
Watermelon	Italy '90	0.3	1	0.16(0), 0.42(7), 0.22(15), 0.11(29)	90/11A
		0.6	1	0.10(0), 0.72(7), 0.49(15), 0.17(29)	90/12A
Pepper (sweet)	Italy '89	0.3	1	0.15 (13), 0.15 (20), 0.38 (35)	89/17A
		0.5	1	0.27 (13), 0.44 (20), 0.56 (35)	89/18A
Tomato	Italy '88	0.3	1	<0.05 (57-76)	88/96,98,100E
		0.5	1	<0.05 (57-76)	88/97,99,101E
	S Afr. '86	0.12	1	<0.05 (53, 61)	86/66A
		0.24	1	<0.05 (53, 61)	86/67A
Endive	Italy '89	0.2	1	<0.05 (43)	89/25E
		0.5	1	<0.05 (43)	89/26E

Crop	Country/ year	Appl., kg/ha	No. of trials	Residues (mg/kg) at intervals (days) after application	Ref.
Lettuce	France '86	0.3	1	0.05 (20)	86/63E
		0.6	1	0.09 (20)	86/64E
	'87	0.3	1	0.13 (15)	87/98E
		0.6	1	0.18 (15)	87/99E
	Italy '88	0.3	1	<0.05 (30)	88/108E
		0.5	1	<0.05 (30)	88/109E
	'89	0.3	1	<0.05 (43)	89/19E
		0.5	1	<0.05 (43)	89/20E
Radicchio	Italy '88	0.3	1	0.32 (11)	88/110E

Crop	Country/ year	Appl., kg/ha	No. of trials	Residues (mg/kg) at intervals (days) after application	Ref.
		0.5	1	0.67 (11)	88/111E
Beans	France '86	0.3	1	<0.05 (30)	86/74E
(dwarf &		0.6	1	<0.05 (30)	86/75E
green)	Italy '88	0.3	2	<0.05 (26 & 49)	88/70,72E
		0.5	2	0.24 (26), <0.05 (49)	88/71,73E
	'89	0.2	1	<0.05 (40)	89/24E
		0.3	2	<0.05 (52-80)	89/16,27E
		0.5	3	<0.05 (40-80)	89/17,23,28E
	N'lands '88	0.6	1	<0.05, <0.05, <0.05, 0.06, 0.07 (35)	88/41-45E
		0.6	1	0.16, 0.24, 0.25, 0.31, 0.31 (42)	88/46-50E
	'90	0.6	1	1.0, 1.2, 1.3, 1.4 (27)	90/3E
		0.6	1	0.64, 0.67, 0.72, 0.90 (23)	90/4E
	S Afr. '86	0.4	1	0.09 (28)	86/20A
	UK '86	0.5+0.3	1	0.37 (34)	86/19A
	'88	0.45+0.2	1	0.17, 0.18, 0.18 (38)	88/1A
(field &	S Afr. '86	0.12	1	<0.05 (65)	86/11A
dry)		0.25	1	<0.05 (65)	86/12A
	UK '86	0.5+0.5	1	2.4, 2.4, 2.7 (69)	86/50A
	'88	0.25	1	<0.05 (162)	88/21A
		0.45+0.2	2	0.14, 0.19, 0.23 (104), 0.32 (129)	88/24, 22A
		0.9+0.4	1	0.86 (129)	88/23A
Peas	France '86	0.3	1	0.55 (44) (Shelled, green)	86/25E
		0.6	1	0.87 (44) (Shelled, green)	86/26E
	Italy '88	0.3	2	<0.05 (42, 44) (Pods)	88/76,78E
		0.6	2	<0.05 (42, 44) (Pods)	88/77,79E
	N'lands '87	0.6	1	1.1, 1.2, 1.3, 1.3 (55) (Dry peas)	87/64-67E
		0.6	1	2.6, 3.0, 3.7, 4.2 (56) (Dry peas)	87/68-71E
	'88	0.6	1	4.3-6.0, mean[8] 5.5 (21) (Pods)	88/33-40E
	'88	0.6	1	0.09, 0.17, 0.18, 0.19 (90) (Dry peas)	88/59-62E
	'90	0.4	1	0.54, 0.65, 0.67, 0.83 (42) (Pods)	90/5E
		0.4	1	0.15, 0.25, 0.28, 0.28 (58) (Pods)	90/7E
		0.4	1	0.26, 0.34, 0.35, 0.35 (42) (Pods)	90/9E
		0.4	1	0.16, 0.24, 0.29, 0.38 (54) (Pods)	90/11E
		0.6	1	0.19, 0.27, 0.33, 0.37 (42) (Pods)	90/6E
Peas cont.		0.6	1	0.13, 0.23, 0.26, 0.26 (58) (Pods)	90/8E
		0.6	1	0.07, 0.08, 0.18, 0.26 (42) (Pods)	90/10E
		0.6	1	<0.05, 0.07, 0.07, 0.08 (54) (Pods)	90/12E
	Sweden '87	0.4	2	0.38 (89); 0.46 (92) (Dry peas)	87/60,62E
		0.6	2	0.68 (89); 0.69 (92) (Dry peas)	87/61,63E
	'89	0.6	4	0.74-7.1 (61-84) (Shelled, green)	89/14-17E

Crop	Country/ year	Appl., kg/ha	No. of trials	Residues (mg/kg) at intervals (days) after application	Ref.
		0.6	4	0.22-3.9 (61-84) (Shelled, green)	89/57-60E
	UK '85	0.5+0.5	4	3.4-4.3; mean[4] 3.7 (28-30) (Pods)	85/10,11A
	'86	0.5+0.3	1	1.3, 1.6, 1.6 (21) (Pods)	86/18A
	'87	0.45+0.2	2	0.09 (47), 0.46 (51) (Pods)	87/6,7A
	'88	0.25	2	0.35 (57) 0.49 (43) (Shelled, green)	88/8,10A
	'88	0.45+0.2	2	1.0, 1.6 (43) (Shelled, green)	88/9,12A
Soya bean	Brazil '88	0.1	4	1.4, 2.9, 4.5, 5.3 (49-53)	88/9,11,13,15E
		0.2	4	3.1, 4.6, 9.2, 10.6 (49-53)	88/10,12,14, 16E
	'89	0.1	4	0.44, 0.54 (85), 1.8,1.9 (28)	89/7,11,13,17E
		0.15	2	0.48 (85), 2.1 (28)	89/8,14E
		0.2	4	0.57, 0.70 (85), 2.9, 3.2 (28)	89/9,12,15,18E
		0.3	2	0.80 (85), 4.8 (28)	89/10,16E
	France '85	0.3	2	<0.05 (109)	85/6,7E
	'86	0.3	2	0.11 (120), 0.26 (99)	86/53,55E
		0.6	2	<0.05 (120), 0.35 (99)	86/54,56E
	'87	0.6	2	<0.05 (145), 0.24 (154)	87/102,103E
	Italy '86	0.4	2	<0.05 (108, 130)	86/69,92E
		0.6	2	<0.05 (108), 0.13 (130)	86/70.93E
	'87	0.3	4	<0.05(108), 0.23(106), 0.38(105), 0.96(90)	87/107,109, 111,113E
		0.6	3	0.06 (108), 0.63 (90), 0.64 (106)	87/108,110, 112E
	'88	0.5	2	0.24 (103), 0.30 (95)	88/74,75E
	'90	0.5	2	0.33, 0.35 (90-91)	90/1,2A
	S Afr. '86	0.12	1	<0.05 (87)	86/13A
		0.24	1	<0.05 (87)	86/14A
Carrot	France '86	0.3	2	<0.05 (97), 0.06 (6)	86/57,59E
		0.6	2	<0.05 (97), 0.20 (6)	86/58,60E
	'87	0.3	1	<0.05 (64)	87/100E
Carrot cont.		0.6	1	0.12 (64)	87101E
	Italy '88	0.3	1	<0.05 (52)	88/92E
		0.5	1	<0.05 (52)	88/93E
	'89	0.2	2	<0.05 (51), 0.1 (52), <0.05 (69)	89/12,21A
		0.5	2	<0.05 (51), 0.08 (52), <0.05 (69)	89/11,22A
	N'lands '90	0.4	2	0.07-0.26, mean[16] 0.14 (42,56)	90/4,6A
		0.6	2	0.08-0.34, mean[16] 0.18 (42,56)	90/5,7A
	Norway '87	0.6+0.4	1	0.08 (66)	87/147E
	'88	0.4+0.4	2	<0.05 (62, 68)	88/141,143E
Parsnip	UK '88	0.25	1	<0.5 (110)	88/25A
		0.45+0.2	1	<0.5 (97)	88/26A
		0.9+0.4	1	<0.5 (97)	88/27A
Potato	France '85	0.3	2	<0.05 (106)	85/4,5E

Crop	Country/ year	Appl., kg/ha	No. of trials	Residues (mg/kg) at intervals (days) after application	Ref.
	'86	0.3	2	<0.05 (103, 108)	86/15,17E
		0.6	2	<0.05 (103, 108)	86/16,18E
	'87	0.6	1	<0.05 (102)	87/81E
	Germany '86	0.25	5	<0.05-0.29 (42-73), mean[5] 0.11 [Early]	86/35-39A
		0.25	5	<0.05 (68-90) [Middle]	86/40-44A
		0.25	5	<0.05-0.09 (56-108), mean[5] 0.06 [Late]	86/45-49A
	'87	0.25	4	<0.05-0.15 (29-98), mean[4] 0.05 [Early]	87/12-15A
		0.25	4	<0.05 (48-98) [Middle]	87/16-19A
		0.25	4	<0.05 (85-112) [Late]	87/20-23A
	Italy '88	0.3	1	0.08 (85)	88/102E
		0.5	1	<0.05 (85)	88/103E
	N'lands '86	0.6	1	0.70, 0.71, 1.1, 1.5 (51)	86/65-68E
	'87	0.6	1	0.33, 0.44, 0.49, 0.53 (56)	87/74-77E
	'88	0.6	2	<0.05-2.0, mean [8] 1.4 (21)	88/1-8E
	'89	0.6	2	<0.05-0.90, mean [8] 0.5 (20)	89/7,8A
	Norway '86	0.6	2	0.44 (75), <0.05 (119)	86/86,88E
	'87	0.6	1	0.21 (85)	87/143E
	S Afr. '87	0.12	1	0.41 (10), 0.53 (17), 0.46 (31), 0.54 (52)	87/1A
		0.24	1	0.53 (10), 1.5 (17), 0.79 (31), 0.72 (52)	87/2A
	Spain '86	0.2	3	<0.05 (52)	86/71-73E
	Sweden '87	0.4	1	0.29 (91)	87/78E
		0.6	1	0.34 (91)	87/79E
Potato cont.	UK '86	0.3	1	1.6 (68)	86/10E
		0.5	1	0.39 (68)	86/9A
		0.5+0.3	2	0.38 (56), 0.75, 0.82, 0.87 (63)	86/6,7A
	'87	0.45+0.2	2	0.14 (57), <0.05 (100)	87/29,30A
Sugar beet	France '85	0.3	3	<0.05 (124-153) [Root & top]	85/1-3E
	'86	0.3	3	<0.05 (113-145) [Root & top]	86/3,5,7E
		0.6	3	<0.05 (113-145) [Root & top]	86/6,8,10E
	Germany '86	0.5	5	<0.05 (24-132) [Root & top]	86/26-30A
	'87	0.5	4	<0.05 (35-151) [Root & top]	87/8-11A
	Italy '87	0.3	2	<0.05 (84,100) [Root & top]	87/115,117E
		0.6	2	<0.05 (84,100) [Root & top]	87/116,118E
	'88	0.3	1	<0.05 (105) [Root]	88/67E
		0.5	1	<0.05 (105) [Root]	87/68E
	N'lands '87	0.6	2	<0.05-0.08, mean[8] 0.05 (58) [Root] 0.22-0.60, mean[8] 0.38 (58) [Top]	87/33-40E
	Spain '85	0.15	1	0.07 (196)	86/50E
		0.2	1	<0.05 (93)	86/51E
	Sweden '87	0.6	8	<0.05 (82-144)	87/123-30E

Crop	Country/ year	Appl., kg/ha	No. of trials	Residues (mg/kg) at intervals (days) after application	Ref.
	Switz. '86	0.5	1	<0.05 (134)	86/48E
	UK '85	0.5+0.5	3	0.22 (30), <0.05 (42), <0.05 (58) [Root] 0.06-2.7 (30), 1.4 (42), <0.05 (58)[Top]	85/1-3A
		0.15	1	<0.05 (114) [Root & top]	86/1E
		0.5	1	<0.05 (88) [Root], 0.06 (88) [Top]	86/2E
		0.4+0.2	2	<0.05 (87) [Root], <0.05, 0.13 (87)[Top]	86/11,12E
		0.5+0.3	2	<0.05 (69) [Root], <0.05-0.38 (69)[Top]	86/2,3A
Swede	Norway '87	0.6	2	0.06 (103), 0.09 (91)	87/149,139E
	UK '86	0.5+0.3	3	<0.05 (20,60,61), 0.46 (53)	86/61,62A
Turnip	UK '86	0.5+0.3	1	0.46 (53)	87/37A
Artichoke	Italy '88	0.3	2	<0.05 (65)	88/122,124E
		0.5	2	<0.05 (65)	88/125,127E
Asparagus	Italy '88	0.3	1	<0.05 (16)	88/90E
		0.5	1	<0.05 (16)	88/91E
	'89	0.2	1	<0.05 (7, 18, 32)	89/14E
		0.5	1	<0.05 (7, 18, 32)	89/13E
Celery	France '86	0.3	1	0.11 (45)	86/61E
		0.6	1	0.28 (45)	86/62E
	'87	0.3	1	0.09 (54)	87/92E
		0.6	1	0.09 (54)	87/93E

Grasses for sugar production

Sugar cane. Cycloxydim is used on sugar cane at a rate of 0.05 kg ai/ha to control the ripening of the crop. In trials in South Africa residues were <0.05 mg/kg at harvest, 56 days after the treatment. A maximum residue of 0.12 mg/kg was found 7 days after application (89/1A; 89/4, 5A).

Nuts and seeds - see Table 3.

Cotton. In trials in South Africa residues in cotton seed were below 0.05 mg/kg 128 days after an early application. However, when applied two months later, residues reached 1.2 mg/kg after 75 days. No data were provided on the content in cotton seed oil.

Linseed. Trials on flax were made in France and the UK and in all cases residues were close to the limit of determination of 0.05 mg/kg after at least 67 days from treatment, the only positive figure being 0.06 mg/kg which resulted from a treatment at the highest level.

Peanut. Two trials in South Africa showed levels of 0.07 and 0.1 mg/kg at 86 days after application.

Rape. Data from many trials of cycloxydim on rape in 7 countries were available. At PHIs ranging from 70 to over 270 days residue levels ranged up to about 2 mg/kg. Some information on processing to rape seed oil was given in the evaluation of the 1992 JMPR.

Sunflower. The data on residues in sunflower seed were obtained by a method whose limit of determination was 0.5 mg/kg; only one determination exceeded this level (0.75 mg/kg).

Table 3. Residues of cycloxydim in oilseed from supervised trials. All trials used EC; mostly 200 g/l a few 100 g/l.

Crop	Country/Year	Appl. ai kg/ha	No. of trials	Residues (mg/kg) at intervals (days) after application	Ref.
Cotton	S Afr. '86	0.14	1	<0.05 (128)	86/33A
		0.2	1	0.63 (75)	86/31A
		0.24	1	<0.05 (128)	86/34A
		0.4	1	1.2 (75)	86/32A
Linseed	France '86	0.6	1	<0.05 (67)	86/89E
	UK '88	0.25	2	<0.05 (135, 136)	88/41,44A
		0.45+0.2	2	<0.05 (100, 103)	88/42,45A
		0.9+0.4	1	0.06 (100)	88/43A
Peanut	S Afr. '86	0.12	1	0.28 (35),0.18 (50), <0.05 (63),0.07 (86)	86/15A
		0.24	1	0.43 (35),0.28 (50),0.11 (63), 0.10 (86)	86/16A
Rape	France '85	0.3	3	0.06 (279), <0.05 (267), <0.05 (272)	86/19,21,23E
		0.6	3	0.05 (279), <0.05 (267), <0.05 (272)	86/20,22,24E
	Germany '86	0.25	4	0.08-0.53 (83-104), mean[4] 0.28	86/22-25A
	'87	0.25	5	<0.05-0.43 (105-119), mean[5] 0.26	87/24-28A
	Italy '87	0.25	1	0.18 (229)	88/69E
	'90	0.5	1	<0.05 (123)	90/8A
	N'lands '86	0.3	1	<0.05 (304)	86/28-31E
	'87	0.2	2	<0.05 (318, 321)	87/41-44,49-52E
		0.4	2	0.07-0.21 (126), mean[8] 0.13	87/45-48,53-56E
	Norway '87	0.6	1	0.39 (97)	87/122E
	'88	0.4	1	0.18 (70)	88/139E
		0.6	1	0.31 (70)	88/140E
	Sweden '87	0.6	8	<0.05-2.9 (78-111), mean[8] 1.1	87/82-89E
	'89	0.4	1	0.10 (91)	89/6E
		0.6	20	<0.05-0.14 (89-120), mean[20] 0.07	89/3-5,7-12E
					89/40-42,45-47E
					89/51-55E
	UK '87	0.5	3	1.5,1.5,1.8,1.9,2.2 (93), mean[5] 1.8	87/32,34,36A
		0.5+0.5	3	1.4,1.6,1.7,2.0,2.3 (93), mean[5] 1.8	87/31,33,35A

Crop	Country/Year	Appl. ai kg/ha	No. of trials	Residues (mg/kg) at intervals (days) after application	Ref.
Sunflower	France '86	0.3	1	<0.5 (120)	86/95E
		0.6	1	<0.5 (120)	86/94E
	'87	0.6	3	<0.5 (125-166)	87/133-135E
	Italy '88	0.3	2	<0.5 (119)	88/104,106E
		0.5	2	<0.5 (119)	88/105,107E
	'89	0.3	1	<0.5 (80)	89/35E
		0.5	1	<0.5 (80)	89/36E
	S Afr. '88	0.1	1	<0.5 (92)	88/136E
		0.2	1	<0.5 (92)	88/137E
		0.4	1	0.75 (92)	88/138E

Animal feed commodities - see Table 4.

Alfalfa. Data on residue trials were provided from Sweden and South Africa. At PHIs from 41 to 138 days, residues ranged from <0.05 to 0.77 mg/kg.

Hay. Residues in hay in The Netherlands were at a maximum of 0.86 mg/kg after 51 days and below 0.5 mg/kg at 264 and 273 days after treatment.

Table 4. Residues of cycloxydim in animal feeds from supervised trials. All trials used EC; mostly 200 g/l, a few 100 g/l.

Crop	Country/ Year	Appl. kg/ha	No. of trials	Residues (mg/kg) at intervals (days) after application	Ref.
Alfalfa	S Afr. '87	0.2	1	3.9 (7), 1.9 (14), 0.70 (28), 0.23 (53)	87/3A
		0.4	1	9.5 (7), 6.3 (15), 3.0 (28), 0.77 (53)	87/4A
		0.8	1	21 (7), 16 (15), 4.4 (28), 0.75 (53)	87/5A
	'89	0.1	1	0.74 (10), 0.70 (20), 0.11 (31), 0.12 (41)	89/1A
		0.2	1	0.57 (10), 0.50 (20), <0.05 (31), <0.05 (41)	89/2A
		0.4	1	6.1 (10), 2.7 (20), 0.82 (31), 0.44 (41)	89/3A
	Sweden '89	0.4	1	1.1 (28), 0.08 (138)	89/10A
		0.6	2	2.9, 3.1 (28); <0.05, 0.07 (138)	89/8,9A
Hay	N'lands '85	0.3	1	<0.5 (264)	86/32-35E
		0.6	1	<0.5 (264)	86/36-39E
	'86	0.3	1	<0.5 (273)	87/17-20E
		0.3	1	<0.5, <0.5, 0.51, 0.77 (57)	87/40-43E
		0.6	1	0.55, 0.73, 0.78, 0.79 (57)	87/44-47E
	'87	0.3	1	<0.5 (51)	87/21-24E
		0.6	1	0.78, 0.80, 0.82, 0.86 (51)	87/25-28E

APPRAISAL

Cycloxidim, a systemic cyclohexanedione herbicide, was reviewed for the first time by the 1992 JMPR. However, the time available did not allow adequate evaluation of the extensive residue data provided by the manufacturer. These data have been reviewed by the present Meeting.

Residue data were reported from supervised trials of cycloxidim carried out in 15 countries and on over 40 commodities. Although many of these trials were according to registered and/or recommended use patterns, some crop/ application rate combinations were not registered or the resultant data were very limited. In addition, residue data on bulb onions, parsnip, sunflower seed and hay were obtained using an analytical procedure with the comparatively high limit of determination of 0.5 mg/kg while that for Brussels sprouts was 0.25 mg/kg; determinations on all other commodities could be made down to 0.05 mg/kg.

Cycloxidim is applied as a foliar spray directly to the growing crop and also to soil as a surface application. Owing to its systemic properties some uptake and distribution is to be expected although the extent is likely to be variable as it is dependent on the growth stage; this is borne out by the wide variations observed in the residue data presented. As there is little alteration in residue level with time after application,

the PHIs are of little real significance.

These factors combined to make the estimation of suitable maximum residue levels for this compound rather complicated. However, despite these potential drawbacks the residue data were deemed to be sufficient to allow recommendations for MRLs to be made for 16 commodities. They were regarded as being inadequate, for various reasons, to support recommendations for the other commodities for which residue data were available. Data on citrus, pome and stone fruits were sparse, as were those on tropical fruits, cucurbits and some root and stem vegetables. Despite the absence of processing data on grapes, potatoes and sugar beet the Meeting felt able to recommend MRLs for those crops.

Residue data on beans (dry), rape seed and soya bean (dry) were deemed adequate and mutually supportive for a maximum residue level of 2 mg/kg to be estimated for each commodity. Similarly, despite the inherent variability, residue data on Brussels sprouts, cabbage and cauliflower were taken together to estimate a maximum level of 2 mg/kg for brassica vegetables. Data on common bean together with those for peas (in pod) supported a maximum residue level of 1 mg/kg for each, while for shelled peas (green) a level of 2 mg/kg was suggested; for peas (dry) the results were too variable to interpret with any degree of assurance. Residue data on carrot, leek, lettuce (head and leaf) and strawberry were also found to be adequate for the recommendations given in Annex I to be made.

RECOMMENDATIONS

On the basis of the data on residues from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits.

Definition of the residue: sum of 3-thian-3-yl-glutaric acid (TME) and 3-hydroxy-3-thian-3-ylglutaric acid (OH-TME), expressed as cycloxidim.

CCN	Commodity Name	Recommended MRL (mg/kg)		PHI on which based, days
		New	Previous	
VD 0071	Beans (dry)	2	-	-
VB 0040	Brassica vegetables	2	-	-
VR 0577	Carrot	0.5	-	-
VP 0526	Common bean (pods &/or immature seeds)	1	-	-

CCN	Commodity Name	Recommended MRL (mg/kg) New	Previous	PHI on which based, days
FB 0269	Grapes	0.5	-	-
VA 0384	Leek	0.2	-	-
VL 0482	Lettuce (Head)	0.2	-	-
VL 0483	Lettuce (Leaf)	0.2	-	-
VP 0063	Peas	1	-	-
VP 0064	Peas, shelled	2	-	-
VR 0589	Potato	2	-	-
SO 0495	Rape seed	2	-	-
VD 0541	Soya bean (dry)	2	-	-
FB 0275	Strawberry	0.5	-	-
VR 0596	Sugar beet	0.2	-	-
AV 0596	Sugar beet tops or leaves	1	-	-

FURTHER WORK OR INFORMATION

Desirable

1. Residue data from supervised trials on bulb onions, parsnip, sunflower seed and hay, using an analytical procedure with a lower limit of determination of about 0.05 mg/kg.

2. Processing studies on grapes, potatoes and sugar beet treated with cycloxidim in supervised trials.

REFERENCES

All residue data quoted in the Tables were provided by the manufacturer, BASF, Limburgerhof, Germany, and are unpublished. Relevant reference numbers for the many reports are given in the appropriate table or place in the text.

DDT (021)

EXPLANATION

DDT was first evaluated by the JMPR in 1966 and has been reviewed several time since. At the 22nd Session of the CCPR (1991) it was agreed that countries should be requested for information about current registered uses of DDT and about actual residue levels based on uses or monitoring data. This request was repeated by the 23rd Session of the CCPR (1992). At this Session it was also decided to withdraw the general TMRL of 1 mg/kg for DDT in fruits and vegetables, and countries were invited to provide data to enable the JMPR to develop ERLs for DDT in commodities within these groups.

The Meeting has not received any information about uses of DDT. Monitoring data were received from Canada, Denmark, Finland, The Netherlands and the USA.

USE PATTERN

No information was supplied to the Meeting on registered or recommended uses of DDT on crops or animals.

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

Data from monitoring for residues of DDT in plant crops and animal products were received from the governments of Canada, Denmark, The Netherlands and the USA.

The presentation of the data received differed from country to country, and the layouts in the Tables are consequently different.

With a few exceptions all the residues in the Tables are expressed as the sum of *p,p′*-DDT, *o,p′*-DDT, *p,p′*-DDE and *p,p′*-TDE (*p,p′*-DDD), in conformity with the Codex definition.

Table 1. Residues of DDT from monitoring of fruits and vegetables in Canada, 1984-1989.

Commodity		No. of samples	No. of samples with residues[1]	DDT, mg/kg in positive samples	
				Range	Mean
Domestic fruits and vegetables	Blueberries	11	0		
	Carrots	94	11	0.01-0.11	0.04
		1	1		0.26
	Grapes	86	5	0.01-0.03	0.02
	Maize	99	0		
	Pears	100	0		

Commodity		No. of samples	No. of samples with residues[1]	DDT, mg/kg in positive samples	
				Range	Mean
	Potatoes	7	1		0.05
Imported fruits and vegetables	Bananas	100	0		
	Carrots	4	2	0.39, 0.48	0.43
	Celery	18	0		
	Cucumbers	9	1		0.02
	Grapes	99	2	0.03, 0.03	0.03
	Grapefruit	15	0		
	Lemons	5	0		
	Lettuce	100	0		
	Melons	105	0		
	Oranges	30	0		
	Pears	112	5	0.04-0.12	0.08
	Peppers, green	13	0		
	Pineapples	15	0		
	Potatoes	16	2	0.01, 0.07	0.04

[1]Limit of determination 0.01 mg/kg.

Table 2. Residues of DDT from monitoring of animal products in Canada, 1984-1989.

Commodity		No. of samples	No. of samples with residues[1]	DDT, mg/kg in positive samples	
				Range	Mean
Domestic products	Butter	40	4	0.02-0.1	
	Cheese	17	5	0.01-0.06	0.03
	Milk	88	0		
	Eggs	10	0		
	Cattle meat (fat)	12	0		
	Chicken meat (fat)	5	2	0.01, 0.01	0.01
	Pig meat (fat)	42	2	0.10, 0.17	0.14
Imported products	Cheese	124	6	0.01-0.11	0.04

[1]Limit of determination 0.01 mg/kg.

Monitoring of fruits and vegetables in Denmark, 1986-1991. Residues of DDT in the period 1986-1991 were monitored in 1,959 samples of domestic and 2,349 samples of imported fruits and vegetables. During 1986-1989 the limit of determination and reporting was 0.02 mg/kg. In 1990-1991 a reporting limit of 0.10 mg/kg was used. No residues of DDT or its metabolites were present in the crops at these reporting limits.

Table 3. Residues of DDT from monitoring of animal products in Denmark, 1986-1991.

Commodity		No. of samples	No. of samples with residues[1]	DDT, mg/kg in positive samples	
				Range	Mean
Domestic	Butter	989	6	0.02-0.03	0.02
	Cheese	252	3	0.02-0.05	0.03
	Eggs	1073	3	0.02-0.03	0.02
	Cattle meat (fat)	715	27	0.02-0.09	0.04
	Pig meat (fat)	598	40	0.02-0.10	0.04

Commodity		No. of samples	No. of samples with residues[1]	DDT, mg/kg in positive samples	
				Range	Mean
		1	1		0.31
	Poultry meat (fat)	150	5	0.02-0.03	0.03
Imported products	Butter	6	2	0.03-0.03	0.03
	Cheese	222	10	0.02-0.04	0.04
	Eggs	75	0		

[1]Limit of determination 0.02 mg/kg

From Finland the Meeting was supplied with the information that in 1990 residues of *p,p'*-DDE were found in 5 samples of imported eels at levels of 0.03-0.18 mg/kg and in one sample of imported carrots at 0.01 mg/kg.

In The Netherlands agricultural plant products are routinely analyzed for residues of DDT. In the period 1987-1991 about 37,500 samples of fruit, vegetables and cereal grains were analyzed. Residues of DDT, with the limit of determination of 0.01 mg/kg, were present in 54 samples of which 6 were apples. One sample of apples contained 0.5 mg/kg: the MRL in The Netherlands is 0.1 mg/kg.

Table 4. Residues of DDT from monitoring of domestic animal products in The Netherlands, 1990-1992.

Commodity[1]	No. of samples	No. of samples with residues[1]	Limit of determ., mg/kg	DDT, mg/kg in positive samples, range
Veal calf	180	1	0.10	0.11-0.50
Dairy cow	144	2	0.10	0.11-0.50
Broiler	143	0	0.10	
Fattened bull	143	0	0.10	
Pig	324	3	0.10	0.11-0.50
Sheep	72	18	0.01-0.10	0.01-0.05
	1	1		0.11-0.20
Goat	48	5	0.01-0.10	0.01-0.05
Horse	37	9	0.01-0.10	0.01-0.10
Egg powder	24	2	0.01-0.10	0.11-0.20

[1]All residues expressed on fat basis.

From the USA information was supplied on residues of DDT from monitoring carried out in 1991 and 1992. Residue data were received for fruit and vegetables as a whole group, except for residues in citrus fruit, pineapples, carrots, sugar beets and tomatoes. Data were also available for residues in crude soya bean oil and in animal products.

Table 5. Residues of DDT from monitoring of fruit, vegetables and cereal grains in the USA, 1991-1992.

Commodity	No. of samples	No. of samples with residues[1]	90th percentile mg/kg	DDT, mg/kg max.
Domestic				
Carrots	227	32	1991: 0.05	0.17
			1992: 0	
Citrus fruit	408	0		
Pineapples	37	0		
Sugar beet	36	1		0.01
Tomatoes	227	0		
Other fruits and vegetables	6132	235	0	0.30
Soyal bean oil (crude)	8	0		
Whole grain	573	5	0	0.09
Imported				
Carrots	126	16	1991: 0.03	0.13
			1992: 0	
Citrus fruit	302	0		
Pineapples	143	0		
Tomatoes	717	1		0.03
Other fruits and vegetables	6459	26		0.05

[1]Limit of determination 0.01 mg/kg.

Table 6. Residues of DDT from monitoring in the USA of domestic produced milk and eggs, 1991-1992.

Commodity	No. of samples	No. of samples with residues[1]	90th percentile mg/kg	DDT, mg/kg max.
Milk (fat)	748	29	<0.01	0.19
Eggs	621	0		

[1]Limit of determination 0.01 mg/kg.

Table 7. Residues of DDT from monitoring in the USA of imported meat products, 1991-1992.

Commodity[1] (meat)	Year	No. of samples	No. of samples with residues[2]	DDT, mg/kg max.
Cattle	1991	1864	5	0.40
	1992	2070	41	1.8

Commodity[1] (meat)	Year	No. of samples	No. of samples with residues[2]	DDT, mg/kg max.
				2 samples >1.0 5 samples <1.0 and >0.5 3 samples <0.5 and >0.25
Pig	1991	1122	1	0.27
	1992	1059	5	0.19
Sheep, lamb and goat	1991	424	4	1.1
	1992	314	8 (sheep)	0.44
Poultry	1991	59	0	
	1992	14	0	

[1]All residues expressed on fat basis
[2]Limit of determination 0.01 mg/kg.

APPRAISAL

At the 22nd and 23rd Sessions of the CCPR (1991 and 1992) it was agreed that countries should be requested to provide information on registered or recommended uses of DDT and also on residue levels from trials with registered uses or from monitoring. At the 23rd Session the existing Extraneous Residue Limits for DDT in cereal grains, eggs, meat and milk were converted to temporary limits, and the general MRL for DDT in fruits and vegetables was withdrawn. Residue data were necessary to support the existing ERLs and possibly to develop ERLs for DDT in commodities in the fruits and vegetables groups.

The Meeting has received no information about registered or recommended uses of DDT on crops or animals. Monitoring data for residues of DDT in fruits, vegetables, cereal grains and products of animal origin were received from the governments of Canada, Denmark, The Netherlands and the USA.

It was obvious from the monitoring data that residues are not often present in fruit, vegetables and cereal grains, but the incidence observed is of course dependent on the limit of determination used in the monitoring. In Canada, The Netherlands and the USA the limit of determination was 0.01 mg/kg, while the limit in Denmark was 0.02 mg/kg. In Canada DDT was present in 29 of 1100 samples. In Denmark no residues were found in any of 4300 samples. In The Netherlands DDT was present in 54 of 37,500 samples, and in the USA in 369 of 15,445 samples. In most cases residues were low and mostly below 0.1 mg/kg. The frequency of residues in fruit and vegetables seems to be highest in carrots, probably owing to the occurrence of DDT in the soil from earlier uses and the ability of carrots to take up pesticides from the surrounding soil. In Canada and the USA DDT was present in 64 of a total of 452 samples of carrots, which is approximately 14%. It is more surprising that DDT residues were also present in several samples of apples.

Residue data from monitoring cereal grains were available only from the USA. In 579 samples of cereal grains DDT was present in 5 samples with the highest residue at 0.09 mg/kg.

Residue data were available from many samples of animal products such as butter, milk, cheese, eggs and the fat of cattle, pigs, poultry, sheep, goats and horses. Residues of DDT and its metabolites occurred more frequently in animal products than plant products. Residues were mostly at very low levels, but in some samples up to 0.5 mg/kg and in a few samples even higher with a maximum of 1.8 mg/ kg in fat from cattle.

Residues in the monitoring data available to the Meeting were usually

considerably lower than the existing temporary ERLs. Residues in the fat of meat were as mentioned above much lower than the existing limit of 5 mg/kg. In eggs residues with a few exceptions were below the limit of determination (0.01 or 0.02 mg/kg), and were at the level of 0.11 and 0.20 mg/kg in only two samples of egg powder. For milk the existing TMRL is 0.05 mg/kg, approximately 1 mg/kg in milk fat. All residues in samples from the monitoring studies were considerably lower, and generally below the limit of determination. Residues in butter and cheese, calculated as the levels in milk fat, were always considerably lower than 1 mg/kg.

The incidence of detection of environmental contaminants is expected to increase if lower limits of determination are employed. The Meeting noted the remarks made at the 24th Session of the CCPR (ALINORM 93/24, 29) concerning realistic limits of determination, that using methods with low limits of determination was costly and not the best use of resources. The Meeting concluded that for the general monitoring of DDT and the metabolites included in the definition, a suitable limit of determination for the total residue would be 0.02 mg/kg.

As the production of the compound ceases and environmental residues decrease, extraneous residues in food will also decrease. The Meeting therefore recommended that monitoring data should be evaluated again in 1998, with the possibility of lowering the ERLs for DDT.

RECOMMENDATIONS

On the basis of the residue data received from monitoring in four countries the Meeting concluded that the residue levels listed below are suitable for establishing ERLs.

Definition of the residue: Sum of *p,p′*-DDT, *o,p′*-DDT, *p,p′*-DDE and *p,p′*-TDE (*p,p′*-DDD) (fat-soluble).

Commodity		Recommended MRL (mg/kg)	
CNN	Name	New	previous
VR 0577	Carrots	0.5 E	
PE 0112	Eggs	0.1 E	0.5 E T
MM 0095	Meat	1 (fat) E	5 (fat) E T
ML 0106	Milks	0.02 F E	0.05 F E T

FURTHER WORK OR INFORMATION

Desirable

Residue data from monitoring DDT in fruit and vegetables in other countries.

DIAZINON (022)

EXPLANATION

Diazinon was evaluated first for residues in 1967 and subsequently in 1968, 1970, 1975, and 1979. The compound was scheduled for periodic review by the 1989 CCPR (ALINORM 89/24A, para 298, Appendix V).

The general MRL for vegetables (0.5 mg/kg) was proposed for withdrawal by the 1990 CCPR, to be replaced with MRLs for individual commodities based on residue data to be provided to the 1993 JMPR (ALINORM 91/24, para 265). GAP and residue data were requested by a circular letter (CL 1990-20/PR).

Since the last evaluation the use of the compound has been extended to other crops or the recommendations have been adapted to current needs, and some uses have been discontinued. Additional residue studies have therefore been carried out, either to support existing MRLs or to justify new ones.

The Meeting received information on registered uses and residue data from the producer, several countries (Australia, New Zealand, The Netherlands, Spain, Portugal, Finland, Canada, Germany, Thailand) and the GTZ Pesticide Service Project, Germany.

USE PATTERN

Diazinon is an organophosphorus insecticide with a broad spectrum of activity against a wide range of pests: sucking, chewing and boring insects including soil-living insects. It is effective by contact and stomach action. The product has been introduced world-wide in many countries and is used on numerous crop groups or commodities. It is generally used as a foliar or soil spray or applied as a granule to the soil.

Major target crops are leafy, fruiting, stem and root vegetables, deciduous fruit, rice and maize. Minor crops include berries, cereals, citrus, grapes, mushrooms, nut trees, olives and sugar beet. It is also applied to ornamentals, grass, turf, and in nurseries.

For foliar spray treatments EC and WP formulations are used at concentrations of active ingredient between 0.01 and 0.3 kg/hl. The corresponding rates are between 0.18 and about 4.5 kg ai/ha. The number of applications varies according to local conditions and may further change depending on the degree of pest infestation during the growing season.

EC, WP and granular formulations are also recommended for single early-season soil treatment, usually before or at planting or sowing, EC and WP at rates between 2.5 and 6.0 kg ai/ha, and granules at 0.5-10 kg ai/ha.

Powder formulations are seldom recommended; the rates tend to be between 1.0 and 4.0 kg ai/ha, usually with a single application.

Diazinon is officially registered and/or approved as a plant protection agent under the trade name of "Basudin" over much of the world or as D.Z.N. in the USA. Information on

recommended uses of diazinon (GAP) in many countries is listed in Table 1.

Table 1. Summary of GAP in the use of diazinon in various countries.

Crop	Country	Form.	Application			PHI, days
			No.	Rate, kg ai/ha	Conc., kg ai/hl	
Alfalfa	Australia	EC		0.28-0.56		2
Almond	USA	EC	1-4		0.06	
		WP	1-4		0.11-0.17	
Apple	France	WP,EC			0.025-0.03	15
	Italy	EC	1-2	0.45-0.6	0.03-0.04	15
Apricots	Chile	WP,EC	1		0.04-0.042	14
	South Africa	EC		0.5-1.5	0.033-0.044	14
Artichoke	Australia	EC		0.56		14
Asparagus	Portugal				0.03	14
Banana	Australia	EC	2		0.1	14
	Costa Rica	EC		0.30-0.60	0.06	7
	Mexico	WP,EC			0.06-0.09	1
Beans	Australia	EC	3-14	0.12-0.56	0.024	14
	Canada	EC		0.55		7
	Mexico	GR	1	1.25		7
		WP,EC	>1		0.60-1.0	7
	Philippines	EC	>1		0.094-0.15	14
	Portugal	GR	1	1.20-10		
	South Africa	EC			0.044	14
	Switzerland	WP,EC	>1		0.025	21
		GR	1	0.12 g/m		
	USA	GR	1	2.2-4.4		
		WP,EC	1-3	0.42-0.84	0.021-0.90	7
Beetroot	Australia	EC		0.56		14
	Netherlands	WP,EC	1-3	0.135		10
Berries	Norway	EC	>1		0.025-0.05	14
	Switzerland	WP,EC	>1		0.025	21
Blackberries	Canada	WP,EC			0.05	
	Netherlands	WP,EC	1-3	0.135-0.16	0.0135	10
Blueberries	Australia	EC		0.056		14
	USA	WP,EC	>1	1.1-2.2	0.12	7
Cabbage	Australia	EC	>1	0.56-1.12	0.052-0.112	14
(Brassicae)	Belgium	GR	1	0.08 g/pl		
		EC,DP	1	2.5-4.86		10
	Brazil	WP,EC	>1	0.25-0.40	0.05-0.06	4
	Canada	WP,EC	>1	0.55		5-7
		GR	1	7.5 g/m		
		WP	1		0.027	

Crop	Country	Form.	Application			PHI, days
			No.	Rate, kg ai/ha	Conc., kg ai/hl	
	Denmark	GR	1	1.0		70
	Finland	GR	1	0.1-0.2 g/pl		7
		GR	1		0.05a	
	France	WP,EC	1		0.025	15
	Indonesia	EC	>1	0.60-0.80	0.12-0.16	14
	Portugal	GR	1	1.2-10		
	Sweden	GR	1	1.0		
		EC	1	3.25		70
	Switzerland	WP,EC	>1		0.025	14
	Thailand	WP,EC	>1		0.05-0.18	14
	UK	FG	1	22 g/500 pl		14
		FG	1	3.38		14
	USA	WP,EC	>1	0.28-0.56		5-7
		WP,EC,GR	1	1.1-4.4		
Caneberry	USA	WP,EC	>1	1.1-2.2	0.12	7
		WP,EC			0.12-0.24	7
Cantaloupe	Australia	EC	1-3	0.28-1.12	0.024	14
	Canada	EC	>1	0.55-1.0		10
Carrots	Australia	EC		0.52-1.12		14
	Belgium	GR	1	3.85-4.95		
		EC	1-2	3.24-4.86		10
		DP		2.5-3.0		10
	Canada	WP,EC		0.55		10
		WP,GR	1	1.1		
	Denmark	GR	1	1.0		70
	Finland	GR	1	0.2 g/m		7
	France	WG,WP,EC	1	8.0		
	Mexico	GR	1	1.25		

Carrots, contd.	Netherlands	WP,EC	1-2	3.6-5.4		60
		GR	1	3.85-5.5		60
		DP	1	3.4-5.1		
	New Zealand	GR	1	2.2		14
	Portugal	GR	1	1.2-10.4		
	Switzerland	GR	1	0.12 g/m		42
	UK	FG	1	2.8		14
	USA	GR	1	1.1-4.4		
		WP,EC	>1	0.55		10
Celery	Australia	EC		0.52-1.12		14
	Canada	WP,EC		0.55		10
Cereals	Australia	EC		0.56-0.8		14
Cereals, winter	Spain	EC			0.03-0.072	15
Cherry	Chile	WP,EC	1		0.04-0.042	14
Citrus fruit	Australia	EC			0.052	14
	New Zealand	WP	5-8	1.5	0.05	14
	Spain	WP		2.4-4.8	0.04-0.08	20
Cotton	Australia	EC		0.28-0.4		14
	Greece	EC	1	4-6		30
		EC	1		0.045-0.081	30
		EC	1	1.5 g/m		30
	Indonesia	GR	1	2.0-2.5		14
	Mexico	GR	1	1.25		14
		WP,EC	>1	0.3-0.6		14
	Philippines	EC	>1		0.095-0.15	14
	Spain	WP	1-2		0.04-0.08	15
	Thailand	WP,EC	>1		0.05-0.18	14
	USA	WP,EC	>1	0.55-1.1		14
Cranberry	USA	WP,EC	>1	1.1-1.65	0.12-0.18	7
	Canada	WP,EC		2.25-3.5	0.056-0.11	7
Cucumber	Australia	EC	1-3	0.28-1.12	0.024	14
	Brazil	WP,EC	>1		0.05-0.06	4
	Canada	WP,EC		1.12		7
	Mexico	WP,EC	>1	0.25-0.5		7
		GR	1	1.25		7

Cucumber, contd.	Netherlands	WP,EC			0.0135	3
	USA	WP,EC	>1	0.27-0.82		7
		WP,EC,GR	1	2.2-4.4		
Cucurbits	Australia	EC		0.56	0.024	14
	Chile	WP,EC	1-2	0.4-0.6		1
		EC,GR	1	2.2-2.5		45
Currants	Canada	WP,EC			0.05	
	Germany	WP	2	0.4-0.6	0.04	14
	Netherlands	WP,EC	1-3	0.135-0.16	0.0135	10
Egg plant	Australia	EC		0.52-1.12		14
Fodder beet	Canada	WP,EC		0.55		14
	Germany	WP	2	0.24	0.06-0.12	42
	Netherlands	WP,EC	1-3	0.18		
Garlic	Australia	EC		0.56	0.024	14
Gherkins	Australia	EC		0.52-1.12		14
Gooseberry	Canada	WP,EC			0.05	
Grapes	Australia	EC	2-3		0.024	14
	Canada	WP,EC	>1	1.6-3.4	0.05-0.125	16
	Chile	WP,EC	1		0.04-0.042	18
	France	EC	3	0.24		15
	Greece	WP,EC			0.03-0.081	15
	Mexico	WP,EC	>1		0.06-0.09	18
		GR	1-2	2.5-4.0		8
	Netherlands	DP	1	0.34-0.51		b
	New Zealand	WP	6-9	1.0	0.05	14
	Portugal	EC		0.5	0.05	14
					0.03	14
	Spain	WP,EC	2		0.03-0.072	15
		EC	1		0.047-0.93	15
		WP		0.24-0.48	0.04-0.08	15
	Switzerland	EC	>1		0.025	21
	Thailand	WP,EC	>1		0.045-0.18	14
	USA	WP,EC	>1	0.43-0.65	0.0225	7
		WP,EC	1	0.55-1.1	0.06	18
Herbs	Netherlands	WP,EC	1-3	0.078-0.10	0.013	10

Hops	Australia	EC			0.052	
	Canada	WP,EC		0.25-1.12		14
Kiwi fruit	New Zealand	WP,EC	6	1.0	0.05	28
Leek	Netherlands	WP,EC	1	5.4		10
		WP,EC	1-3		0.135-0.36	10
		GR	1	5.5		
		DP	1	5.1		
Lettuce	Australia	EC	1-3	0.56-1.12		14
	Canada	WP,EC		0.55		10
Lettuce	France	WP,EC	1		0.025	15
	Mexico	WP,EC	>1	0.25-0.50		10
		GR	1	1.25		10
	Switzerland	WP,EC	>1		0.025	14
		GR	1	0.12 g/m		28
	UK	FG	1	1.12		14
	USA	WP	>1	0.27-0.55		10
		WP,EC,GR	1	1.1-4.4		
Lima beans	Canada	WP,EC		0.55		3
Loganberry	Canada	WP,EC			0.05	
Macadamia nuts	Australia	EC			0.1	14
Maize/corn	Brazil	EC	>1	0.48		14
	Germany	WP	1	0.80	0.133	
	Greece	GR	1-2	1.5-2.0		15
		WP,EC	1	2.4-6.0		20-30
		WP,EC			0.045-0.08	
		WP	1	0.06-0.15 g/m		20-30
		WP	1		0.04-0.06d	
	Indonesia	EC	>1	0.6-0.8	0.12-0.16	14
	Italy	GR	1	1.0-1.5		15
	Mexico	WP,EC	>1	0.25-0.40		
	Philippines	EC	>1		0.094-0.150	14
	Portugal	GR	1	1.2-10		
	Spain	GR	1	0.50-4.50		30
		WP,EC	2		0.03-0.08	15
	Thailand	WP,EC	>1		0.045-0.18	14
	USA	WP,EC	1	0.55-4.4		
		WP,EC	>1	0.55-1.4		
Melon	Australia	EC	1-3	0.28-1.12	0.024	14
	Brazil	WP,EC	>1		0.05-0.06	14
	Canada	EC	>1	0.55-1.0		10
	Mexico	WP,EC	>1	0.25-0.5		7
		GR	1	1.25		7
	Netherlands	WP,EC			0.0135	3
	Portugal	GR	1	1.2-10		

	USA	WP,EC	>1	0.27-0.82		7
		WP,EC,GR	1	2.2-4.4		
Misc. fruits	Spain	WP		0.6-1.2	0.04-0.08	30
Mushrooms	Australia	EC		112 g/t compost		14
				24 g/t compost after casing		14
	Netherlands	WP,EC	2-3	0.36	0.036	10
		DP	2-3	0.34-0.51		10
		GR,DP	1	55 g/t compost		
		DP	1	1.7 g/t compost		
Nectarines	Chile	WP,EC	1		0.04-0.042	10
Oil seeds	Australia	EC		0.56-0.68		14
Olives	France	WP,EC	1		0.03	21
	Greece	EC			0.03-0.051	
		WP	1	1.5 g/m		
	Italy	EC	1-2	0.60-0.90	0.04-0.06	15
	Mexico	WP,EC	>1		0.06-0.09	75
	Portugal	EC	4	0.60-0.90	0.06-0.09	56
			1		0.03	21
	Spain	WP,EC	1		0.03-0.04	21
		WP		0.4-0.8	0.04-0.08	60
	Turkey	EC	1-2		0.04-0.06	15
	USA	WP,EC	1		0.045-0.06	75
Onions	Australia	EC	2-4	0.56	0.052	14
		EC	1	2.4-4.0		
	Belgium	GR	1	3.85-4.95		
		EC,DP	1	2.5-4.86		10

Onions, contd.	Brazil	EC	>1		0.06	14
	Canada	WP,EC		0.55		10
		WP,GR	1	1.1-2.2		
	Denmark	GR	1	1.0		70
	Finland	GR	1	0.2 g/m		7
	France	WG,WP,EC	1	8.0		15
	Mexico	GR	1	1.25		10
		WP,EC	>1	0.25-0.6		10
	Netherlands	WP,EC	1	5.4		10
		GR	1	1.65		
		DP	1	5.1		
	Philippines	EC	>	1	0.094-0.15	14
	Portugal	GR	1	1.2-10		
	Sweden	EC	1	3.25		
	USA	WP,EC	>1	0.55		10
		WP,EC,GR	1	2.2-4.4		
Parsley	Canada	WP,EC		0.55		7
Parsnip	Australia	EC		0.52-1.12		14
	Canada	WP,EC		0.55		10
		WP,GR	1	1.1		
Pasture	Australia	EC		0.48-1.12		2
	Canada	WP,EC		0.55		21
Peaches	France	WP,EC	1		0.025	14
	Mexico	WP,EC	>1		0.06-0.09	20
		GR	1-2	2.5-4.0		20
	Netherlands	DP	1	0.34-0.51		b
	Portugal	EC	1	0.3	0.03	14
	South Africa	EC		0.5-1.5	0.033-0.044	14
Pear	Italy	EC	1-2	0.45-0.6	0.03-0.04	15
Peas	Australia	EC		0.52-1.12		14
	France	WP,EC	1	0.25		
	Netherlands	WP,EC		0.18-0.45		
	Mexico	GR	1	1.25		4
		WP,EC	>1	0.60-1.0		4
	Philippines	EC	>1		0.094-0.15	4
	Portugal	GR	1	1.20-10		

Peas, contd.	Switzerland	WP,EC	>1		0.025	1
	USA	GR	1	2.2-4.4		
		WP,EC	1-3	0.42-0.56	0.021-0.60	
Peppers	Australia	EC	2-4	0.56-1.12		14
	Canada	WP,EC		0.25-0.55		5
	Chile	WP,EC	1-2	0.40-0.60		5
	Mexico	GR	1	1.25		
		WP,EC	>1	0.60-1.0		5
	Portugal	GR	1	1.2-10		
	USA	WP,EC	>1	0.55		5
		WP,EC,GR	1	1.1-4.4		
Persimmons	New Zealand	WP	6-7	0.75-1.0	0.05	7
Pineapple	Australia	EC			0.052	14
	Costa Rica	EC		0.3-0.6	0.06	7
	USA	WP,EC		2.2-5.8	0.12-0.23	1
Plums	Italy	EC	1-2	0.45-0.6	0.03-0.04	15
	Netherlands	DP	1	0.34-0.51		b
	South Africa	EC		0.5-0.83	0.033	14
Pome fruit	Australia	EC	3-4		0.052	14
	Brazil	EC	>1		0.06	14
	Belgium	EC			0.0012-0.002	10
	Canada	WP,EC	1-8	0.9-2.25	0.025-0.050	14
		WP,EC	1	2.5		c
	Chile	WP,EC	1		0.04	14
	Germany	WP	2-6	0.4-0.9	0.04-0.06	42
	Greece	WP,EC			0.045-0.06	20
	Mexico	GR	1-2	2.5-4.0		14
		WP,EC	>1		0.06-0.09	14
	Netherlands	WP,EC	1	0.135-0.33	0.013-0.022	10
	New Zealand	WP	7-10	1.25-1.5	0.05	14
	Norway	EC	>1		0.025-0.05	14
	Portugal	EC	4	0.36	0.036	14
					0.03-0.06	14
	South Africa	EC	>1	0.5-2.0	0.033-0.044	14
	Spain	WP,EC	4		0.03-0.08	30
		EC+oil	1		0.047-0.0093	

Pome fruit, contd.	Switzerland	WP,EC	>1		0.025	21
	Turkey	EC	1-2		0.04-0.05	21
	USA	WP	2-8		0.06	14
Potatoes	Australia	EC	2-5	0.8		14
	Brazil	EC	>1	0.48		14
	Canada	WP,EC		0.55		14
	Chile	WP,EC	1-2	0.4-0.6		35
	Finland	GR	1	0.3-0.4 g/m		
	Greece	WP,EC	1	2.4-6.0		20-30
		WP	1	0.06-0.15 g/m		20-30
		WP	1		0.04-0.06	
	Italy	EC	1-2	0.18	0.03	15
		DP	1	1.0-4.0		15
	Mexico	GR	1	1.25		35
		WP,EC	>1	0.3-0.6		35
	Norway	EC	>1		0.025-0.05	14
	Portugal	GR	1	1.2-10		
	Spain	GR	1	2.0-4.5		30
		WP		0.4-1.2	0.04-0.08	30
	Thailand	WP,EC	>1		0.05-0.18	14
	USA	GR	1	2.2-4.4		
		WP,EC	>1	0.55-1.1		35
Pumpkin	Australia	EC	1-3	0.28-1.12	0.024	14
Radish	Belgium	EC,DP	1	2.5-4.86		7-21
	Canada	WP,EC		0.55		10
		GR	1	2.2		
	Netherlands	WP,EC	1	3.6		10
		GR	1	3.85		
		DP	1	3.4		
	USA	GR	1	1.1		
		WP,EC	>1	0.28-0.55		10
Rape seed	Australia	EC		0.56		14
Raspberries	Canada	WP,EC			0.05	
	Netherlands	WP,EC	1-3	0.135-0.27	0.0135-0.022	10
Rice	Australia	EC	>1	0.06-0.28		14
	Bangladesh	GR	1	1.68		60

Rice, contd.		EC	2	1.0		30
	Columbia	GR	1-2	1.0-2.0		
		EC	1-3	0.6		15
	Greece	GR	2-3	1.0-1.5		15
		WP,EC	1	2.4-6.0		
		WP	1	0.06-0.15 g/m		
		WP	1		0.04-0.06d	
		WP,EC			0.045-0.08	
	Italy	GR	1	1.0		15
	India	GR	1	1.0-1.5		
		EC	1-3	0.15-0.6	0.03-0.24	14
	Malaysia	EC	2-4		0.10-0.133	7
	Pakistan	GR	2	1.5-2.25		14
		EC,SC	3	0.6-0.9		14
	Philippines	EC	>1		0.095-0.15	14
	Thailand	WP,EC	>1		0.045-0.18	14
Rhubarb	Australia	EC		0.52-1.12		14
Rutabaga	Canada	WP,EC		0.55		14
		WP	1	1.1		
Salsify	Canada	WP,EC		0.55		10
Sorghum	Australia	EC		0.8-1.12		14
Soya beans	Australia	EC		0.56-0.68		14
Spinach	Canada	WP,EC		0.55		10
	France	WP,EC	1		0.025	15
	Switzerland	WP,EC	>1		0.025	14
		GR	1	0.12 g/m		28
	UK	FG	1	1.12		14
	USA	WP	>1	0.27-0.55		10
		WP,EC,GR	1	1.1-4.4		
Squash	Australia	EC	1-3	0.28-1.12	0.024	14
	Canada	EC		0.55-1.0		3
	Mexico	WP,EC	>1	0.25-0.5		7
		GR	1	1.25		7
	USA	WP,EC	>1	0.27-0.82		7e
		WP,EC,GR	1	2.2-4.4		

Stone fruit	Australia	EC	3-4		0.052	14
	Brazil	EC	>1		0.06	14
	Belgium	EC			0.012-0.02	10
	Canada	WP,EC	>1		0.05-0.062	10f
	Germany	WP	3	0.4-0.6	0.04	14
	Greece	WP,EC			0.045-0.08	15
	Netherlands	WP,EC	1	0.135-0.33	0.013-0.022	10
	New Zealand	WP	5-8	0.75-1.0	0.05	14
	Norway	EC	>1		0.025-0.05	14
	Spain	WP,EC	4		0.03-0.08	30
		EC+oil	1		0.0465-0.092	
	Switzerland	WP,EC	>1		0.025	21
	Turkey	EC	1-2		0.024-0.05	21
	USA	WP,EC	>1		0.03-0.06	10f
Strawberries	Canada	WP,EC	1-2	0.5-1.12	0.05-0.1	5
	Chile	WP,EC	>1	0.4-0.6		5
	Indonesia	EC	>1		0.12-0.16	14
	Mexico	GR	1	1.25		10
		WP,EC	>1	0.6-1.0		5
	Netherlands	WP,EC	1-3	0.081-0.135	0.013	10a
	New Zealand	WP	1	0.755		
	Portugal	GR	1	1.2-10		
	Spain	WP		0.4-1.2	0.04-0.08	30
	Switzerland	WP,EC	>1		0.025	21
	USA	WP	1	1.1		
		WP,EC	>1	0.55-1.1	0.06-0.12	5
Sugar beet	Canada	WP,EC		0.55		14
	Chile	WP,EC	1-2	0.4-0.6		14
	France	WP	1	0.15		
		EC	1	1.6		
	Germany	WP	2	0.24	0.06-0.12	42
	Greece	WP,EC	1		0.04-0.081	
		WP,EC	1	4.0-6.0		
		WP,EC	1	0.06-0.15 g/m		
		WP,EC	1		0.04-0.06d	
	Hungary	GR	1	1.75		

Sugar beet, contd.	Italy	EC	1-2	0.18-0.24	0.03-0.04	15
		DP	1	1.0-4.0		15
	Netherlands	WP,EC	1-3	0.18		
	Spain	GR	1	2.0-4.5		30
	Switzerland	EC	>1		0.025	21
	USA	WP,GR	1	1.1-4.4		
		WP,EC	>1	0.82-1.1		
Sugar cane	Australia	EC		0.56-0.68		14
Swedes	Finland	GR	1	0.2 g/m		
Sweet corn	Australia	EC		0.52-1.12		14
	USA	WP,EC,GR	1	2.2-4.4		
		WP	2-3	1.1-1.4		
Swiss chard	Canada	WP,EC		0.55		14
Tomatoes	Australia	EC	3-5	0.28-1.12	0.024	14
	Brazil	EC	>1		0.06	4
	Canada	WP,EC,GR		0.55-1.1		1
	Chile	WP,EC	1-2	0.4-0.6		1
	Mexico	GR	1	1.25		1
		WP,EC	>1	0.6-1.0		1
	Netherlands	WP,EC			0.0135	3
	Portugal	EC	4	0.3	0.03-0.06	14g
	South Africa	EC	>1		0.33-0.44	14
	Switzerland	GR	1	0.12 g/m		
		WP,EC	>1		0.025	14
	USA	WP,EC	>1	0.28-0.82		1
		WP,EC,GR	1	1.1-4.4		
Tree nuts	Spain	WP		0.6-1.2	0.04-0.08	30h
Turf	Australia		>1	0.72-6.0	0.048-0.4	2
Turnips	Australia	EC		0.56		14
	Canada	WP,EC		0.55		14
		WP	1	1.1		
Vegetables	Canada	WP	1	3.4		
	Netherlands	WP,EC	1-3	0.135		10i
	(exc. parsley, celery)	DP	1-6	0.34-0.51		j
	Spain	WP		0.4-1.2	0.04-0.08	30k

Walnuts	USA	EC	1-4		0.06	
		WP	1-4		0.11-0.17	
Watermelon	Australia	EC	1-3	0.28-1.12	0.024	14
	Brazil	WP,EC	>1		0.05-0.06	14
	Canada	EC	>1	0.55-0.85		10
	Mexico	WP,EC	>1	0.25-0.5		7
		GR	1	1.25		7
	USA	WP,EC	>1	0.27-0.82		7
		WP,EC,GR	1	2.2-4.4		

a dip application
b in greenhouse: PHI 17 days in period 01.03-01.11 and 21 days in period 01.11-01.03.
c post-harvest use
d soil drenching, 150 ml solution/m
e PHI 3 days for winter squash
f PHI 20 days for peaches
g PHI 3 days for tomatoes for processing
h PHI 10 days for hazelnuts
i PHI 60 days for carrots
j greenhouse: PHI 3 days for cucumber, melons, tomatoes, and peppers; other vegetables PHI 17 days in period 01.03-01.11 and 21 days in period 01.11-01.03.
k PHI 20 days for artichokes

Diazinon is also applied to cattle against horn fly and face fly in Canada. In Australia it is mainly applied to sheep against lice, keds, and blowfly but also to goats, cattle and pigs against lice, mange, and buffalo fly.

RESIDUES RESULTING FROM SUPERVISED TRIALS

Plant commodities

Residue data from more than 380 trials or projects conducted between 1973 and 1991 are summarized in the following Tables. Trials were carried out with different formulations, mainly in Europe and North America. Trials conducted in the USA were mostly in two States. Residue data from supervised trials were presented in the 1967 monograph and repeated with additions in 1970 (FAO/WHO 1971b). Tables summarizing the results of the residue trials which have been carried out since the last evaluation are as follows.

Table 2: Residues from supervised trials on citrus fruits
Table 3: Residues from supervised trials on pome fruits
Table 4: Residues from supervised trials on stone fruits
Table 5: Residues from supervised trials on grapes
Table 6: Residues from supervised trials on strawberries
Table 7: Residues from supervised trials on other berries
Table 8: Residues from supervised trials on other fruits
Table 9: Residues from supervised trials on bulb vegetables
Table 10: Residues from supervised trials on brassica vegetables
Table 11: Residues from supervised trials on fruiting vegetables, cucurbits
Table 12: Residues from supervised trials on fruiting vegetables, other than cucurbits
Table 13: Residues from supervised trials on leafy vegetables
Table 14: Residues from supervised trials on legume vegetables
Table 15: Residues from supervised trials on root and tuber vegetables
Table 16: Residues from supervised trials on stalk and stem vegetables
Table 17: Residues from supervised trials on cereals
Table 18: Residues from supervised trials on tree nuts
Table 19: Residues from supervised trials on oil seeds

Some samples were an alysed for residues of diazoxon (G 24576) and

hydroxydiazinon (CGA 14128). In nearly all these samples residues were undetectable (<0.01 mg/kg).

Citrus fruits (Table 2)

Six supervised trials on oranges (one variety, one location) and three on mandarins (one variety, one location) were available from Spain. One application was made at a rate of 72 g ai/hl. Two additional supervised trials on oranges with two and four applications at a rate of 60 g ai/hl were reported from Portugal.

Residues in the whole fruit varied between 0.06 and 0.20 mg/kg 14 days after the last application.

Pome fruits (Table 3)

Supervised trials were carried out in Germany (13), Switzerland (6), and the USA (7) with EC and WP formulations. The number of applications varied from two to six foliar treatments at a rate of 0.53-1.2 kg ai/ha (37.5-60 g ai/hl) with spray intervals of 1-3 weeks in the European trials. In the US trials the first application was at the dormant stage (3.36 kg ai/ha) followed by 6 foliar sprays (3.36 kg ai/ha) made at intervals of 1-2 weeks; the concentrations of the spray solutions were 0.12-0.36 kg ai/hl.

For pears supervised trials were conducted in the USA (6) and Germany (1) with the number and rate of applications the same as in the apple trials.

Residues of diazinon in fruit at harvest (PHI 14-30 days) varied between <0.02 and 0.52 mg/kg in the trials with low-concentration spray solutions and between <0.01 and 2.0 mg/kg in the US trials.

Stone fruits (Table 4)

Cherries. Supervised trials were carried out in Germany (11), Switzerland (3), and the USA (29) with EC and WP formulations. The number of applications varied from two to four foliar treatments at a rate of 0.375-0.8 kg ai/ha (25-75 g ai/hl) and spray intervals of 1-2 weeks in the European trials. In the US trials the first application was at the dormant stage (3.3 kg ai/ha) followed by 4 foliar sprays (3.3 kg ai/ha) at intervals of 1-2 weeks, the concentration of the spray solutions being 0.083-0.33 kg ai/hl. Oil was added to all applications.

Residues of diazinon in fruit at harvest (PHI 14-21 days) varied between <0.01 and 0.28 mg/kg with a rapid decrease within the pre-harvest period.

Peaches. Twenty four supervised residue trials (5 Germany, 1 Portugal, 1 Switzerland, 17 the USA) using EC and WP formulations were reported. Two to four foliar sprays were applied at 14-day intervals at a rate of 0.35-0.80 kg ai/ha (23.5-40 g ai/hl). In the US trials there was one application at the dormant stage and four foliar applications at one-week intervals, each at a rate of 3.3 kg ai/ha with added oil.

Residues of diazinon in fruit at harvest (PHI 14-21 days) varied between <0.01 and 0.13 mg/kg.

Plums, prunes. Supervised trials were carried out in Germany (11), Switzerland (1), and the USA (21 with plums, 13 with prunes) with EC and WP formulations. The number of applications varied from two to five foliar treatments at a rate of 0.35-0.94 kg ai/ha (23.5-70 g ai/hl) at intervals of 2-3 weeks in the European trials. In the US trials an application at the dormant stage was followed by 4-5 foliar applications (each 3.3 kg ai/ha) at weekly intervals with oil added to all sprays.

Residues of diazinon in plums at harvest (PHI 10-53 days) varied

between <0.01 and 0.37 mg/kg. In a set of four trials residues were between 0.53 and 0.78 mg/kg after 10 days and between 0.20 and 0.53 mg/kg after 20 days.

In prunes residues ranged from <0.01 to 0.25 mg/kg 10 -20 days after the last application, leading to corresponding residues in the dried fruits of <0.01-1.90 mg/kg.

Berries and other small fruits

Grapes (Table 5). Supervised trials with EC and WP formulations were conducted in France (9), Germany (3), Switzerland (1), and the USA (18) with 1-5 applications at 1-2 week intervals at an application rate of 0.24-1.3 kg ai/ha (24-260 g ai/hl).

Residues of diazinon in grapes at harvest (PHI 7 -49 days) varied between <0.01 and 0.72 mg/kg. In two trials results seemed to be rather high with residues from 1.2-2.6 mg/kg 7-21 days after the last treatment.

In clarified juice and wine from treated grapes no diazinon was detectable (<0.001-<0.02 mg/kg, depending on the laboratory).

Strawberries (Table 6). Thirty nine supervised residue trials, three from New Zealand and the others from the USA, using EC and WP formulations were reported. One foliar application at a rate of 0.5 -1.0 kg ai/ha or one pre-plant application followed by three foliar applications (each at 1.12 kg ai/ha) at weekly intervals were carried out.

After 5-7 days residues of diazinon ranged from <0.01 to 0.12 mg/kg.

Cranberries (Table 7). Three residue trials with granules were reported by Canada. With 1 or 2 applications rates of 3.36 or 6.72 kg ai/ha no residues of diazinon were detectable after 51-59 days.

Currants (Table 7). Supervised trials were carried out in Germany (16) and Switzerland (1) with EC and WP formulations. Trials were conducted with one application of 0.75 g ai/plant or two applications of 0.35 -0.8 kg ai/ha (24-40 g ai/hl) at an interval of 2 weeks.

Seven to 21 days after application, residues of diazinon were between <0.02 and 0.53 mg/kg.

Blackberries, boysenberries, raspberries (Table 7). Twenty residue trials with raspberries, 16 with blackberries and 4 with boysenberries were conducted in the USA using EC and WP formulations. The first application was at the dormant stage with 2.2 kg ai/ha (0.12 -0.6 kg ai/hl) followed by 5 foliar applications at 1.1 kg ai/ha (0.06 -0.3 kg ai/hl) at 2-week intervals.

Residues of diazinon in the berries at harvest (PHI 7 -21 days) were <0.01-0.18 mg/kg.

Assorted tropical and sub-tropical fruits - edible peel (Table 8)

Olives. Four Spanish and two Portugese trials were reported using 1 -2 applications at a rate of 40 or 90 g ai/hl.

Residues of diazinon in mature fruits 14, 35 -41 and about 85 days after application reached levels of 1.2-1.5 mg/kg, 0.03 -1.8 mg/kg, and <0.02-0.07 mg/kg respectively.

In preserved olives (PHI 185 days) residues of diazinon were between 0.04 and 0.09 mg/kg. Residues in crude oil (PHI 35 -41 days and about 85 days) were in the range of 2.7-6.4 mg/kg and 0.10-0.29 mg/kg respectively.

Persimmons. New Zealand reported the results of three residue trials. One

application of 1.25 kg ai/ha (50 g ai/hl) or seven applications at a rate of 0.675-0.9 kg ai/ha (45 g ai/hl) resulted in residues of diazinon of 0.16-0.36 mg/kg seven days after the last application.

Assorted tropical and sub-tropical fruits - inedible peel (Table 8)

Bananas. Seven supervised trials were conducted in Australia (1), Costa Rica (3) and Honduras (3) using GR and EC formulations. The granules were used at a rate of 4 g ai/plant at planting time. For the foliar treatments three applications at 2-5-week intervals were used at concentrations of 40-90 g ai/hl or at 0.6 kg ai/hl for spot treatment.

No residues of diazinon (<0.01/<0.02 mg/kg) were detectable in the whole fruit, pulp or peel at various days after application.

Kiwifruit. Two Italian trials and four trials from New Zealand were reported using EC formulations. Trials were with 48-60 g ai/hl applied one to seven times at 2-4-week intervals.

Residues of diazinon at harvest (PHI 28-42 days) were 0.04-0.19 mg/kg.

Pineapples. Supervised trials were carried out in Costa Rica (4), Honduras (4), and the USA (8) with EC and WP formulations. The number of applications varied from one to nine, at rates of 60-100 g ai/hl sprayed to run-off or 100 ml of spray solution per plant or amounting to a total of 9.7-13.6 kg ai/ha (US trials). Multiple treatments were at 2-5-week intervals. Additional uses involved dip treatments of seedlings at planting time, either as a single treatment (with 0.6 or 1.0% ai solutions) or prior to multiple foliar applications (US trials).

After a single treatment no residues of diazinon were detectable (<0.02 mg/kg) 558 or 574 days after application except one residue of 0.02 mg/kg. After multiple treatments residues between <0.01 and 0.08 mg/kg were detectable 7 days after the last application. Residues after 14 and 21 days were all below 0.01 mg/kg (US trials). No residues (<0.01/<0.02 mg/kg) were detectable in juice prepared from samples harvested 7 days after the last application. Residues between <0.02 and 0.05 mg/kg were detectable in bran or filter cake from the same samples.

In one case hydroxydiazinon (CGA 14128) was detectable in filter cake at a level of 0.02 mg/kg on the day of application.

Table 2. Residues in whole fruit from supervised trials on citrus fruits.

Crop	Application				PHI, days	Residue, mg/kg	Ref.
Country/year	Form	No.	kg ai/ha	kg ai/hl			
Orange							
Spain/1990	EC	1	2.88	0.072	0	0.58	54
					7	0.24	
					14	0.11	
					21	0.05	
					28	0.10	
	EC	1	2.88	0.072	0	0.78	54
					7	0.43	
					14	0.18	
					21	0.10	
					28	0.06	
	EC	1	2.88	0.072	0	0.32	54

Crop	Application				PHI, days	Residue, mg/kg	Ref.
Country/year	Form	No.	kg ai/ha	kg ai/hl			
					7	0.20	
					14	0.06	
					21	0.12	
Spain/1990 cont.					28	0.10	
Spain/1991	EC	1	3.17	0.072	0	0.39	54
					7	0.20	
					14	0.12	
					21	0.06	
					28	0.08	
					42	0.05	
					56	0.05	
	EC	1	3.17	0.072	0	0.44	54
					7	0.22	
					14	0.11	
					21	0.10	
					28	0.10	
					42	0.06	
					56	0.03	
	EC	1	3.17	0.072	0	0.43	54
					7	0.22	
					14	0.11	
					21	0.08	
					28	0.08	
					42	0.04	
					56	0.06	
Portugal/1982		2		0.06	1	0.39	47
					4	0.25	
					7	0.24	
					11	0.15	
					14	0.15	
					21	0.13	
		4		0.06	1	0.50	47
					4	0.36	
					7	0.29	
					11	0.20	
					14	0.20	
					21	0.14	
Mandarin							
Spain/1991	EC	1	2.52	0.072	0	0.45	54
					7	0.17	
					14	0.13	

Crop	Application				PHI, days	Residue, mg/kg	Ref.
Country/year	Form	No.	kg ai/ha	kg ai/hl			
					21	0.13	
Spain 1991 cont.					28	0.14	
					42	0.07	
					56	0.06	
	EC	1	2.52	0.072	0	0.38	54
					7	0.16	
					14	0.10	
					21	0.07	
					28	0.09	
					42	0.07	
					56	0.06	
	EC	1	2.52	0.072	0	0.29	54
					7	0.18	
					14	0.09	
					21	0.13	
					28	0.10	
					42	0.07	
					56	0.06	

Table 3. Residues from supervised trials on pome fruits. Underlined residues are from treatments according to GAP.

Crop	Application				PHI, days	Residue, mg/kg	Ref.
Country/year	Form	No.	kg ai/ha	kg ai/hl			
Apples							
Germany/1974	EC	4	0.53	0.0375	0	0.46	58
					3	0.36	
					7	0.20	
					10	0.16	
					14	0.11	
					21	0.07	
					28	0.05	
					36	<0.02	
	EC	4	0.53	0.0375	0	0.56	58
					3	0.30	
					7	0.24	
					10	0.21	
					14	0.14	
					21	0.13	
					28	0.08	
Germany/1974 cont.					57	0.03	

Crop	Application				PHI, days	Residue, mg/kg	Ref.
Country/year	Form	No.	kg ai/ha	kg ai/hl			
	EC	3	0.53	0.0375	0	0.48	58
					3	0.22	
					7	0.13	
					10	0.11	
					14	0.20	
					21	0.04	
					28	0.03	
					57	<0.02	
	EC	3	0.53	0.0375	0	0.90	58
					3	0.31	
					7	0.34	
					10	0.12	
					14	0.05	
					21	0.05	
					28	0.04	
					57	<0.02	
Germany/1977	WP	4	0.8	0.04	0	0.42	58
		2	1.2	0.06	3	0.40	
					7	0.45	
					14	0.07	
					21	0.10	
					28	0.04	
	WP	4	0.8	0.04	-0	0.70	58
		2	1.2	0.06	+0	0.78	
					3	0.73	
					7	0.84	
					14	0.52	
					21	0.40	
					28	0.30	
	WP	6	0.8-	0.04-	0	1.65	55
			1.2	0.06	14	1.14	
					21	0.61	
					28	0.35	
					35	0.22	
	WP	6	0.8-	0.04-	0	2	55
			1.2	0.06	14	0.44	
					21	0.28	
					28	0.29	
					35	0.22	

Crop	Application				PHI, days	Residue, mg/kg	Ref.
Country/year	Form	No.	kg ai/ha	kg ai/hl			
	WP	6	0.6-	0.04-	7	0.95	55
			0.9	0.06	14	0.76	
					21	0.48	
					28	0.28	
					35	0.22	
	WP	6	0.8-	0.04-	0	2.64	55
			1.2	0.06	14	1.52	
					21	1.1	
					28	0.82	
					35	0.57	
Germany/1980	EC	5	0.75	0.0375	0	0.95	58
					14	0.13	
					21	0.18	
					28	0.08	
					34	<0.02	
					42	<0.02	
	EC	5	0.75	0.0375	-0	<0.02	58
					+0	0.07	
					14	0.02	
					21	0.02	
					28	<0.02	
					35	<0.02	
					42	<0.02	
					49	<0.02	
Germany/1983	WP	5	0.6-	0.04-	0	1.5	55
			0.9	0.06	14	0.31	
					21	0.14	
					28	0.1	
					35	0.1	
Switzerland/1974	EC	2		0.05	0	0.79	58
					4	0.44	
					7	0.49	
					11	0.40	
					14	0.27	
Switzerland cont.					20	0.25	
					28	0.23	
Switzerland/1976	WP	3	0.8	0.04	0	0.13	58
		2	1.2	0.06	3	0.08	
					7	0.07	

Crop	Application				PHI, days	Residue, mg/kg	Ref.
Country/year	Form	No.	kg ai/ha	kg ai/hl			
					10	0.04	
					14	0.03	
					21	0.03	
	WP	3	0.8	0.04	0	0.10	58
		2	1.2	0.06	3	0.06	
					7	0.04	
					10	0.05	
					14	0.04	
					21	<0.02	
Switzerland/1977	WP	4	0.8	0.04	0	0.30	58
		2	1.2	0.06	4	0.09	
					7	0.11	
					14	0.11	
					21	0.07	
					28	0.08	
	WP	4	0.8	0.04	0	0.59	58
		2	1.2	0.06	2	0.15	
					7	0.33	
					14	0.25	
					21	0.08	
					28	0.09	
Switzerland/1980	EC	5	0.75	0.0375	-0	0.02	58
					+0	0.10	
					14	0.04	
					21	0.02	
					28	<0.02	
					35	<0.02	
					42	<0.02	
USA/1988	WP	1 + 6[1]	3.36[1]		14	1.32	58
					21	0.91	
					30	0.32	
	WP	1 + 6	3.36		14	0.42	58
USA/1988 cont.					21	1.13	
					30	0.25	
	WP	1 + 6	3.36		14	0.71	58
					21	1.28	
					30	0.46	
	WP	1 + 6	3.36		14	<0.01	58
					21	0.01	

Crop	Application				PHI, days	Residue, mg/kg	Ref.
Country/year	Form	No.	kg ai/ha	kg ai/hl			
					30	0.01	
	WP	1 + 6	3.36		14	0.14	58
					21	0.10	
					30	0.03	
	WP	1 + 6	3.36		0	0.46	58
					7	0.32	
					14	0.50	
					21	0.20	
					30	0.1	
	WP	1 + 6	3.36		14	A) 0.36	31
					21	A) 0.16	
					30	A) 0.17	
		A) ground			14	B) 0.12	
		B) aerial			21	B) 0.03	
					30	B) 0.07	
					Processed fractions		
				apples	0	0.98	
				culls		2.20	
				wet pomace		1.40	
				dry pomace		0.40	
				fresh juice		0.02	
				canned juice		<0.01	
				canned slices		<0.01	
				frozen slices		<0.01	
				apple sauce		<0.01	

Crop	Application				PHI, days	Residue, mg/kg	Ref.
Country/year	Form	No.	kg ai/ha	kg ai/hl			
Pears							
Germany/1983	WP	5	0.6-	0.04-	0	1.13	55
			0.9	0.06	14	0.04	
					21	0.06	
					28	<0.03	
					35	<0.03	
USA/1988	WP	1 + 6	3.36		14	0.12	58
					21	0.17	
					30	0.10	
	WP	1 + 6	3.36		0	2.75	58
					7	0.31	
					14	0.21	
					21	0.14	
					30	0.03	
	WP	1 + 6			14	0.55	58
					21	0.45	
					30	0.26	
	WP	1 + 6	3.36		14	0.06	58
					21	0.05	
					30	0.02	
	WP	1 + 8	3.36		14	A) 0.01	58
					21	A) <0.01	
					30	A) <0.01	
		A) ground			14	B) <0.01	
		B) aerial			21	B) <0.01	
					30	B) <0.01	
	WP	1 + 6	3.36		14	A) 0.06	58
					21	A) 0.02	
					30	A) <0.01	
		A) ground			14	B) <0.01	
		B) aerial			21	B) <0.01	
					30	B) <0.01	

[1] All US trials were with 1 application of 3.36 kg/ha + oil at the dormant stage followed by 6 (in 1 case 8) foliar sprays at 3.36 kg/ha.

Table 4. Residues from supervised trials on stone fruits. Underlined residues are from treatments according to GAP.

Crop	Application				PHI, days	Residue, mg/kg	Ref.
Country/year	Form	No.	kg ai/ha	kg ai/hl			
Cherries							
Germany/1975	EC	2	0.75	0.0375	0	0.16	58
			g/tree		3	<0.02	
					7	<0.02	
					11	<0.02	
					14	<0.02	
					21	<0.02	
Germany/1980	EC	4	0.5	0.025	-0	<0.02	58
					+0	0.26	
					10	0.04	
					14	0.02	
					21	<0.02	
	EC	4	0.5	0.025	-0	0.08	58
					+0	2.21	
					7	0.72	
					10	0.06	
					14	0.02	
					21	<0.02	
	WP	4	0.8	0.04	-0	0.04	58
					+0	0.53	
					10	0.08	
					14	0.03	
					21	<0.02	
	WP	4	0.8	0.04	-0	0.23	58
					+0	4.24	
					7	0.64	
					10	0.16	
					14	0.04	
					62	<0.02	
Germany/1981	WP	4	0.6	0.12	-0	<0.02	58
					+0	0.30	
					7	<0.02	
Germany/1981 cont.					11	<0.02	

Crop	Application				PHI, days	Residue, mg/kg	Ref.
Country/year	Form	No.	kg ai/ha	kg ai/hl			
					14	<0.02	
					19	<0.02	
	EC	4	0.6	0.075	-0	0.02	58
					+0	0.36	
					7	0.02	
					11	<0.02	
					14	<0.02	
					19	<0.02	
Germany/1983	WP	4	0.6	0.04	-0	0.20	58
					+0	1.10	
					7	0.32	
					10	0.15	
					14	<0.02	
					21	<0.02	
	WP	4	0.6	0.04	-0	0.09	58
					+0	1.60	
					7	0.38	
					10	0.24	
					14	0.04	
					21	0.02	
	EC	4	0.375	0.025	-0	0.42	58
					+0	1.70	
					7	0.34	
					10	0.16	
					14	0.11	
					21	0.09	
	EC	4	0.375	0.025	-0	0.18	58
					+0	2.80	
					7	0.51	
					10	0.30	
					14	0.26	
					21	0.14	
Switzerland/1966	EC	3		0.025	-0	0.20	58
					+0	2.10	
					3	0.80	

Crop	Application				PHI, days	Residue, mg/kg	Ref.
Country/year	Form	No.	kg ai/ha	kg ai/hl			
					5-7	0.40	
					10	0.30	
					14	0.05	
					21-27	0.02	
	EC	2		0.025	-0	0.20	58
					+0	1.50	
					3	0.50	
					5	0.30	
					7-10	0.20	
					14	0.10	
					21	0.04	
					26	0.02	
	EC	1		0.025	-0	<0.05	58
					+0	1.30	
					3	0.40	
					5	<0.05	
					10	0.09	
					13	<0.05	
USA/1988	EC	1+4[1]	3.3		10	0.18	58
	EC+WP	1+4	3.3		10	0.27	58
	WP+EC	1+4	3.3		10	0.09	58
	WP	1+4	3.3		10	0.09	58
	EC	1+4	3.3		10	0.17	58
					20	0.08	
	EC+WP	1+4	3.3		10	0.18	58
					20	0.04	
	WP+EC	1+4	3.3		10	0.29	58
					20	0.12	
	WP	1+4	3.3		10	0.36	58
					20	0.04	
USA 1988 cont.	EC	1+4	3.3		10	0.02	58
					20	<0.01	
	EC+WP	1+4	3.3		10	0.01	58
					20	<0.01	
	WP+EC	1+4	3.3		10	0.03	58

Crop	Application				PHI, days	Residue, mg/kg	Ref.
Country/year	Form	No.	kg ai/ha	kg ai/hl			
					20	<0.01	
	WP	1+4	3.3		10	0.03	58
					20	0.03	
	EC	1+4	3.3		0	13.0	58
					7	0.40	
					10	0.27	
					20	0.07	
					30	0.02	
	EC+WP	1+4	3.3		0	7.95	58
					7	0.77	
					10	0.52	
					20	0.19	
					30	0.04	
	WP+EC	1+4	3.3		0	10.8	58
					7	1.10	
					10	0.59	
					20	0.15	
					30	0.04	
	WP	1+4	3.3		0	6.4	58
					7	1.0	
					10	0.73	
					20	0.28	
					30	0.02	
	EC	1+4	3.3		10	0.13	58
					20	0.01	
	EC+WP	1+4	3.3		10	0.18	58
					20	0.02	
	WP+EC	1+4	3.3		10	0.12	58
USA 1988 cont.					20	0.02	
	WP	1+4	3.3		10	0.32	58
					20	0.03	
	EC+WP	1+4	3.3		10	0.05	58
		(aerial)			20	<0.01	
	EC	1+4	3.3		10	0.08	58
					20	0.01	

Crop	Application				PHI, days	Residue, mg/kg	Ref.
Country/year	Form	No.	kg ai/ha	kg ai/hl			
	EC+WP	1+4	3.3		10	0.06	58
					20	<0.01	
	WP+EC	1+4	3.3		10	0.09	58
					20	<0.01	
	WP	1+4	3.3		10	0.06	58
					20	<0.01	
	EC	1+4	3.3		0	1.05	58
					7	0.05	
					10	0.03	
					20	<0.01	
					30	<0.01	
	EC+WP	1+4	3.3		0	0.09	58
					7	0.02	
					10	<0.01	
					20	<0.01	
					30	<0.01	
	WP+EC	1+4	3.3		0	0.16	58
					7	0.04	
					10	0.02	
					20	<0.01	
					30	<0.01	
	WP	1+4	3.3		0	0.43	58
					7	0.02	
					10	0.01	
					20	<0.01	
					30	<0.01	
Peaches							
Portugal/1976		2	0.03		1	0.82	47
					7	0.09	
					14	0.03	
Germany/1980	WP	4	0.8	0.04	0	0.55	58
					7	<0.02	
					10	<0.02	
					14	<0.02	
					21	<0.02	

Crop	Application				PHI, days	Residue, mg/kg	Ref.
Country/year	Form	No.	kg ai/ha	kg ai/hl			
	WP	2+2	0.56	0.04	0	2.67	58
			0.8	0.04	7	0.39	
					10	0.27	
					14	0.13	
					21	0.05	
	EC	4	0.47	0.0235	0	0.76	58
					7	<0.02	
					10	<0.02	
					14	<0.02	
					21	<0.02	
Germany/1981	WP	4	0.6	0.04	0	0.90	58
					7	0.13	
					10	0.13	
					14	0.08	
	EC	4	0.35	0.0235	0	0.64	58
					7	0.22	
					10	0.14	
					14	0.09	
					21	<0.02	
Switzerland/1981	EC	4	0.47	0.0235	0	0.88	58
					7	0.15	
					10	0.12	
					14	0.06	
					21	<0.02	
USA/1988	EC	1+4	3.3		10	0.04	58
					20	<0.01	
	EC+WP	1+4	3.3		10	<0.01	58
					20	<0.01	
	WP+EC	1+4	3.3		10	<0.01	58
					20	<0.01	
	WP	1+4	3.3		10	0.11	58
					20	0.02	
	EC	1+5	3.3		10	0.11	58
					20	0.02	
	EC+WP	1+5	3.3		10	0.85	58

Crop	Application				PHI, days	Residue, mg/kg	Ref.
Country/year	Form	No.	kg ai/ha	kg ai/hl			
					20	0.05	
	WP+EC	1+5	3.3		10	0.15	58
					20	0.02	
	WP	1+5	3.3		10	1.18	58
					20	0.1	
	EC+WP	1+5	3.3		10	1.03	58
		(aerial)			20	0.11	
	EC	1+4	3.3		9	0.62	58
					19	0.05	
	EC+WP	1+4	3.3		9	0.42	58
					19	0.01	
	WP+EC	1+4	3.3		9	0.77	58
					19	0.05	
	WP	1+4	3.3		9	0.69	58
					19	0.07	
	EC	1+4	3.3		9	0.81	58
					19	0.03	
	EC+WP	1+4	3.3		9	0.78	58
					19	0.03	
	WP+EC	1+4	3.3		9	0.52	58
					19	0.03	
USA 1988 cont.	WP	1+4	3.3		9	0.67	58
					19	0.04	
Plums							
Germany/1974	EC	2	0.94	0.05	0	0.69	58
					7	0.05	
					15	<0.02	
					24	<0.02	
					32	<0.02	
					52	<0.02	
	EC	3	0.705	0.05	0	0.22	58
					3	0.05	
					7	<0.02	
					10	<0.02	
					14	<0.02	

Crop	Application				PHI, days	Residue, mg/kg	Ref.
Country/year	Form	No.	kg ai/ha	kg ai/hl			
					21	<0.02	
					28	<0.02	
					46	<0.02	
Germany/1980	WP	5	0.8	0.04	0	0.33	58
					7	0.04	
					9	0.02	
					14	<0.02	
					21	<0.02	
	WP	5	0.8	0.04	0	0.15	58
					7	<0.02	
					10	<0.02	
					14	<0.02	
					21	<0.02	
					28	<0.02	
					35	<0.02	
	EC	5	0.47	0.0235	0	0.45	58
					7	<0.02	
					10	<0.02	
					14	<0.02	
Germany 1980 cont.					21	<0.02	
					28	<0.02	
Germany/1981	WP	5	0.6	0.04	0	0.15	58
					7	0.03	
					10	0.02	
					14	0.02	
					21	<0.02	
	EC	5	0.375	0.0235	0	0.07	58
					7	0.02	
					10	0.02	
					14	<0.02	
					21	<0.02	
	EC	5	0.352	0.07	0	0.11	58
					7	<0.02	
					10	<0.02	
					14	<0.02	

Crop	Application				PHI, days	Residue, mg/kg	Ref.
Country/year	Form	No.	kg ai/ha	kg ai/hl			
Germany/1982	WP	4	0.6	0.04	0	0.26	55
					7	0.06	
					14	0.01	
					21	<0.01	
	WP	4	0.6	0.04	0	0.14	55
					7	0.02	
					14	0.01	
					21	0.01	
	WP	4	0.6	0.04	0	0.5	55
					7	0.02	
					14	0.06	
					21	0.02	
Switzerland/1974	EC	3	2.5 g	0.05	0	3.0	58
			/tree		3	0.77	
					7	0.18	
					10	0.08	
					14	0.09	
Switzerland 1974					19	0.04	
cont.					33	<0.02	
USA/1988	EC	1+4	3.3		10	0.01	58
					20	<0.01	
	EC+WP	1+4	3.3		10	<0.01	58
					20	<0.01	
	WP+EC	1+4	3.3		10	0.01	58
					20	<0.01	
	WP	1+4	3.3		10	0.01	58
					20	<0.01	
	EC	1+4	3.3		10	0.07	58
					20	<0.01	
	EC+WP	1+4	3.3		10	0.37	58
					20	0.03	
	WP+EC	1+4	3.3		10	0.09	58
					20	0.02	
	WP	1+4	3.3		10	0.10	58
					20	0.02	

Crop	Application				PHI, days	Residue, mg/kg	Ref.
Country/year	Form	No.	kg ai/ha	kg ai/hl			
	EC	1+4	3.3		10	0.78	58
					20	0.28	
	EC+WP	1+4	3.3		10	0.64	58
					20	0.46	
	WP+EC	1+4	3.3		10	0.58	58
					20	0.53	
	WP	1+4	3.3		10	0.53	58
					20	0.20	
	EC	1+4	3.3		10	0.10	58
					20	0.02	
	EC+WP	1+4	3.3		10	0.04	58
					20	0.02	
	WP+EC	1+4	3.3		10	0.10	58
					20	0.01	
	WP	1+4	3.3		10	0.10	58
USA 1988 cont.					20	0.02	
	EC	1+4	3.3		10	0.15	58
					20	<0.01	
	EC+WP	1+4	3.3		10	0.12	58
					20	<0.01	
	WP+EC	1+4	3.3		10	0.22	58
					20	<0.01	
	WP	1+4	3.3		10	0.20	58
					20	<0.01	
	EC+WP	1+4	3.3		10	0.07	58
		(aerial)			20	<0.01	
Prunes	Form.	No.	kg ai/ha	mg/kg fresh	PHI, days	mg/kg, dry	
USA/1988	EC	1+5	3.3	0.13	10	0.02	58
				0.02	20	<0.01	
	EC+WP	1+5	3.3	0.14	10	0.01	58
				0.02	20	<0.01	
	WP+EC	1+5	3.3	0.16	10	<0.01	58
				0.02	20	<0.01	
	WP	1+5	3.3	0.11	10	0.02	58

Crop	Application				PHI, days	Residue, mg/kg	Ref.
Country/year	Form	No.	kg ai/ha	kg ai/hl			
				0.08	20	<0.01	
	EC	1+5	3.3	0.06	10	0.09	58
				0.01	20	0.01	
	EC+WP	1+5	3.3	0.08	10	0.57	58
				0.02	20	0.01	
	WP+EC	1+5	3.3	0.14	10	0.09	58
				0.02	20	0.01	
	WP	1+5	3.3	0.25	10	1.90	58
				0.02	20	<0.01	
	EC+WP	1+5	3.3	0.07	10	0.49	58
		(aerial)		<0.01	20	0.01	
	EC	1+4	3.3	<0.01	10	<0.01	58
				<0.01	20	<0.01	
	EC+WP	1+4	3.3	<0.01	10	<0.01	58
				mg/kg fresh		mg/kg, dry	
				<0.01	20	<0.01	
	WP+EC	1+4	3.3	<0.01	10	<0.01	58
				<0.01	20	<0.01	
	WP	1+4	3.3	<0.01	10	<0.01	58
				<0.01	20	<0.01	

[1]All Us trials were with a single application at the dormant stage followed by 4 or 5 foliar, all at the same rate.

Table 5. Residues from supervised trials on grapes. Underlined residues are from treatments according to GAP.

Country/year	Application				Sample analysed	PHI, days	Residue, mg/kg	Ref.
	Form	No	kg ai/ha	kg ai/hl				
France/1982	EC	1	0.24	0.024	grapes	34	<0.02	58
					wine	52	<0.02	
	EC	1	0.24	0.024	grapes	34	0.02	58
					wine	52	<0.02	
	EC	1	0.24	0.024	grapes	25	<0.02	58
					wine	50	<0.02	
	EC	1	0.24	0.024	grapes	34	0.06	58
					wine	53	<0.02	
France/1983	EC	1	0.067	0.027	grapes	69	0.05	58

Country/year	Application				Sample	PHI,	Residue,	Ref.
	Form	No	kg ai/ha	kg ai/hl	analysed	days	mg/kg	
					wine	83	<0.02	
	EC	1	0.23	0.027	grapes	32	<0.02	58
					wine	44	<0.02	
	EC	1	0.43	0.027	grapes	24	0.15	58
					wine	42	<0.02	
	EC	1	0.43	0.027	grapes	0	0.90	58
						3	0.20	
						7	0.20	
						14	0.09	
						23	0.06	
	EC	1	0.27	0.027	grapes	0	0.20	58
						3	<0.02	
						14	<0.02	
France cont.						21	<0.02	
						28	0.02	
Germany/1980	EC	4	1.04	0.26	grapes	0	0.2	58
						14	0.04	
						21	0.02	
						28	<0.02	
						35	<0.02	
					wine	35	<0.001	
	EC	3	0.98	0.13	grapes	0	1.10	58
		+ 1	1.30	0.13		14	0.16	
						21	0.10	
						28	0.05	
						39	0.07	
					wine	39	<0.001	
	EC	1	0.98	0.098	grapes	0	0.18	58
		+ 3	6.76	0.20		14	0.07	
						21	0.06	
						28	0.06	
						35	0.03	
					wine	35	<0.001	
Switzerland/1980	EC	4	1.3	0.07	grapes	0	0.52	58
						14	0.16	
						21	0.04	
						28	0.06	

Country/year	Application				Sample	PHI,	Residue,	Ref.
	Form	No	kg ai/ha	kg ai/hl	analysed	days	mg/kg	
						35	0.06	
						42	0.04	
						47	0.04	
					wine	47	<0.001	
USA/1988	EC	5	1.12		grapes	7	0.12	58
						14	0.08	
						21	0.03	
	WP	5	1.12			7	0.11	58
						14	0.03	
						21	0.02	
	EC	5	1.12			7	0.03	58
USA cont.						14	0.05	
						21	0.02	
	EC	5	1.12		grapes	7	0.03	58
						14	0.01	
						21	<0.01	
	WP	5	1.12		grapes	7	0.03	58
						14	<0.01	
						21	<0.01	
	EC	5	1.12		grapes	7	0.29	58
						14	0.27	
						21	0.39	
	WP	5	1.12		grapes	7	0.34	58
						14	0.06	
						21	0.1	
	EC	5	1.12		grapes	7	0.26	58
						14	0.13	
						21	0.17	
	WP	5	1.12		grapes	7	0.41	58
						14	0.08	
						21	0.18	
	EC	5	1.12		grapes	7	2.6	58
						14	1.3	
						21	1.3	
	WP	5	1.12		grapes	7	1.2	58
						14	1.9	
						21	1.2	

Country/year	Application				Sample	PHI,	Residue,	Ref.
	Form	No	kg ai/ha	kg ai/hl	analysed	days	mg/kg	
	EC	5	1.12		grapes	7	0.66	58
						14	0.72	
						21	0.39	
	WP	5	1.12		grapes	7	0.43	58
						14	0.57	
						21	0.32	
	EC	5	1.12		grapes	7	0.18	58
						14	0.06	
						21	0.05	
USA cont.	WP	5	1.12		grapes	7	0.13	58
						14	0.11	
						21	0.04	
	EC	5	1.12		grapes	7	0.04	58
						14	0.01	
						21	<0.01	
	WP	5	1.12		grapes	7	<0.01	58
						14	<0.01	
						21	<0.01	
	WP	5	1.12			7	0.05	45
						14	0.07	
						21	0.02	
					grapes, unwashed	7	0.06	45
					wash water		<0.01	
					stems		0.04	
					grapes, destemmed		0.06	
					juice		0.02	
					wet pomace		0.13	
					dry pomace		0.36	
					filter cake		0.01	
					Agrol settling		<0.01	
					sediment		<0.01	
					juice, clarified		<0.01	
					juice, canned		<0.01	

Table 6. Residues from supervised trials on strawberries, all ref. 58. Underlined residues are from treatments according to GAP.

Country/year	Application			PHI, days	Residue, mg/kg
	Form	No.	kg ai/ha		
New Zealand/	WP	1	0.50	0	0.06
1989				3	0.05
				5	0.03
				7	0.01
	WP	1	0.75	0	0.17
				3	0.06
				5	0.02
				7	0.02
	WP	1	1.0	0	0.81
				3	0.14
				5	0.07
				7	0.02
USA/1988	EC	1+3[1]	1.12	3	0.16
				5	0.09
	EC+WP	1+3	1.12	3	0.12
				5	0.08
	WP+EC	1+3	1.12	3	0.18
				5	0.08
	WP	1+3	1.12	3	0.16
				5	0.12
	EC	1+3	1.12	3	0.16
				5	0.08
				7	0.12
	EC+WP	1+3	1.12	3	0.10
				5	0.07
				7	0.07
	WP+EC	1+3	1.12	3	0.11
				5	0.08

Country/year	Application			PHI, days	Residue, mg/kg
	Form	No.	kg ai/ha		
USA cont.				7	0.07
	WP	1+3	1.12	3	0.09
				5	0.07
				7	0.11
	EC	1+3	1.12	3	0.14
				5	0.05
				7	0.05
	EC+WP	1+3	1.12	3	0.16
				5	0.06
				7	0.04
	WP+EC	1+3	1.12	3	0.12
				5	0.05
				7	0.04
	WP	1+3	1.12	3	0.08
				5	0.03
				7	0.05
	EC	1+3	1.12	3	0.11
				5	0.09
				7	0.04
	EC+WP	1+3	1.12	3	0.07
				5	0.07
				7	0.04
	WP+EC	1+3	1.12	3	0.07
				5	0.06
				7	0.06
	WP	1+3	1.12	3	0.07
				5	0.06
				7	0.07
	EC	1+3	1.12	3	<0.01

Country/year	Application			PHI, days	Residue, mg/kg
	Form	No.	kg ai/ha		
				5	<0.01
USA cont.				7	<0.01
	EC+WP	1+3	1.12	3	<0.01
				5	<0.01
				7	<0.01
	WP+EC	1+3	1.12	3	<0.01
				5	<0.01
				7	<0.01
	WP	1+3	1.12	3	<0.01
				5	<0.01
				7	<0.01
	EC	1+3	1.12	3	0.09
				5	0.08
				7	0.03
	EC+WP	1+3	1.12	3	0.06
				5	0.04
				7	0.01
	WP+EC	1+3	1.12	3	0.11
				5	0.06
				7	0.02
	WP	1+3	1.12	3	0.06
				5	0.04
				7	0.01
	EC	1+3	1.12	3	0.13
				5	0.06
				7	0.05
	EC+WP	1+3	1.12	3	0.03
				5	0.02
				7	0.03

Country/year	Application			PHI, days	Residue, mg/kg
	Form	No.	kg ai/ha		
	WP+EC	1+3	1.12	3	0.13
				5	0.08
USA cont.				7	0.06
	WP	1+3	1.12	3	0.08
				5	0.05
				7	0.04
	EC	1+3	1.12	3	0.44
				7	0.04
	EC+WP	1+3	1.12	3	0.09
				7	0.04
	WP+EC	1+3	1.12	3	0.07
				7	0.05
	WP	1+3	1.12	3	0.1
				7	0.05
	EC	1+3	1.12	3	0.01
				5	0.01
				7	<0.01
	EC+WP	1+3	1.12	3	0.02
				5	0.03
				7	0.02
	WP+EC	1+3	1.12	3	0.02
				5	<0.01
				7	0.01
	WP	1+3	1.12	3	0.04
				5	0.01
				7	0.02

[1] 1 pre-plant + 3 foliar

Table 7. Residues from supervised trials on other berries, all ref. 58 except cranberries, Canada, ref. 19. Underlined residues are from treatments according to GAP.

Crop Country/year	Application	PHI, days	Residue, mg/kg

	Form	No	kg ai/ha	kg ai/hl		
Blackberries						
USA/1988	EC	1	2.2	0.12	7	0.06
		+5[1]	1.1	0.06	14	0.06
	EC+WP	1	2.2	0.12	7	0.08
		+5	1.1	0.06	14	0.04
	WP+EC	1	2.2	0.12	7	0.09
			1.1	0.06	14	0.06
	WP	1	2.2	0.12	7	0.08
		+5	1.1	0.06	14	0.07
	EC	1	2.2	0.12	7	0.05
		+5	1.1	0.06	14	0.06
	EC+WP	1	2.2	0.12	7	0.05
		+5	1.1	0.06	14	0.06
	WP+EC	1	2.2	0.12	7	0.07
		+5	1.1	0.06	14	0.08
	WP	1	2.2	0.12	7	0.06
		+5	1.1	0.06	14	0.07
	EC	1	2.2	0.2	7	0.02
		+5	1.1	0.2	14	0.01
	EC+WP	1	2.2	0.2	7	0.02
		+5	1.1	0.2	14	0.01
	WP+EC	1	2.2	0.2	7	0.01
		+5	1.1	0.2	14	0.01
	WP	1	2.2	0.2	7	0.02
		+5	1.1	0.2	14	0.01
	EC	1	2.2	0.2	7	0.02
		+5	1.1	0.2	14	0.01
	EC+WP	1	2.2	0.2	7	0.02
		+5	1.1	0.2	14	0.01
	WP+EC	1	2.2	0.2	7	0.02
		+5	1.1	0.2	14	0.01
	WP	1	2.2	0.2	7	0.02
		+5	1.1	0.2	14	<0.01
Boysenberries						
USA/1988	EC	1	2.2	0.2	7	0.04
		+5	1.1	0.2	14	0.01
	EC+WP	1	2.2	0.2	7	0.03
		+5	1.1	0.2	14	<0.01
	WP+EC	1	2.2	0.2	7	0.05
		+5	1.1	0.2	14	0.02
	WP	1	2.2	0.2	7	0.06
		+5	1.1	0.2	14	0.02
Cranberries						
Canada/1987	GR	2	6.72		59	n.d.
	GR	2	3.36		59	n.d.
	GR	1	3.36		51	n.d.
Currants						
Germany/1975	EC	1	0.75 g/plant	0.0375	0	0.21
					4	0.02

Crop Country/year	Application				PHI, days	Residue, mg/kg
	Form	No	kg ai/ha	kg ai/hl		
					7	0.02
					10	<0.02
					14	<0.02
					21	<0.02
	EC	1	0.75 g/plant	0.0375	0	1.84
					3	0.30
					8	0.16
					11	0.03
					14	0.02
					21	0.05
	EC	1	0.75 g/plant	0.0375	0	2.46
					3	0.30
					6	0.23
					10	0.13
					14	0.06
					21	0.03
Germany/1980	EC	2	0.47	0.024	-0	0.06
					+0	0.39
					7	0.11
					10	0.09
					14	0.07
					21	0.03
	EC	2	0.47	0.024	-0	0.04
					+0	1.22
					7	0.14
					10	0.07
					14	0.02
					21	<0.02
	EC	2	0.47	0.024	-0	0.08
					+0	1.02
					7	0.19
					10	0.13
					14	0.08
					21	0.06
Germany cont.	WP	2	0.8	0.04	-0	0.09
					+0	0.51
					7	0.21
					10	0.13
					14	0.06
					21	0.04
	WP	2	0.8	0.04	-0	0.10
					+0	3.96
					7	0.11
					10	0.05
					14	0.04
					21	0.02
	WP	2	0.8	0.04	-0	0.06

Crop Country/year	Application				PHI, days	Residue, mg/kg
	Form	No	kg ai/ha	kg ai/hl		
					+0	2.12
					7	0.53
					10	0.34
					14	0.21
					21	0.16
	WP	2	0.8	0.04	-0	0.07
					+0	1.94
					7	0.12
					10	0.09
					14	0.06
					21	0.04
Germany/1981	EC	2	0.35	0.025	-0	<0.02
					+0	0.34
					6	0.06
					9	0.06
					13	0.05
					20	0.02
	EC	2	0.35	0.025	0	0.09
					7	0.02
					10	0.02
					15	<0.02
					21	<0.02
	EC	2	0.35	0.025	-0	0.03
					+0	0.34
					7	0.04
					10	<0.02
					14	<0.02
					21	<0.02
Germany cont.	WP	2	0.6	0.04	0	0.4
					7	0.12
					10	0.11
					15	0.08
					21	0.02
	WP	2	0.6	0.04	-0	0.04
					+0	0.46
					7	0.11
					10	0.06
					14	0.02
					21	0.03
	WP	2	0.6	0.04	-0	0.02
					+0	0.17
					6	0.05
					10	0.03
					13	0.02
					20	0.02
Switzerland/	EC	2	0.47	0.024	-0	0.04
1981					+0	0.62

Crop Country/year	Application				PHI, days	Residue, mg/kg
	Form	No	kg ai/ha	kg ai/hl		
					7	0.28
					10	0.23
					14	0.13
					21	<0.02
Raspberries						
USA/1988	EC	1	2.2	0.2	0	1.11
		+5	1.1	0.2	7	0.12
					14	0.04
					21	0.03
	EC+WP	1	2.2	0.2	0	0.76
		+5	1.1	0.2	7	0.06
					14	0.02
					21	0.04
	WP+EC	1	2.2	0.2	0	1.30
		+5	1.1	0.2	7	0.08
					14	0.04
					21	0.03
	WP	1	2.2	0.2	0	1.50
		+5	1.1	0.2	7	0.07
					14	0.02
					21	0.01
USA cont.	EC	1	2.2	0.6	0	3.10
		+5	1.1	0.3	7	0.18
					14	0.04
					21	0.01
	EC+WP	1	2.2	0.6	0	3.20
		+5	1.1	0.3	7	0.11
					14	0.02
					21	0.01
	WP+EC	1	2.2	0.6	0	2.80
		+5	1.1	0.3	7	0.17
					14	0.03
					21	0.01
	WP	1	2.2	0.6	0	3.20
		+5	1.1	0.3	7	0.12
					14	0.03
					21	0.02
	EC	1	2.2	0.2	7	0.02
		+5	1.1	0.2	14	0.02
	EC+WP	1	2.2	0.2	7	0.01
		+5	1.1	0.2	14	0.01
	WP+EC	1	2.2	0.2	7	0.03
		+5	1.1	0.2	14	0.02
	WP	1	2.2	0.2	7	0.02
		+5	1.1	0.2	14	<0.01
	EC	1	2.2	0.6	7	0.09
		+5	1.1	0.3	14	0.02

Crop Country/year	Application				PHI, days	Residue, mg/kg
	Form	No	kg ai/ha	kg ai/hl		
	EC+WP	1	2.2	0.6	7	0.08
		+5	1.1	0.3	14	0.02
	WP+EC	1	2.2	0.6	7	0.06
		+5	1.1	0.3	14	0.01
	WP	1	2.2	0.6	7	0.03
		+5	1.1	0.3	14	<0.01
	EC	1	2.2	0.12	7	0.08
		+5	1.1	0.06	14	0.02
	EC+WP	1	2.2	0.12	7	0.07
		+5	1.1	0.06	14	0.02
	WP+EC	1	2.2	0.12	7	0.07
		+5	1.1	0.06	14	0.02
	WP	1	2.2	0.12	7	0.03
		+5	1.1	0.06	14	0.01

[1] All US trials with 1 application at dormant stage and 5 foliar

Table 8. Residues from supervised trials on other fruits. Underlined residues are from treatments according to GAP.

Crop/country/year	Application				Sample analysed	PHI, days	Residue, mg/kg	Ref.
	Form	No	kg ai/ha	kg ai/hl				
Olives								
Spain/1975		1		0.04	fruit	39	0.09	4
					preserved	39	0.09	
						185	0.09	
				0.04	fruit	39	0.03	4
					preserved	39	0.04	
						185	0.04	
Spain/1976		2		0.04	fruit	89	<0.02	5
					oil	89	0.10	
				0.04	fruit	83	0.07	5
					oil	83	0.29	
Portugal/1980		2		0.09	fruit	1	4.2	47
						7	3.3	
						14	1.5	
						21	2.6	
						28	2.3	
						35	1.8	
						41	0.91	
					oil, crude	14	6.6	
						21	8.4	
						28	9.6	

Crop/country/year	Application				Sample analysed	PHI, days	Residue, mg/kg	Ref.
	Form	No	kg ai/ha	kg ai/hl				
						35	6.4	
						41	4.9	
Portugal/1982		1		0.09	fruit	1	3.0	47
						14	1.2	
						21	1.1	
						28	0.84	
						35	0.62	
						41	0.85	
						49	0.47	
						55	0.56	
Portugal cont.					oil, crude	1	9.0	
						14	5.5	
						21	3.7	
						28	3.6	
						35	3.1	
						41	2.7	
						49	2.0	
						55	1.7	
Persimmons								
N. Zealand/1988	WP	1	1.25	0.05	fruit	1	1.40	24,
						4	0.54	59
						7	0.36	
						14	0.18	
						21	0.09	
						28	0.06	
	WP	1	1.25	0.05	fruit	1	0.88	24,
						4	0.69	59
						7	0.25	
						14	0.10	
						21	0.13	
						28	0.05	
N. Zealand/1991	WP	7	0.675-	0.045	fruit	150	0.22	
			0.900			3	0.38	
						7	0.16	
						14	0.12	
						21	0.04	
						28	<0.02	

Crop/country/year	Application				Sample analysed	PHI, days	Residue, mg/kg	Ref.
	Form	No	kg ai/ha	kg ai/hl				
						42	<0.02	
Bananas								
Australia/1988	EC	3		0.04	whole fruit	76	<0.01	58
Costa Rica/1989	EC	3	0.09	0.6	pulp	0	<0.02	58
						3	<0.02	
						7	<0.02	
						14	<0.02	
					peel	0	<0.02	
Costa Rica cont.						3	<0.02	
						7	<0.02	
						14	<0.02	
Costa Rica/1990	GR	1	4g/plant		pulp	323	<0.02	58
						337	<0.02	
					peel	323	<0.02	
						337	<0.02	
	GR	1	4g/plant		pulp	323	<0.02	58
						337	<0.02	
					peel	323	<0.02	
						337	<0.02	
Honduras/1990	EC	3	1.62	0.09	pulp	0	<0.02	58
						3	<0.02	
						7	<0.02	
						14	<0.02	
					peel	0	<0.02	
						3	<0.02	
						7	<0.02	
						14	<0.02	
	GR	1	4g/plant		whole fruit	260	<0.02	58
	GR	1	4g/plant		whole fruit	274	<0.02	58
Kiwifruit								
Italy/1988	EC	1	0.9	0.06		0	2.30	58
						13	0.34	
						27	0.12	
						34	0.09	
						41	0.04	
	EC	1	1.2	0.06		0	2.20	58
						14	0.40	

Crop/country/year	Application				Sample analysed	PHI, days	Residue, mg/kg	Ref.
	Form	No	kg ai/ha	kg ai/hl				
						28	0.06	
						35	0.04	
						42	0.04	
N. Zealand/1988	EC	5	0.768-0.864	0.05		0	0.67	58
						7	0.16	
						14	0.13	
N. Zealand cont.						21	0.07	
						28	0.05	
	EC	7	0.96-1.2	0.05		0	2.20	58
						7	1.40	
						14	0.97	
						21	0.24	
						28	0.19	
N. Zealand/1989	EC	5	to run off	0.048		0	0.96	58
						7	0.57	
						14	0.11	
						21	0.08	
						28	0.07	
	EC	6	to run off	0.048		0	0.85	58
						7	0.11	
						14	0.11	
						21	0.11	
						28	0.08	
Pineapples								
Costa Rica/1989	EC	3	0.1	0.0625	whole fruit	0	0.10	32
						1	<0.02	
						3	<0.02	
						7	<0.02	
					juice	0	<0.02	
						1	<0.02	
						3	<0.02	
						7	<0.02	
					filter cake	0	0.09	
						1	<0.02	
						3	<0.02	
						7	<0.02	
	EC	3	0.1	0.0625	whole fruit	0	0.05	33

Crop/country/year	Application				Sample analysed	PHI, days	Residue, mg/kg	Ref.
	Form	No	kg ai/ha	kg ai/hl				
						1	<0.02	
						3	<0.02	
						7	<0.02	
					juice	0	0.03	
Costa Rica cont.						1	<0.02	
						3	<0.02	
						7	<0.02	
					filter cake	0	0.59	
						1	<0.02	
						3	<0.02	
						7	<0.02	
Costa Rica/1991	EC	1	0.6% in dip solution	0.6	whole fruit	574	<0.02	58
	EC	1	0.6% in dip solution	0.6	whole fruit	574	<0.02	58
Honduras/1990	EC	3	0.1 l/plant	0.1	whole fruit	0	0.62	49
						1	0.47	
						3	0.06	
						7	0.04	
					juice	0	0.10	
						1	0.10	
						3	<0.02	
						7	<0.02	
					filter cake	0	0.38	
						1	0.38	
						3	0.05	
						7	0.03	
	EC	3	0.1 l/plant	0.1	whole fruit	0	0.24	49
						1	0.16	
						3	0.10	
						7	0.07	
					juice	0	0.10	
						1	0.04	
						3	<0.02	
						7	<0.02	
					filter cake	0	0.39	
						1	0.19	
						3	0.04	

Crop/country/year	Application				Sample analysed	PHI, days	Residue, mg/kg	Ref.
	Form	No	kg ai/ha	kg ai/hl				
						7	0.05	
Honduras/1991	EC	1	1.0% in dip solution	1.0	whole fruit	558	<0.02	58
Honduras cont.	EC	1	1.0% in dip solution	1.0	whole fruit	558	0.02	58
USA/1989	WP	1+8[1]	10.0		whole fruit	7	<0.01	58
						14	<0.01	
						21	<0.01	
					juice	7	<0.01	
					bran	7	0.03	
	WP	1+8	10.8		whole fruit	7	<0.01	58
						14	<0.01	
						21	<0.01	
	WP	1+8	13.6		whole fruit	7	0.08	58
						14	<0.01	
						21	<0.01	
	WP	1+8	13.6		whole fruit	7	0.04	58
						14	<0.01	
						21	<0.01	
	WP	1+8	13.6		whole fruit	7	0.07	58
						14	<0.01	
						21	<0.01	
	WP	1+8	10.1		whole fruit	7	0.03	58
						14	<0.01	
						20	<0.01	
	WP	1+8	9.7		whole fruit	7	0.02	58
						14	<0.01	
						21	<0.01	
	WP	1+8	9.7		whole fruit	7	0.02	58
						14	<0.01	
						21	<0.01	

[1] 1 dip + 8 foliar treatments

Bulb vegetables (Table 9)

Bulb onions. Supervised trials were conducted in Germany (5), Switzerland (6), and the USA (10) using EC, WP, and GR formulations. The result of one trial was reported by Finland. The European trials were carried out with 0.06-0.125 g ai/m row after sowing or planting, or 5.0 kg ai/ha. In US trials a granule was used pre-planting with an application rate of 4.4 kg ai/ha and followed by three foliar sprays at weekly intervals with WP or EC formulations at a rate of 0.55 kg ai/ha.

Residues of diazinon after a single treatment never exceeded 0.1 mg/kg in the bulbs 43 -176 days after application. No residues were detectable in samples taken at normal harvest (<0.02 mg/kg). After pre-plant and foliar treatments residues of diazinon 10 -21 days after the last application varied between <0.01 and 0.04 mg/kg in the bulb.

Spring onions. Eight trials were carried out in the USA with pre-planting granules at an application rate of 4.4 kg ai/ha and three foliar sprays at weekly intervals with WP or EC formulations at a rate of 0.55 kg ai/ha.

Residues in the whole plant 10 to 21 days afte r the last application ranged from <0.01 to 0.65 mg/kg.

Brassica vegetables (Table 10)

Broccoli. Ten trials were carried out in the USA using granules pre-planting at 4.4 kg ai/ha and five foliar sprays at weekly intervals with WP or EC formulations at a rate of 0.55 kg ai/ha.

Residues in the plant 7 to 21 days after the last application were <0.01-0.23 mg/kg.

Head cabbage. Supervised trials were conducted in Germany (4), Switzerland (3), and the USA (20) using EC, WP, and GR formulations. The European trials were carried out with one or two applications after transplanting at 0.1 g ai/m row or 0.02 g ai/plant (25 g ai/hl) at an interval of 1-20 days. In US trials a GR or EC formulation was used before planting at an application rate of 4.4 kg ai/ha followed by five foliar sprays at weekly intervals with WP or EC formulations at a rate of 0.55 kg ai/ha.

Residues of diazinon after one or two treatments ranged from <0.02 to 0.60 mg/kg 21 -86 days after the last application. After pre-plant and foliar applications residues of diazinon 7-21 days after the last treatment varied between <0.01 and 1.8 mg/kg.

Cauliflower. Six supervised trials from Germany (5) and Switzerland using EC and GR formulations were carried out with one or two applications after transplanting at 0.1 g ai/m row or 0.02 g ai/plant (25 g ai/hl) at an interval of 1-14 days.

Residues of diazinon were not detectable (<0.02 mg/kg) 36 -131 days after the last treatment.

Kohlrabi. In six supervised trials from Germany (4) and Switzerland EC or GR formulations were applied once or twice at 0.1 g ai/m row or 0.018 g ai/plant (23.5 g ai/hl) at an early growth stage.

Residues of diazinon ranged from <0.02 to 0.12 mg/kg 14 -113 days after the last treatment.

Fruiting vegetables, Cucurbits (Table 11)

Melon, cantaloupe. Fourteen trials were carried out in the USA using a granule before planting with an application rate of 4.4 kg ai/ha and five foliar sprays at weekly intervals with WP or EC formulations at a rate of 0.83 kg ai/ha.

Residues in the whole fruit 3 to 7 days after the last application ranged from 0.01 to 0.30 mg/kg.

Cucumber. Supervised trials were conducted in Japan (4), Switzerland (1), and the USA (10) with EC, WP, GR and DP formulations applied at 4.48 kg ai/ha to the soil and/or at 0.8 -0.84 kg ai/ha 3 -5 times as foliar treatments.

Residues of diazinon in the cucumbers at harvest (PHI 3 -38 days)

varied between <0.01 and 0.4 mg/kg.

Summer squash, zucchini . Eight trials with summer squash and four trials with zucchini were carried out in the USA using granules pre-planting with an application rate of 4.48 kg ai/ha and five foliar sprays at weekly intervals with WP or EC formulations at a rate of 0.83 kg ai/ha.

Residues of diazinon 3 to 14 days after the last application were <0.01-0.18 mg/kg.

Fruiting vegetables, other than cucurbits (Table 12)

Mushrooms. In four trials in the Netherlands either the compost was treated with 0.05 kg ai/t or the cells twice with 6 g ai/cell.

Residues of diazinon were not detectable (<0.02 mg /kg) 35 days after treatment of the compost. Cell treatment led to residues of 0.07 to 0.17 mg/kg on the day of the last application. Two days later residues ranged from 0.05 to 0.11 mg/kg.

Peppers. Ten trials were carried out in the USA using a granule before planting at 4.48 kg ai/ha and five foliar sprays at weekly intervals with WP or EC formulations at a rate of 0.56 kg ai/ha.

Residues of diazinon 3 to 14 days after the last application ranged from <0.01 to 0.09 mg/kg.

Sweet corn . Supervised trials were conducted in Thailand (4) and the USA (36) using EC, WP, and GR formulations. In the US trials a granule or an EC formulation was used pre-planting at 4.4 kg ai/ha followed by five foliar sprays at approximately two-week intervals with WP or EC formulations at a rate of 1.38 kg ai/ha. Thailand reported results from trials with an EC formulation applied four times at rates between 0.65 and 2.12 kg ai/ha (0.051 and 0.102 kg ai/hl).

No residues of diazinon were detectable (<0.01 mg/k g) 10 or 14 days after the last application. After seven days residues were <0.01 -0.02 mg/kg. In the forage residues were detectable at levels of 0.04 -7.95 and <0.01-4.95 mg/kg 7 and 14 days after the last application.

Tomatoes. Supervised trials were reported from Canada (1), The Netherlands (2, indoor) and the USA (38) using EC, WP, SP and GR formulations. In the US trials a pre-planting granule or EC at 4.48 kg ai/ha was followed by five foliar sprays at weekly intervals with WP or EC formulations at 0.84 kg ai/ha. Canada reported one treatment with an EC formulation at a rate of 0.8 kg ai/ha (0.1 kg ai/hl) and The Netherlands one treatment with an SP formulation at 0.19 kg ai/ha (12.7 g ai/hl).

In the indoor trials no residues were detectable (<0.04 m g/kg) three days after the application. In the other trials residues of diazinon ranged from <0.01 to 0.48 mg/kg 1 -14 days after the last application. Residues in processed fractions are discussed in "Fate of residues" below.

Leafy vegetables (Table 13)

Chinese cabbage . Supervised trials were reported by Finland (2) and Thailand (4). In Finland granules were applied at 0.1 and 0.2 g ai/plant. In Thailand four foliar EC sprays were applied at 0.373 -2.31 kg ai/ha (0.06-0.228 kg ai/hl).

After 62 days residues of 0.17 and 0.34 mg/kg were detectable in the trials with granules. Seven days after the last spray application, residues of diazinon were within a range of 0.02-0.62 mg/kg.

Endive. The Netherlands reported the result of a trial after one application of a WP formulation at a rate of 0.255 kg ai/ha (25.5 g ai/hl).

In the head a residue of 0.55 mg/kg was found two days after application, which was reduced by washing and cooking to 0.33 mg/kg and 0.02 mg/kg respectively.

Kale. Six supervised trials were reported by GTZ from Thailand using five foliar EC sprays at 1.8 and 3.6 kg ai/ha (0.18 and 0.36 kg ai/hl).

Fourteen days after the last spray residues of diazinon were within a range of <0.02 to 0.05 mg/kg.

Lettuce, head . Twenty-five supervised trials were reported from The Netherlands, Switzerland, and the USA (23) using EC, WP, and GR formulations. In the US trials 4.48 kg ai/ha (GR or EC) before planting was followed by five foliar sprays at weekly intervals with WP or EC formulations at a rate of 0.56 kg ai/ha. In Switzerland one granular application was made at a rate of 0.2 g ai/m row at the pre-plant stage, and in The Netherlands one application of a WP formulation at 0.255 kg ai/ha (25.5 g ai/hl).

Residues of diazinon in the heads ranged from <0.01 to 0.20 mg/kg 14-21 days after the last application. After the row treatment residues were detectable at 0.05 and 0.03 mg/kg at 70 and 76 days respectively. In the trial in The Netherlands a residue of 0.32 mg/kg was detectable two days after application, which was reduced by washing to 0.21 mg/kg.

Lettuce, leaf. Twenty supervised trials were reported from the the USA in which 4.48 kg ai/ha (GR or EC) was applied before planting, followed by five foliar sprays at weekly intervals with WP or EC formulations at a rate of 0.56 kg ai/ha.

Residues of diazinon in the leaves ranged from <0.01 to 0.15 mg/kg 14-21 days after the last application.

Spinach. Supervised trials were conducted in Italy (2), Switzerland (1), and the USA (8) using EC, WP, and GR formulations. The European trials were with foliar applications of 0.3 -0.8 kg ai/ha. In the US trials a granule was used pre-planting at 4.48 kg ai/ha followed by five foliar sprays at weekly intervals with WP or EC formulations at a rate of 0.56 kg ai/ha.

Residues of diazinon in the leaves ranged from <0.01 to 0.37 mg /kg 14-21 days after the last application.

Legume vegetables (Table 14)

Common beans. Supervised trials were conducted in Canada (1), Switzerland (3), Thailand (6), and the USA (20) using EC, WP, GR, and DP formulations. In the US trials a pre-plant GR or EC formulation at 4.4 kg ai/ha was followed by three foliar sprays at 5 -day intervals with WP or EC formulations at 0.83 kg ai/ha (0.11-0.46 kg ai/hl). In the Canadian trial a WP formulation was applied at 0.5 kg ai/ha (62.5 g ai/hl). The trials in Thailand, reported by the GTZ, were with an EC formulation at rates of 3.9 and 7.8 kg ai/ha (0.18 and 0.36 kg ai/hl, 5 foliar sprays).

In all cases the pods were analysed. Residues of diazinon were <0.01-0.38 mg/kg and <0.01 -0.05 mg/kg after 5 -8 and 10 -14 day s respectively.

Peas. Twenty supervised trials from the USA using EC, WP and GR formulations were reported. Trials were with 4.4 kg ai/ha as an EC or GR formulation at the pre-plant stage followed by 3 -5 foliar sprays at a rate of 0.83 kg ai/ha (0.11 -0.45 kg ai/hl) at weekly intervals with EC or WP formulations.

Residues of diazinon in peas were <0.01 -0.15 and <0.01-0.09 mg/kg at 7 and 14 days, respectively, after the last application.

Root and tuber vegetables (Table 15)

Carrots. Supervised trials were conducted in Germany (3), The Netherlands (6), Switzerland (5), and the USA (16) using EC, WP, and GR formulations. In US trials a GR or EC formulation was used pre-planting or at seeding time with an application rate of 4.4 kg ai/ha, followed by five foliar sprays at weekly intervals with WP or EC formulations at 0.55 kg ai/ha. The European trials were with single applications of either granules at 0.08 g ai/m row or 5.0 kg ai/ha, or an EC formulation at a rate of 0.125 g ai/m row or 2.7-5.4 kg ai/ha (0.1-0.54 kg ai/hl), all at stages from sowing up to a plant height of about 10 cm.

After the single treatment residues of diazinon in the roots ranged from <0.005 to 0.20 mg/kg 1-6 months after the application. In one Swiss trial a residue of 1.08 mg/kg after 57 days seemed to be unrepresentative. In trials with multiple treatments residues varied from 0.02 to 0.41 mg/kg after 7-14 days.

In some samples traces up to 0.01 mg/kg of hydroxydiazinon (CGA 14128) were detectable.

Celery. The result of one trial was available from The Netherlands. One application was made at a rate of 3.6 kg ai/ha (0.36 kg ai/hl) using an EC formulation. The residue of diazinon in the tuber reached 4.05 mg/kg 27 days after the application.

Parsley, turnip-rooted. One trial was reported by The Netherlands. An EC formulation was applied once at 3.6 kg ai/ha (0.36 kg ai/hl). The residue of diazinon in the root reached 3.49 mg/kg after 27 days.

Potatoes. Supervised trials were conducted in Canada (4), France (1), and the USA (23) using EC, WP, and GR formulations. In the US trials a GR or EC pre-planting application at 4.4 kg ai/ha was followed by five foliar sprays at weekly intervals with WP or EC formulations at a rate of 0.55 kg ai/ha. The other trials were with granules at 2.25 or 10 kg ai/ha one month after planting.

No residues of diazinon were detectable (<0.01 or <0.02 mg/kg) in any of the harvested samples except in three trials where residues of 0.01 mg/kg were found.

Residues in processed fractions only occurred in dry peel.

Radish. Seventeen supervised trials from Switzerland and the USA (16) using EC, WP and GR formulations were reported. In the US trials a GR or EC formulation was used pre-planting at 4.48 kg ai/ha followed by three or four foliar sprays at weekly intervals with WP or EC formulations at 0.56 kg ai/ha. The Swiss trial was with granules at 8.7 kg ai/ha at the 4-6 leaf stage.

Residues of diazinon in the roots ranged from <0.01 to 0.08 mg/kg 7-33 days after the last application.

Sugar beet. Supervised trials were conducted in Germany (4), Switzerland (2), and the USA (11) using EC, WP, and GR formulations. The European trials were with 2-4 applications of 0.14-0.5 kg ai/ha (0.023-0.5 kg ai/hl) at 2-4 week intervals. In the US trials a GR or EC formulation was used pre-planting at an application rate of 4.4 or 5.5 kg ai/ha followed by four or five foliar sprays at weekly intervals with WP or EC formulations at 0.55 kg ai/ha.

Residues of diazinon were not detectable (<0.01 or <0.02 mg/k g) in the roots or tops after 21 days and longer. Fourteen days after the last application residues in roots and tops were <0.01-0.10 mg/kg and <0.02-2.51 mg/kg respectively. No residues were detectable (<0.01 mg/kg) in processed

fractions, except a residue of 0.01 mg/kg in pulp.

Residues of hydroxydiazinon (CGA 14128) were detectable up to 0.02 mg/kg in some samples harvested 14 days after the last application.

Sugar beet roots containing no residues (<0.01 mg/kg) in harvest samples after treatment at the normal recommended rate were processed. The only diazinon residues detected were in pulp at 0.01 mg/kg.

Stalk and stem vegetables (Table 16)

Artichoke. Three supervised trials (one variety, one location) were available from Spain. One application was made at 1.512 kg ai/ha (72 g ai/hl) using an EC formulation. Residues in the fruit varied between 0.18 and 0.30 mg/kg 21 days after the application.

Witloof. The result of one trial was available from The Netherlands. One application was made at 3.3 g ai/cell using tablets. No residue was detectable (<0.01 mg/kg) after 14 days.

Cereal grains (Table 17)

Maize/corn. Supervised trials were conducted in Germany (4), Switzerland (2), and the USA (4) with DS, EC, and GR formulations. The European trials were with either 0.5 kg ai/100 kg seed or 0.94 -1.0 kg ai/ha (0.1 -0.164 kg ai/hl) at the 1 -3 leaf stage. In the US trials a granule was used four times with an application rate of 1.1 kg ai/ha at weekly intervals.

No residues of diazinon were detectable (<0.02 mg/kg) 58 -156 days in whole plants, grain or residual plants 58-156 days after single seed or early foliar treatments. After multiple applications residues in the grain were at the limit of determination (<0.01 mg/kg) 43 -45 days after the last application. In maize fodder residues were higher, ranging from 0.29 to 3.15 mg/kg. Maize silage harvested 10 days after the last application contained residues between 1.18 and 5.4 mg/kg.

Whole kernels from harvest samples after treatment at the normal recommended rate were processed by dry and wet milling. Residues were low in all fractions, never exceeding 0.1 mg/kg except in maize oil.

Rice. Seven supervised trials were conducted in India (5), Indonesia, and Pakistan using EC and GR formulations. One to three granular treatments at 1.0-1.68 kg ai/ha or 3-5 foliar treatments at 0.125-0.6 kg ai/ha (0.05-0.12 kg ai/hl) at 2-3-week intervals were carried out.

No residues were detectable (<0.02 or <0.03 mg/kg) in the grain or the unhusked grain 23 -59 days after the last application. Small residues were detectable in the straw, reaching levels up to 0.04 mg/kg.

Tree nuts (Table 18)

Almonds. The results of four projects (18 trials) were reported from California/USA using EC and WP formulations. Trials were with 3.3 kg ai/ha at the dormant stage followed by three foliar sprays at the same rate (0.18-1.77 kg ai/hl) at two-week intervals. Ground and aerial applications were reported.

Residues in the whole nut were small, ranging from <0.01 to 0.02 mg/kg 28 -45 days after last treatment. At the same intervals residues in the hull varied between 0.02 and 4.35 mg/kg.

In some cases diazoxon (G 24576) and hydroxydiazinon (CGA 14128) were detectable in the hulls at levels of 0.02 -0.10 and 0.01 -0.08 mg/kg respectively.

Walnuts. Twenty four supervised trials were conducted in California/USA

using EC and WP formulations. Trial conditions were similar to those for almonds, but oil was added to all applications.

No residues were detectable in the whole nuts (<0.01 mg/kg) 14 -45 days after the last application.

Oilseed (Table 19)

Cotton seed. Four supervised trials were reported from India with two or five applications of EC at 0.4 or 0.75 kg ai/ha (0.064 or 0.12 kg ai/hl).

No residues of diazinon (<0.05 mg/kg) were detectable in delinted seeds 7-93 days after the last application.

Table 9. Residues from supervised trials on bulb vegetables. Underlined residues are from treatments according to GAP.

Crop Country/year	Application	Sample analysed & maturity	PHI, days	Residue, mg/kg	Ref.

	Form	No	kg ai/ha	kg ai/hl				
Onions, bulb								
Finland/1980		1	5.0		bulbs	108	0.003	52
Germany/1975	EC	1	0.125 g/m	0.025	whole plant	0	8.0	58
					bulbs	16	0.03	
					bulbs	43	<0.02	
	GR	1	60 mg/m		bulbs	100	0.02	58
						114	0.02	
						135	<0.02	
						174	<0.02	
	GR	1	60 mg/m		bulbs	90	0.02	58
						106	0.08	
						133	<0.02	
Germany/1978	GR	1	60 mg/m		whole plant	50	0.12	58
						70	0.02	
					bulbs	134	<0.02	
	GR	1	60 mg/m		whole plant	50	0.19	58
						70	0.06	
					bulbs	104	<0.02	
Switzerl'd/1975	EC	1	0.125 g/m	0.025	bulbs	61	<0.02	58
						76	<0.02	
						91	<0.02	
						106	<0.02	
						122	<0.02	
	EC	1	0.125 g/m	0.025	bulbs	52	0.02	58
						67	<0.02	
						82	<0.02	
						98	<0.02	
						109	<0.02	
	GR	1	60 mg/m		bulbs	61	0.02	58
						76	<0.02	
						91	<0.02	
						106	<0.02	
						123	<0.02	
Switzerl'd/1977	EC	1	0.125 g/m	0.025	bulbs	70	<0.02	58
Switzerland						82	<0.02	
cont.						90	<0.02	
						103	<0.02	
	GR	1	60 mg/m		bulbs	81	<0.02	58
						91	<0.02	

Crop Country/year	Application				Sample analysed & maturity	PHI, days	Residue, mg/kg	Ref.
	Form	No	kg ai/ha	kg ai/hl				
						103	<0.02	
						124	<0.02	
Switzerl'd/1978	GR	1	60 mg/m		whole plant	70	<0.02	58
					bulbs	90	<0.02	
						112	<0.02	
						132	<0.02	
USA/1988	GR+EC	1+3[1]	4.4+0.55		bulbs	11	<0.01	58
						14	<0.01	
	GR+WP	1+3	4.4+0.55		bulbs	11	<0.01	58
						14	<0.01	
	GR+EC	1+3	4.4+0.55		bulbs	10	<0.01	58
						14	<0.01	
	GR+WP	1+3	4.4+0.55		bulbs	10	<0.01	58
						14	<0.01	
	GR+EC	1+3	4.4+0.55		bulbs	10	0.01	58
						14	<0.01	
	GR+WP	1+3	4.4+0.55		bulbs	10	<0.01	58
						14	<0.01	
	GR+EC	1+3	4.4+0.55		bulbs	10	<0.01	58
						14	<0.01	
	GR+WP	1+3	4.4+0.55		bulbs	10	<0.01	58
						14	<0.01	
	GR+EC	1+3	4.4+0.55		bulbs	0	0.81	58
						7	0.09	
						10	0.03	
						14	0.02	
						21	<0.01	
	GR+WP	1+3	4.4+0.55		bulbs	0	1.16	58
						7	0.05	
						10	0.04	
USA cont.						14	0.02	
						21	<0.01	
Spring onions								
USA/1988	GR+EC	1+3	4.4+0.55		whole plant	0	4.80	58
						7	0.71	
					bunch	10	0.56	
						14	0.28	

Crop Country/year	Application				Sample analysed & maturity	PHI, days	Residue, mg/kg	Ref.
	Form	No	kg ai/ha	kg ai/hl				
					mature	21	0.16	
	GR+WP	1+3	4.4+0.55		whole plant	0	6.80	58
						7	0.62	
					bunch	10	0.65	
						14	0.40	
					mature	21	0.11	
	GR+EC	1+3	4.4+0.55		whole plant	10	<0.01	58
						14	<0.01	
	GR+WP	1+3	4.4+0.55		whole plant	10	<0.01	58
						14	<0.01	

USA cont.	GR+EC	1+3	4.4+0.55		whole plant	10	0.01	58
						14	<0.01	
	GR+WP	1+3	4.4+0.55		whole plant	10	0.02	58
						14	<0.01	
USA/1989	GR+EC	1+3	4.4+0.55		whole plant	0	0.05	58
						4	0.03	
	GR+WP	1+3	4.4+0.55		whole plant	0	0.04	58
						4	0.01	

[1] All US trials 1 pre-plant + 3 foliar.

Table 10. Residues from supervised trials on brassica vegetables, all ref. 58. Underlined residues are from treatments according to GAP.

Crop Country/year	Application	Sample analysed & maturity	Residue, mg/kg	PHI, days

	Form	No	kg ai/ha	kg ai/hl			
Broccoli							
USA/1988	GR+EC	1+5[1]	4.4+0.55		bunch[2]	3.1	0
						0.02	7
					mature	<0.01	14
						<0.01	21
	GR+WP	1+5	4.4+0.55		bunch[2]	7.4	0
						0.10	7
					mature	0.01	14
						<0.01	21
	GR+EC	1+5	4.4+0.55		bunch	0.03	3
						0.01	14
					mature	<0.01	21
	GR+WP	1+5	4.4+0.55		bunch	0.02	3
						<0.01	14
					mature	<0.01	21
	GR+EC	1+5	4.4+0.55		bunch	0.23	7
						0.05	14
						<0.01	21
	GR+WP	1+5	4.4+0.55		bunch	0.22	7
						0.1	14
						<0.01	21
	GR+EC	1+5	4.4+0.55		bunch	0.12	7
USA cont.					normal harvest	0.02	14
						0.03	21
	GR+WP	1+5	4.4+0.55		bunch	0.15	7
					normal harvest	0.06	14
						0.02	21
	GR+EC	1+5	4.4+0.55		mature	0.05	7
						<0.01	14
						<0.01	21
	GR+WP	1+5	4.4+0.55		mature	0.01	7
						<0.01	14
						<0.01	21
Cabbage, Head							
Germany/1975	EC	2		0.025	head	2.9	18
						0.6	26
						0.13	39
						0.09	51
	GR	1	0.1g/m		head	0.03	34
						0.05	42

Crop Country/year	Application				Sample analysed & maturity	Residue, mg/kg	PHI, days
	Form	No	kg ai/ha	kg ai/hl			
						<0.02	56
						<0.02	81
	GR	1	0.1g/m		head	0.1	33
						0.03	41
						<0.02	54
						<0.02	66
Germany/1980	EC	2		0.025	head	0.57	-0
						4.61	+0
						0.27	14
						0.15	21
						0.23	42
						0.18	49
Switzerl'd/1974	EC	2		0.025	head	14	0
						0.3	9
						0.05	18
						<0.02	27
						<0.02	37
						<0.02	47
						<0.02	59
Swtzlnd cont.						<0.02	66
Switzerl'd/1975	GR	1	0.1g/m	0.025	leaves	4	0
						1.74	7
						0.11	14
						0.02	21
						0.03	42
					head	<0.02	72
						<0.02	86
	EC	2		0.025	head	0.03	14
						<0.02	28
						<0.02	42
						<0.02	56
USA/1988	GR+EC	1+5	4.4+0.55		head	0.03	7
						0.01	14
						<0.01	21
	GR+WP	1+5	4.4+0.55		head	0.03	7
						0.03	14
						<0.01	21
	EC	1+5	4.4+0.55		head	0.01	7
						0.09	14

Crop Country/year	Application				Sample analysed & maturity	Residue, mg/kg	PHI, days
	Form	No	kg ai/ha	kg ai/hl			
						<0.01	21
	EC+WP	1+5	4.4+0.55		head	0.03	7
						0.04	14
						<0.01	21
	GR+EC	1+5	4.4+0.55		head	0.11	7
						0.08	14
						0.03	21
	GR+WP	1+5	4.4+0.55		head	0.05	7
						0.07	14
						<0.01	21
	EC	1+5	4.4+0.55		head	<0.01	7
						0.03	14
						0.01	21
	EC+WP	1+5	4.4+0.55		head	0.26	7
						0.03	14
						0.02	21
	GR+EC	1+5	4.4+0.55		head	0.78	7
USA cont.						0.02	14
						<0.01	21
	GR+WP	1+5	4.4+0.55		head	1.8	7
						0.02	14
						<0.01	21
	EC	1+5	4.4+0.55		head	0.48	7
						0.08	14
						<0.01	21
	EC+WP	1+5	4.4+0.55		head	0.74	7
						0.11	14
						<0.01	21
	GR+EC	1+5	4.4+0.55		head	1.7	0
						0.07	7
						0.02	13
						0.02	20
	GR+WP	1+5	4.4+0.55		head	1.35	0
						0.14	7
						0.02	13
						<0.01	20
	EC	1+5	4.4+0.55		head	1.4	0
						0.16	7
						0.03	13

Crop Country/year	Application				Sample analysed & maturity	Residue, mg/kg	PHI, days
	Form	No	kg ai/ha	kg ai/hl			
						<0.01	20
	EC+WP	1+5	4.4+0.55		head	2.90	0
						0.15	7
						0.03	13
						0.02	20
	GR+EC	1+5	4.4+0.55		head	1.34	7
						1.13	14
						0.16	21
	GR+WP	1+5	4.4+0.55		head	1.3	7
						0.45	14
						0.13	21
	EC	1+5	4.4+0.55		head	1.12	7
						0.28	14
						0.2	21
	EC+WP	1+5	4.4+0.55		head	1.07	7
USA cont.						0.26	14
						0.09	21
Cauliflower							
Germany/1977	EC	1	20 mg/pl	0.025	whole plant	1.99	0
						0.51	8
						0.05	22
					head	<0.02	36
	EC	2	20 mg/pl	0.025	whole plant	0.75	0
						0.13	14
						<0.02	28
					head	<0.02	42
Germany/1978	GR	1	0.1 g/m row		head	<0.02	111
						<0.02	125
						<0.02	131
	GR	1	0.1 g/m row		head	<0.02	47
						<0.02	61
						<0.02	68
Switzerl'd/1978	GR	4	0.1 g/m row		whole plant	<0.02	33
						<0.02	47
						<0.02	61
Kohlrabi							
Germany/1978	GR	1	0.1 g/m row		roots	<0.02	91
						<0.02	105
						<0.02	113

Crop Country/year	Application				Sample analysed & maturity	Residue, mg/kg	PHI, days
	Form	No	kg ai/ha	kg ai/hl			
	GR	1	0.1 g/m row		roots	<0.02	48
						<0.02	62
						<0.02	69
Germany/1980	EC	1	18.8 mg/pl	0.0235	roots	1.19	0
						0.06	14
						<0.02	21
						<0.02	42
						<0.02	49
						<0.02	56
	EC	2	18.8 mg/pl	0.0235	plant	15	0
						0.12	14
						<0.03	28
					roots	<0.02	42
Germany cont.						<0.03	45
Switzerl'd/1978	GR	1	0.1 g/m row		roots	<0.02	23
						<0.02	37
						<0.02	51
Switzerl'd/1980	EC	2	18.8 mg/pl	0.0235	roots	5.28	0
						<0.02	14
						<0.02	28
						<0.02	35
						<0.02	42
						<0.02	49

[1] All US treatments 1 pre-plant granule + 5 foliar.
[2] 30-50% mature

Table 11. Residues from supervised trials on fruiting vegetables, cucurbits, all ref. 58. Underlined residues are from treatments according to GAP.

Crop Country/year	Application	PHI, days	Residue, mg/kg

	Form	No	kg ai/ha	kg ai/hl		
Cantaloupe						
USA/1988	GR+EC	1	4.4		3	0.08
		+5[1]	0.83		7	0.06
	GR+WP	1	4.4		3	0.15
		+5	0.83		7	0.04
	GR+EC	1	4.4		0	0.14
		+5	0.83		3	0.04
	GR+WP	1	4.4		0	0.06
		+5	0.83		3	0.07
	GR+EC	1	4.4		0	0.01
		+5	0.83		7	0.02
	GR+WP	1	4.4		0	0.02
		+5	0.83		7	0.01
	GR+EC	1	4.4		0	0.17
		+5	0.83		3	0.10
					7	0.05
					14	0.02
	GR+WP	1	4.4		0	0.17
USA cont.		+5	0.83		3	0.11
					7	0.05
					14	0.01
	GR+EC	1	4.4		3	0.21
		+			7	0.08
	GR+WP	1	4.4		3	0.3
		+5	0.83		7	0.09
	GR+EC	1	4.4		3	0.1
		+5	0.83		7	0.02
	GR+WP	1	4.4		3	0.06
		+5	0.83		7	0.01
	GR+EC	1	4.4		3	0.09
		+5	0.83		7	0.18
	GR+WP	1	4.4		3	0.03
		+5	0.83		7	0.11

Crop Country/year	Application				PHI, days	Residue, mg/kg
	Form	No	kg ai/ha	kg ai/hl		
Cucumber						
Japan/1976	EC	3	0.8	0.04	10	0.002
					15	0.002
					20	0.002
	EC	3	0.8	0.04	10	0.012
					15	0.003
					20	0.001
	EC	3	0.8	0.04	10	0.005
					31	0.001
					38	0.001
	EC	3	0.8	0.04	10	0.007
					31	0.001
					38	0.001
Switzerl'd/1972	DP	1	0.8		0	0.38
					5	0.03
USA/1988	GR+EC	1	4.48		3	0.08
		+5	0.84		7	0.01
	GR+WP	1	4.48		3	0.06
		+5	0.84		7	0.01
USA cont.	GR+EC	1	4.48		0	0.84
		+5	0.84		3	0.16
					7	<0.01
					14	<0.01
	GR+WP	1	4.48		0	0.88
		+5	0.84		3	0.11
					7	0.01
					14	<0.01
	GR+EC	1	4.48		3	0.2
		+5	0.84		7	0.4
	GR+WP	1	4.48		3	0.16
		+5	0.84		7	0.02
	GR+EC	1	4.48		3	0.15

Crop Country/year	Application				PHI, days	Residue, mg/kg
	Form	No	kg ai/ha	kg ai/hl		
		+5	0.84		7	0.1
	GR+WP	1	4.48		3	0.06
		+5	0.84		7	0.04
	GR+EC	1	4.48		3	0.05
		+5	0.84		7	0.02
	GR+WP	1	4.48		3	0.03
		+5	0.84		7	<0.01
Summer squash						
USA/1988	GR+EC	1	4.4		0	0.09
		+5	0.83		3	0.02
					7	<0.01
					14	<0.01
	GR+WP	1	4.4		0	0.13
		+5	0.83		3	0.04
					7	0.01
					14	<0.01
	GR+EC	1	4.4		3	0.04
		+5	0.83		14	<0.01
	GR+WP	1	4.4		3	0.07
		+5	0.83		14	0.01
	GR+EC	1	4.4		3	0.04
USA cont.		+5	0.83		7	0.03
	GR+WP	1	4.4		3	0.02
		+5	0.83		7	0.01
	GR+EC	1	4.4		3	0.04
		+5	0.83		7	<0.01
	GR+WP	1	4.4		3	0.03
		+5	0.83		7	<0.01
Zucchini						
USA/1988	GR+EC	1	4.4		3	0.03
		+5	0.83		7	0.01
	GR+WP	1	4.4		3	0.01

Crop Country/year	Application				PHI, days	Residue, mg/kg
	Form	No	kg ai/ha	kg ai/hl		
		+5	0.83		7	<0.01
	GR+EC	1	4.4		3	0.18
		+5	0.83		7	0.03
	GR+WP	1	4.4		3	0.18
		+5	0.83		7	0.05

[1] All US trials with 1 pre-plant granule + 5 foliar.

Table 12. Residues from supervised trials on fruiting vegetables, other than cucurbits. Underlined residues are from treatments according to GAP.

Crop Country/year	Application	Sample analyzed	PHI, days	Residue, mg/kg	Ref.

	Form	No	kg ai/ha	kg ai/hl				
Mushrooms								
Netherl'ds/1969	WP	2	6 g/cell			0	0.09	53
						2	0.07	
	WP	2	6 g/cell			0	0.07	53
						2	0.11	
	WP	2	6 g/cell			0	0.17	53
						2	0.05	
Netherl'ds/1972	GR	1	50g ai/t compost			35	<0.02	58
Pepper (green)								
USA/1988	GR+EC	1	4.48			1	0.13	58
		+5[1]	0.56			3	0.08	
USA cont.	GR+WP	1	4.48			1	0.11	58
		+5	0.56			3	0.03	
	GR+EC	1	4.48			1	<0.01	58
		+5	0.56			4	<0.01	
	GR+WP	1	4.48			1	<0.01	58
		+5	0.56			4	<0.01	
	GR+EC	1	4.48			1	0.09	58
		+5	0.56			3	0.08	
	GR+WP	1	4.48			1	0.03	58
		+5	0.56			3	0.05	
	GR+EC	1	4.48			1	0.08	58
		+5	0.56			3	0.03	
	GR+WP	1	4.48			1	0.08	58
		+5	0.56			3	0.02	
	GR+EC	1	4.48			0	0.52	58
		+5	0.56			1	0.24	
						3	0.09	
						7	0.02	
						14	0.02	
	GR+WP	1	4.48			0	0.53	58
		+5	0.56			1	0.11	
						3	0.06	
						7	0.03	
						14	0.01	
Sweet corn								
Thailand/1988	EC	4	0.647	0.051		0	0.01	51
						1	0.01	

Crop Country/year	Application				Sample analyzed	PHI, days	Residue, mg/kg	Ref.
	Form	No	kg ai/ha	kg ai/hl				
						3	<0.01	
						5	n.d.	
						7	n.d.	
						10	n.d.	
	EC	4	1.294	0.102		0	<0.01	51
						1	0.01	
						3	<0.01	
						5	n.d.	
						7	n.d.	
						10	n.d.	
	EC	4	1.06	0.051		0	<0.01	51
						1	n.d.	
						3	n.d.	
						5	n.d.	
						7	n.d.	
						10	n.d.	
	EC	4	2.12	0.102		0	0.01	51
						1	n.d.	
						3	n.d.	
						5	n.d.	
						7	n.d.	
						10	n.d.	
USA/1988	GR+EC	1	4.4		ears	7	<0.01	58
		+5	1.38			14	<0.01	
					forage	7	1.4	
						14	0.09	
	GR+WP	1	4.4		ears	7	<0.01	58
		+5	1.38			14	<0.01	
					forage	7	0.94	
						14	0.06	
	EC	1	4.4		ears	7	<0.01	58
		+5	1.38			14	<0.01	
					forage	7	0.01	
						14	0.14	
	EC+WP	1	4.4		ears	7	<0.01	58
		+5	1.38			14	<0.01	
					forage	7	0.41	

Crop Country/year	Application				Sample analyzed	PHI, days	Residue, mg/kg	Ref.
	Form	No	kg ai/ha	kg ai/hl				
						14	0.04	
	GR+EC	1	4.4		ears	7	<0.01	58
		+5	1.38			14	<0.01	
					forage	7	2.25	
						14	0.6	
	GR+WP	1	4.4		ears	7	<0.01	58
USSA cont.		+5	1.38			14	<0.01	
					forage	7	1.08	
						14	0.13	
	EC	1	4.4		ears	7	<0.01	58
		+5	1.38			14	<0.01	
					forage	7	1.5	
						14	1.05	
	EC+WP	1	4.4		ears	7	<0.01	58
		+5	1.38			14	<0.01	
					forage	7	0.32	
						14	0.04	
	GR+EC	1	4.4		ears	7	<0.01	58
		+5	1.38			14	<0.01	
					forage	7	0.2	
						14	0.09	
	GR+WP	1	4.4		ears	7	<0.01	58
		+5	1.38			14	<0.01	
					forage	7	0.11	
						14	0.07	
	EC	1	4.4		ears	7	<0.01	58
		+5	1.38			14	<0.01	
					forage	7	0.08	
						14	0.08	
	EC+WP	1	4.4		ears	7	<0.01	58
		+5	1.38			14	<0.01	
					forage	7	0.14	
						14	0.05	
	GR+EC	1	4.4		ears	7	<0.01	58
		+5	1.38			14	<0.01	
					forage	7	0.13	
						14	2.7	

Crop Country/year	Application				Sample analyzed	PHI, days	Residue, mg/kg	Ref.
	Form	No	kg ai/ha	kg ai/hl				
	GR+WP	1	4.4		ears	7	<0.01	58
		+5	1.38			14	<0.01	
					forage	7	0.70	
						14	0.71	
USA cont.	EC	1	4.4		ears	7	<0.01	58
		+5	1.38			14	<0.01	
					forage	7	1.43	
						14	3.95	
	EC+WP	1	4.4		ears	7	<0.01	58
		+5	1.38			14	<0.01	
					forage	7	0.8	
						14	1.8	
	GR+EC	1	4.4		ears	7	<0.01	58
		+5	1.38			14	<0.01	
					forage	7	1.03	
						14	0.38	
	GR+WP	1	4.4		ears	7	<0.01	58
		+5	1.38			14	<0.01	
					forage	7	0.03	
						14	0.16	
	EC	1	4.4		ears	7	<0.01	58
		+5	1.38			14	<0.01	
					forage	7	0.46	
						14	0.22	
	EC+WP	1	4.4		ears	7	<0.01	58
		+5	1.38			14	<0.01	
					forage	7	0.33	
						14	0.12	
	GR+EC	1	4.4		ears	7	<0.01	58
		+5	1.38			14	<0.01	
					forage	7	0.72	
						14	0.25	
	GR+EC	1	4.4		ears	7	<0.01	58
		+5	1.38			14	<0.01	
					forage	7	0.23	
						14	0.01	
	EC	1	4.4		ears	7	<0.01	58

Crop Country/year	Application				Sample analyzed	PHI, days	Residue, mg/kg	Ref.
	Form	No	kg ai/ha	kg ai/hl				
		+5	1.38			14	<0.01	
					forage	7	0.85	
USA cont.						14	0.39	
	EC+WP	1	4.4		ears	7	<0.01	58
		+5	1.38			14	<0.01	
					forage	7	0.29	
						14	0.07	
	GR+EC	1	4.4		ears	7	0.01	58
		+5	1.38			14	<0.01	
					forage	7	7.95	
						14	4.95	
	GR+WP	1	4.4		ears	7	0.01	58
		+5	1.38			14	<0.01	
					forage	7	2.05	
						14	0.97	
	EC	1	4.4		ears	7	0.02	58
		+5	1.38			14	<0.01	
					forage	7	6.6	
						14	2.4	
	EC+WP	1	4.4		ears	7	<0.01	58
		+5	1.38			14	<0.01	
					forage	7	1.7	
						14	0.45	
	GR+EC	1	4.4		ears	7	<0.01	58
		+5	1.38			14	<0.01	
					forage	7	0.43	
						14	0.55	
	GR+WP	1	4.4		ears	7	<0.01	58
		+5	1.38			14	<0.01	
					forage	7	0.17	
						14	0.04	
	EC	1	4.4		ears	7	<0.01	58
		+5	1.38			14	<0.01	
					forage	7	0.16	
						14	0.12	
	EC+WP	1	4.4		ears	7	<0.01	58
		+5	1.38			14	<0.01	

Crop Country/year	Application				Sample analyzed	PHI, days	Residue, mg/kg	Ref.
	Form	No	kg ai/ha	kg ai/hl				
USA cont.					forage	7	0.1	
						14	0.03	
	GR+EC	1	4.4		ears	0	0.02	58
		+5	1.38			7	<0.01	
						14	<0.01	
						21	<0.01	
					forage	0	22	
						7	1.75	
						14	0.4	
						21	0.37	
	GR+WP	1	4.4		ears	0	0.01	58
		+5	1.38			7	<0.01	
						14	<0.01	
						21	<0.01	
					forage	0	14	
						7	0.49	
						14	0.13	
						21	0.18	
	EC	1	4.4		ears	0	0.02	58
		+5	1.38			7	<0.01	
						14	<0.01	
						21	<0.01	
					forage	0	20	
						7	2.25	
						14	0.28	
						21	0.1	
	EC+WP	1	4.4		ears	0	0.03	58
		+5	1.38			7	<0.01	
						14	<0.01	
						21	<0.01	
					forage	0	13	
						7	0.57	
						14	0.12	
						21	0.05	
	GR+EC	1	4.4		ears	7	<0.01	58
USA cont.		+5	1.38			14	<0.01	
					forage	7	2.05	

Crop Country/year	Application				Sample analyzed	PHI, days	Residue, mg/kg	Ref.
	Form	No	kg ai/ha	kg ai/hl				
						14	0.12	
	GR+WP	1	4.4		ears	7	<0.01	58
		+5	1.38			14	<0.01	
					forage	7	0.03	
						14	0.18	
	EC	1	4.4		ears	7	<0.01	58
		+5	1.38			14	<0.01	
					forage	7	0.09	
						14	0.44	
	EC+WP	1	4.4		ears	7	<0.01	58
		+5	1.38			14	<0.01	
					forage	7	0.02	
						14	0.22	
	GR+EC	1	4.4		ears	0	<0.01	58
		+5	1.38			7	<0.01	
						15	<0.01	
						21	<0.01	
					forage	0	4.45	
						7	0.08	
						15	0.03	
						21	0.03	
	GR+WP	1	4.4		ears	0	<0.01	58
		+5	1.38			7	<0.01	
						15	<0.01	
						21	<0.01	
					forage	0	48	
						7	0.18	
						15	0.05	
						21	0.01	
	EC	1	4.4		ears	0	<0.01	58
		+5	1.38			7	<0.01	
						15	<0.01	
						21	<0.01	
USA cont.					forage	0	8.8	
						7	0.25	
						15	0.1	
						21	0.01	

Crop Country/year	Application				Sample analyzed	PHI, days	Residue, mg/kg	Ref.
	Form	No	kg ai/ha	kg ai/hl				
	EC+WP	1	4.4		ears	0	<0.01	58
		+5	1.38			7	<0.01	
						15	<0.01	
						21	<0.01	
					forage	0	14	
						7	0.08	
						15	0.02	
						21	0.03	
	GR+EC	1	4.4		ears	7	<0.01	58
		+5	1.38			14	<0.01	
					forage	7	0.29	
						14	0.33	
	GR+WP	1	4.4		ears	7	<0.01	58
		+5	1.38			14	<0.01	
					forage	7	1.05	
						14	0.05	
	EC	1	4.4		ears	7	<0.01	58
		+5	1.38			14	<0.01	
					forage	7	0.47	
						14	0.97	
	EC+WP	1	4.4		ears	7	<0.01	58
		+5	1.38			14	<0.01	
					forage	7	0.22	
						14	0.12	
Tomatoes								
Canada/1989	EC	1	0.8	0.1		1	0.04	37
						3	0.03	
						7	0.03	
						14	0.07	
						21	<0.005	
						28	<0.005	
Netherl'ds/1973	SP	1	0.191	0.0127	fruit	3	<0.04	53
	SP	1	0.191	0.0127	fruit	3	<0.04	53
USA/1988	GR+EC	1	4.48			1	0.17	58
		+5	0.84			3	0.14	
	GR+WP	1	4.48			1	0.19	58
		+5	0.84			3	0.16	

Crop Country/year	Application				Sample analyzed	PHI, days	Residue, mg/kg	Ref.
	Form	No	kg ai/ha	kg ai/hl				
	EC	1	4.48			1	0.18	58
		+5	0.84			3	0.15	
	EC+WP	1	4.48			1	0.18	58
		+5	0.84			3	0.13	
	GR+EC	1	4.48			1	0.03	58
		+5	0.84			3	0.02	
	GR+WP	1	4.48			1	0.03	58
		+5	0.84			3	0.01	
	EC	1	4.48			1	0.04	58
		+5	0.84			3	0.05	
	GR+EC	1	4.48			1	0.02	58
		+5	0.84			3	0.05	
	GR+WP	1	4.48			1	0.03	58
		+5	0.84			3	0.01	
	EC	1	4.48			1	0.05	58
		+5	0.84			3	0.03	
	EC+WP	1	4.48			1	0.02	58
		+5	0.84			3	0.01	
	GR+EC	1	4.48			1	0.16	58
		+5	0.84			3	0.05	
	GR+WP	1	4.48			1	0.13	58
		+5	0.84			3	0.03	
	EC	1	4.48			1	0.12	58
		+5	0.84			3	0.05	
	EC+WP	1	4.48			1	0.03	58
		+5	0.84			3	0.02	
	GR+EC	1	4.48			1	0.20	58
		+5	0.84			3	0.17	
	GR+WP	1	4.48			1	0.18	58
USA cont.		+5	0.84			3	0.04	
	EC	1	4.48			1	0.48	58
		+5	0.84			3	0.10	
	EC+WP	1	4.48			1	0.15	58
		+5	0.84			3	0.08	
	GR+EC	1	4.48			1	0.08	58
		+5	0.84			3	0.06	
	GR+WP	1	4.48			1	0.19	58

Crop Country/year	Application				Sample analyzed	PHI, days	Residue, mg/kg	Ref.
	Form	No	kg ai/ha	kg ai/hl				
		+5	0.84			3	0.10	
	EC	1	4.48			1	0.02	58
		+5	0.84			3	0.01	
	EC+WP	1	4.48			1	0.03	58
		+5	0.84			3	0.01	
	GR+EC	1	4.48			1	0.22	58
		+5	0.84			3	0.11	
	GR+WP	1	4.48			1	0.15	58
		+5	0.84			3	0.16	
	EC+WP	1	4.48			1	0.12	58
		+5	0.84			3	0.22	
	GR+EC	1	4.48			1	0.05	58
		+5	0.84			2	0.03	
	GR+WP	1	4.48			1	0.04	58
		+5	0.84			2	0.02	
	EC	1	4.48			1	0.07	58
		+5	0.84			2	0.07	
	EC+WP	1	4.48			1	0.08	58
		+5	0.84			2	0.03	
	GR+EC	1	4.48			0	0.20	58
		+5	0.84			1	0.14	
						3	0.08	
						7	0.07	
						14	0.01	
USA cont.	GR+WP	1	4.48			0	0.32	58
		+5	0.84			1	0.10	
						3	0.06	
						7	0.07	
						14	<0.01	
	EC	1	4.48			0	0.30	58
		+5	0.84			1	0.15	
						3	0.10	
						7	0.04	
						14	0.01	
	EC+WP	1	4.48			0	0.22	58
		+5	0.84			1	0.08	
						3	0.07	

Crop Country/year	Application				Sample analyzed	PHI, days	Residue, mg/kg	Ref.
	Form	No	kg ai/ha	kg ai/hl				
						7	0.04	
						14	<0.01	
	GR+WP	1	4.48			1	0.27	58
		+5	0.84			3	0.16	
	EC	1	4.48			1	0.47	58
		+5	0.84			3	0.14	
	EC+WP	1	4.48			1	0.26	58
		+5	0.84			3	0.14	
	GR+EC	1	4.48			1	0.20	36
		+5	0.84			3	0.15	
				Processed fractions:				
					unwashed	1	0.09	
					washed	1	0.15	
					canned	1	<0.01	
					wet pomace	1	2.20	
					dry pomace	1	2.60	
					juice, canned	1	0.03	
					puree	1	0.13	
					paste	1	0.11	
					juice	1	<0.01	
					ketchup	1	0.05	

[1]All US trials 1 pre-plant +5 foliar

Table 13. Residues from supervised trials on leafy vegetables. Underlined residues are from treatments according to GAP.

Crop Country/year	Application	PHI, days	Residue, mg/kg	Ref.

	Form	No	kg ai/ha	kg ai/hl			
Chinese cabbage							
Finland/1987	GR	1	0.2 g/pl		62	0.34	52
	GR	1	0.1 g/pl		62	0.17	52
Thailand/1987	EC	4	0.373	0.06	0	2.91	51
					1	1.97	
					3	1.14	
					5	0.73	
					7	0.31	
					10	0.04	
	EC	4	0.746	0.12	0	4.80	51
					1	3.75	
					3	2.44	
					5	1.34	
					7	0.62	
					10	0.08	
Thailand/1988	EC	4	1.155	0.114	0	7.98	51
					1	1.87	
					3	0.44	
					5	0.04	
					7	0.02	
	EC	4	2.31	0.228	0	16.4	51
					1	5.98	
					3	1.62	
					5	0.03	
Endive							
Netherl'ds/1971	WP	1	0.255	0.0255	2	0.55 head	53
						0.33 washed	
						0.02 cooked	
Kale							
Thailand/1991	EC	5	1.8	0.18	0	27.9	57
					1	11.1	
					3	2.4	
Thailand cont.					5	0.69	
					8	0.11	
					11	0.05	
					14	<0.02	
					17	<0.02	
					21	<0.02	

Crop Country/year	Application				PHI, days	Residue, mg/kg	Ref.
	Form	No	kg ai/ha	kg ai/hl			
	EC	5	3.6	0.36	0	52.3	57
					1	16.9	
					3	5.1	
					5	1.19	
					8	0.27	
					11	0.07	
					14	0.02	
					17	<0.02	
					21	<0.02	
Thailand/1992	EC	5	1.8	0.18	0	29.6	57
					1	16.3	
					3	4.1	
					5	1.07	
					8	0.20	
					11	0.05	
					14	<0.02	
					17	<0.02	
					21	<0.02	
	EC	5	3.6	0.36	0	55.4	57
					1	20.6	
					3	5.7	
					5	1.39	
					8	0.36	
					11	0.11	
					14	0.03	
					17	<0.02	
					21	<0.02	
	EC	5	1.8	0.18	0	32.6	57
					1	14.7	
Thailand cont.					3	3.0	
					5	0.77	
					8	0.13	
					11	0.06	
					14	<0.02	
					17	<0.02	
					21	<0.02	
	EC	5	3.6	0.36	0	60	57

Crop Country/year	Application				PHI, days	Residue, mg/kg	Ref.
	Form	No	kg ai/ha	kg ai/hl			
					1	22	
					3	6.1	
					5	1.7	
					8	0.28	
					11	0.14	
					14	0.05	
					17	<0.02	
					21	<0.02	
Lettuce, Head							
Netherl'ds/1971	WP	1	0.255	0.0255	2	0.32 head	53
						0.21 washed	
Switzerl'd/1973	GR	1	0.2 g/m		70	0.05	58
					76	0.03	
USA/1988	GR+EC	1	4.48		0	3.35	58
		+5[1]	0.56		7	0.23	
					14	0.08	
					21	0.02	
	GR+WP	1	4.48		0	3.2	58
		+5	0.56		7	0.38	
					14	0.15	
					21	0.01	
	EC	1	4.48		0	8.15	58
		+5	0.56		7	0.61	
					14	0.05	
					21	0.12	
	EC+WP	1	4.48		0	11.4	58
		+5	0.56		7	0.34	
USA cont.					14	0.09	
					21	0.08	
	GR+WP	1	4.48		0	26.0	58
		+5	0.56		7	0.60	
					14	0.14	
		(aerial)			21	0.20	
	GR+EC	1	4.48		7	0.12	58
		+5	0.56		14	0.02	
					21	<0.01	
	GR+WP	1	4.48		7	0.10	58

Crop Country/year	Application				PHI, days	Residue, mg/kg	Ref.
	Form	No	kg ai/ha	kg ai/hl			
		+5	0.56		14	0.02	
					21	<0.01	
	EC	1	4.48		7	0.05	58
		+5	0.56		14	0.03	
					21	<0.01	
	EC+WP	1	4.48		7	0.05	58
		+5	0.56		14	0.03	
					21	0.01	
	GR+EC	1	4.48		7	0.26	58
		+5	0.56		14	0.01	
					21	<0.01	
	GR+WP	1	4.48		7	0.22	58
		+5	0.56		14	0.02	
					21	<0.01	
	EC	1	4.48		7	0.28	58
		+5	0.56		14	0.01	
					21	<0.01	
	EC+WP	1	4.48		7	0.44	58
		+5	0.56		14	0.01	
					21	<0.01	
	GR+EC	1	4.48		7	0.39	58
		+5	0.56		14	0.08	
					21	<0.01	
	GR+WP	1	4.48		7	0.05	58
		+5 (aerial)	0.56		14	<0.01	
USA cont.					21	<0.01	
	GR+EC	1	4.48		7	0.22	58
		+5	0.56		14	<0.01	
					21	<0.01	
	GR+WP	1	4.48		7	0.12	58
		+5	0.56		14	<0.01	
					21	<0.01	
	EC	1	4.48		7	0.17	58
		+5	0.56		14	0.03	
					21	<0.01	
	EC+WP	1	4.48		7	0.05	58
		+5	0.56		14	0.03	

Crop Country/year	Application				PHI, days	Residue, mg/kg	Ref.
	Form	No	kg ai/ha	kg ai/hl			
					21	<0.01	
	GR+EC	1	4.48		7	<0.01	58
		+5	0.56		14	0.02	
					21	<0.01	
	GR+WP	1	4.48		7	0.02	58
		+5	0.56		14	<0.01	
					21	<0.01	
	EC	1	4.48		7	0.02	58
		+5	0.56		14	<0.01	
					21	<0.01	
	EC+WP	1	4.48		7	0.04	58
		+5	0.56		14	<0.01	
					21	0.01	
Lettuce, Leaf							
USA/1988	GR+EC	1	4.48		7	0.10	58
		+5	0.56		14	0.12	
					21	<0.01	
	GR+WP	1	4.48		7	0.07	58
		+5	0.56		14	0.08	
					21	<0.01	
	EC	1	4.48		7	0.06	58
		+5	0.56		14	0.07	
					21	<0.01	
USA cont.	EC+WP	1	4.48		7	0.11	58
		+5	0.56		14	0.04	
					21	<0.01	
	GR+EC	1	4.48		7	0.04	58
		+5	0.56		14	0.03	
					21	<0.01	
	GR+WP	1	4.48		7	0.08	58
		+5	0.56		14	0.05	
					21	<0.01	
	EC	1	4.48		7	0.04	58
		+5	0.56		14	0.05	
					21	<0.01	
	EC+WP	1	4.48		7	0.10	58
		+5	0.56		14	0.15	

Crop Country/year	Application				PHI, days	Residue, mg/kg	Ref.
	Form	No	kg ai/ha	kg ai/hl			
					21	<0.01	
	GR+EC	1	4.48		0	34	58
		+5	0.56		7	0.21	
					14	0.10	
					21	0.08	

USA cont.	GR+WP	1	4.48		0	40	58
		+5	0.56		7	0.35	
					14	0.12	
					21	0.12	
	EC	1	4.48		0	42	58
		+5	0.56		7	0.20	
					14	0.11	
					21	0.08	
	EC+WP	1	4.48		0	39	58
		+5	0.56		7	0.34	
					14	0.10	
					21	0.12	
	GR+EC	1	4.48		7	0.25	58
		+5	0.56		14	0.10	
					21	0.03	
	GR+WP	1	4.48		7	0.13	58
		+5	0.56		14	0.03	
					21	0.01	
	EC	1	4.48		7	0.12	58
		+5	0.56		14	0.06	
					21	0.02	
	EC+WP	1	4.48		7	0.09	58
		+5	0.56		14	0.04	
					21	0.02	
	GR+EC	1	4.48		7	0.19	58
		+5	0.56		14	0.03	
					21	0.02	
	GR+WP	1	4.48		7	0.19	58
		+5	0.56		14	0.03	
					21	<0.01	
	EC	1	4.48		7	0.16	58
		+5	0.56		14	0.03	
					21	<0.01	
	EC+WP	1	4.48		7	0.13	58
		+5	0.56		14	0.01	
USA cont.					21	<0.01	
Spinach							
Italy/1988	EC	1	0.3	0.06	0	52	58
					7	0.14	
					14	<0.02	

					21	0.03	
	EC	1	0.36	0.06	0	11	58
					7	0.45	
					14	0.04	
					21	0.02	
					28	<0.02	
Switzerland/1972	EC	1	0.8		0	42	58
					10	0.73	
USA/1988	GR+EC	1	4.48		7	0.01	58
		+5	0.56		14	<0.01	
					21	<0.01	
	GR+WP	1	4.48		7	<0.01	58
		+5	0.56		14	0.01	
					21	<0.01	
	GR+EC	1	4.48		7	0.57	58
		+5	0.56		14	0.16	
					21	0.03	
	GR+WP	1	4.48		7	0.78	58
		+5	0.56		14	0.18	
					21	0.03	
	GR+EC	1	4.48		7	0.02	58
		+5	0.56		14	<0.01	
					21	0.01	
	GR+WP	1	4.48		7	0.02	58
		+5	0.56		14	0.01	
					21	<0.01	
	GR+EC	1	4.48		0	8.9	58
		+5	0.56		7	0.16	
					14	0.06	
					21	0.06	
USA cont.	GR+WP	1	4.48		0	29	58
		+5	0.56		7	0.52	
					14	0.37	
					21	0.02	

[1] All US trials 1 pre-plant + 5 foliar

Table 14. Residues from supervised trials on legume vegetables. Underlined residues are from treatments according to GAP.

Crop Country/year	Application	PHI, days	Residue, mg/kg	Ref.

	Form	No	kg ai/ha	kg ai/hl			
Common beans							
Switzerl'd/1972	DP	1	0.8		0	1.78	58
					10	0.05	
	DP	1	0.8		0	1.04	58
					10	0.05	
	DP	1	0.8		0	1.25	58
					10	0.05	
USA/1988	GR+EC	1	4.4		7	0.01	58
		+3[1]	0.83	0.37	14	<0.01	
	GR+EC	1	4.4		6	0.09	58
		+3	0.83	0.46	13	0.01	
	GR+EC	1	4.4		7	<0.01	58
		+3	0.83	0.11	14	<0.01	
	GR+EC	1	4.4		14	<0.01	58
		+3	0.83	0.37			
	GR+EC	1	4.4		0	0.76	58
		+3	0.83	0.18	7	0.15	
					14	0.04	
					21	0.05	
	GR+WP	1	4.4		7	<0.01	58
		+3	0.83	0.37	14	<0.01	
	GR+WP	1	4.4		6	0.04	58
		+3	0.83	0.46	13	<0.01	
	GR+WP	1	4.4		7	0.01	58
		+3	0.83	0.11	14	<0.01	
USA cont.	GR+WP	1	4.4		14	<0.01	58
		+3	0.83	0.37			
	GR+WP	1	4.4		0	0.50	58
		+3	0.83	0.18	7	0.09	
					14	0.03	
					21	0.03	
	EC	1	4.4	1.95	7	0.03	58
		+3	0.83	0.37	14	<0.01	
	EC	1	4.4	2.4	6	0.11	58
		+3	0.83	0.46	13	0.01	
	EC	1	4.4	1.6	7	0.02	58
		+3	0.83	0.11	14	<0.01	
	EC	1	4.4	0.94	14	<0.01	58

Crop Country/year	Application				PHI, days	Residue, mg/kg	Ref.
	Form	No	kg ai/ha	kg ai/hl			
		+3	0.83	0.37			
	EC	1	4.4	0.96	0	0.63	58
		+3	0.83	0.18	7	0.12	
					14	0.03	
					21	0.04	
	EC	1	4.4	1.95	7	0.03	58
		+3	0.83	0.37	14	<0.01	
	EC	1	4.4	2.4	6	0.04	58
		+3	0.83	0.46	14	<0.01	
	EC	1	4.4	1.6	7	0.01	58
		+3	0.83	0.11	14	<0.01	
	EC	1	4.4	0.94	14	<0.01	58
		+3	0.83	0.37			
	EC	1	4.4	0.96	0	0.66	58
		+3	0.83	0.19	7	0.09	
					14	0.03	
					21	0.03	
Dwarf french beans							
Canada/1988	WP	1	0.5	0.0625	2	0.15 fruit	25
					9	0.02 fruit	
					2	0.75 ends	
					9	0.17 ends	
Yardlong beans							
Thailand/1991	EC	5	3.9	0.18	0	15.9	56
					1	5.2	
					3	0.58	
					5	0.16	
					8	0.05	
					11	0.02	
					14	<0.01	
	EC	5	7.8	0.36	0	23.2	56
					1	8.8	
					3	0.84	
					5	0.21	
					8	0.08	
					11	0.03	
					14	0.01	

Crop Country/year	Application				PHI, days	Residue, mg/kg	Ref.
	Form	No	kg ai/ha	kg ai/hl			
	EC	5	3.9	0.18	0	16.4	56
					1	5.4	
					3	0.67	
					5	0.23	
					8	0.08	
					11	0.03	
					14	0.01	
	EC	5	7.8	0.36	0	26.9	56
					1	10.0	
					3	0.96	
					5	0.31	
					8	0.14	
					11	0.05	
					14	0.02	
Thailand/1992	EC	5	3.9	0.18	0	17.8	56
					1	6.6	
					3	0.61	
					5	0.18	
					8	0.06	
					11	0.03	
Thailand cont.					14	<0.01	
	EC	5	7.8	0.36	0	28.2	56
					1	9.3	
					3	1.11	
					5	0.38	
					8	0.10	
					11	0.04	
					14	0.01	
Peas							
USA/1988	GR+EC	1	4.4		7	0.09	58
		+5	0.83	0.45	14	0.05	
	GR+EC	1	4.4		7	0.10	58
		+5	0.83	0.45	14	0.03	
	GR+EC	1	4.4		7	0.02	58
		+3	0.83	0.11	14	<0.01	
	GR+EC	1	4.4		7	0.03	58
		+3	0.83	0.41	14	0.01	

Crop Country/year	Application				PHI, days	Residue, mg/kg	Ref.
	Form	No	kg ai/ha	kg ai/hl			
	GR+EC	1	4.4		0	1.45	58
		+3	0.83	0.45	7	0.07	
					14	0.09	
					21	0.03	
	GR+WP	1	4.4		7	0.05	58
		+5	0.83	0.45	14	0.03	
	GR+WP	1	4.4		7	0.10	58
		+5	0.83	0.45	14	0.02	
	GR+WP	1	4.4		7	0.01	58
		+3	0.83	0.11	14	<0.01	
	GR+WP	1	4.4		7	0.02	58
		+3	0.83	0.41	14	0.01	
	GR+WP	1	4.4		0	0.64	58
		+3	0.83	0.45	7	0.03	
					14	0.03	
					21	0.01	
	EC	1	4.4	2.4	7	0.07	58
		+5	0.83	0.45	14	0.05	
USA cont.	EC	1	4.4	2.4	7	0.15	58
		+5	0.83	0.45	14	0.04	
	EC	1	4.4	1.6	7	0.02	58
		+3	0.83	0.11	14	<0.01	
	EC	1	4.4	2.2	7	0.03	58
		+3	0.83	0.41	14	<0.01	
	EC	1	4.4	2.2	0	1.1	58
		+3	0.83	0.45	7	0.05	
					14	0.01	
					21	0.02	
	EC+WP	1	4.4	2.4	7	0.05	58
		+5	0.83	0.45	14	0.02	
	EC+WP	1	4.4	2.4	7	0.04	58
		+5	0.83	0.45	14	0.02	
	EC+WP	1	4.4	1.6	7	<0.01	58
		+3	0.83	0.11	14	<0.01	
	EC+WP	1	4.4	2.2	7	0.02	58
		+3	0.83	0.41	14	<0.01	
	EC+WP	1	4.4	2.4	0	0.7	58

Crop Country/year	Application				PHI, days	Residue, mg/kg	Ref.
	Form	No	kg ai/ha	kg ai/hl			
		+3	0.83	0.45	7	0.02	
					14	<0.01	
					21	0.01	

[1] All US trials 1 pre-plant + 3 or 5 foliar

Table 15. Residues from supervised trials on root and tuber vegetables. Underlined residues are from treatments according to GAP.

Crop	Application				No. of trials if >1	Residue, mg/kg	PHI, days	Ref.
Country/year	Form	No	kg ai/ha	kg ai/hl				
Carrots								
Germany/1974	GR	1	0.08 g/m			<0.005	117	55
	GR	1	0.08 g/m			0.02	164	55
Germany/1975	GR	1	0.08 g/m			0.20	90	58
						0.04	106	
						0.03	133	
						<0.02	174	
Netherl'ds/1977	EC	1	3.6	0.72	2	0.07, 0.07	84	53
Netherl'ds/1986	EC	1	5.4	0.54		0.04	98	53
	EC	1	2.7	0.27		0.03	98	53
	EC	1	5.4	0.54		0.06	67	53
	EC	1	2.7	0.27		0.02	67	53
Switzerl'd/1973	GR	1	5.0			0.17	70	58
Switzerl'd/1974	GR	1	5.0		2	0.06, 0.07	110	58
Switzerl'd/1975	GR	1	0.08 g/m			1.08	57	58
						0.10	72	
						<0.02	87	
						<0.02	102	
						<0.02	119	
	EC	1	0.125 g/m			0.13	64	58
						0.03	79	
						<0.02	94	
						<0.02	108	
	EC	1	5.0	0.1		0.10	34	58
USA/1988	GR+EC	1	4.4		4	0.03, 0.41, 0.03, 0.19	7	58
		+5[1]	0.55			0.06, 0.17, 0.07, 0.11	14	
	GR+WP	1	4.4		4	0.03, 0.28, 0.05, 0.11	7	58

Crop	Application				No. of trials if >1	Residue, mg/kg	PHI, days	Ref.
Country/year	Form	No	kg ai/ha	kg ai/hl				
		+5	0.55			0.03, 0.25, 0.06, 0.09	14	
	EC	1	4.4		4	0.02, 0.20, 0.04, 0.13	7	58
		+5	0.55			0.07, 0.25, 0.04, 0.07	14	
	EC+WP	1	4.4		4	0.02, 0.29, 0.05, 0.11	7	58
		+5	0.55			0.03, 0.12, 0.04, 0.04	14	
Celery, tuber analysed								
Netherl'ds/1969	EC	1	3.6	0.36		4.05	27	53
Parsley, turnip rooted, root analysed								
Netherl'ds/1972	EC	1	3.6	0.36		3.49	27	53
Potatoes								
Canada/1975	GR	1	2.25			<0.02	119	58
	GR	1	2.25			<0.02	86	58
Canada/1976	GR	1	2.25			<0.02	129	58
	GR	1	2.25			<0.02	96	58
France/1972	GR	1	10			<0.02	101	58
	GR	1	10			<0.02	122	
USA/1988	GR+EC	1	4.4			<0.01	0	58
		+5	0.55			<0.01	7	
						<0.01	14	
						<0.01	21	
	GR+WP	1	4.4			<0.01	0	58
		+5	0.55			<0.01	7	
						<0.01	14	
						0.01	21	
	EC	1	4.4			<0.01	0	58
		+5	0.55			0.01	7	
						<0.01	14	
						0.01	21	
	EC+WP	1	4.4			<0.01	0	58
		+5	0.55			0.01	7	
						<0.01	14	
						<0.01	21	
	GR+EC	1	4.4		4	<0.01	7	58
		+5	0.55			<0.01	14	
	GR+WP	1	4.4		5	<0.01	7	58

Crop	Application				No. of trials if >1	Residue, mg/kg	PHI, days	Ref.
Country/year	Form	No	kg ai/ha	kg ai/hl				
		+5	0.55			<0.01	14	
	EC	1	4.4		4	<0.01	7	58
	EC+WP	1	4.4		5	<0.01	7	58
		+5	0.55			<0.01	14	

Crop	Application				No. of trials if >1	Residue, mg/kg	PHI, days	Ref.
Country/year	Form	No	kg ai/ha	kg ai/hl				
					Processing:			
	GR+EC	1	4.4		tubers	<0.01	7	34
					" before processing	<0.01	7	
					culls	<0.01	7	
	Processing for chips:				wet peel	<0.01	7	
					dry peel	0.02	7	
					sliced/peeled potato	<0.01	7	
					wash water	<0.01	7	
					potato chips	<0.01	7	
	Processing for flakes:				wet peel	<0.01	7	
					dry peel	0.03	7	
					sliced/peeled potato	<0.01	7	
					wash water	<0.01	7	
					potato flakes	<0.01	7	
Radish, roots analysed								
					No. of trials, if >1			
Switzerl'd/1974	GR	1	8.7			<0.02	33	58
USA/1988	GR+EC	1	4.48		2	<0.01, 0.08	7	58
		+3	0.56			<0.01, 0.03	14	
	GR+WP	1	4.48		2	<0.01, 0.08	7	58
		+3	0.56			<0.01, 0.04	14	
	EC	1	4.48		2	<0.01, 0.04	7	58
		+3	0.56			<0.01, 0.03	14	
	EC+WP	1	4.48		2	<0.01, 0.04	7	58
		+3	0.56			<0.01, 0.07	14	
	GR+EC	1	4.48			0.21	0	58
		+3	0.56			<0.01	7	
						<0.01	14	
						<0.01	21	
	GR+WP	1	4.48			0.08	0	58
		+3	0.56			<0.01	7	
						<0.01	14	
						<0.01	21	
USA cont.	EC	1	4.48			0.12	0	58
		+3	0.56			<0.01	7	

Crop	Application				No. of trials if >1	Residue, mg/kg	PHI, days	Ref.
Country/year	Form	No	kg ai/ha	kg ai/hl				
						<0.01	14	
						<0.01	21	
	EC+WP	1	4.48			0.06	0	58
		+3	0.56			<0.01	7	
						<0.01	14	
						<0.01	21	
	GR+EC	1	4.48			<0.01	7	58
		+4	0.56			<0.01	14	
	GR+WP	1	4.48			<0.01	7	58
		+4	0.56			<0.01	14	
	EC	1	4.48			<0.01	7	58
		+4	0.56			<0.01	14	
	EC+WP	1	4.48			0.01	7	58
		+4				<0.01	14	
Sugar beet					No. of trials if >1 & sample analysed			
Germany/1980	WP	4	0.24	0.04	leaves	3.06	0	58
						<0.02	14-68	
					tubers	<0.02	0	
						<0.02	14	
						<0.02	28-68	
	WP	4	0.24	0.06	leaves	1.78	0	58
						<0.02	14-76	
					tubers	<0.02	0	
						<0.02	14	
						<0.02	28-76	
	EC	4	0.14	0.023	leaves	0.03	0	58
						<0.02	14-68	
					tubers	<0.02	0	
						<0.02	14	
						<0.02	28-68	
	EC	4	0.14	0.035	leaves	1.35	0	58
						<0.02	14-76	
Germany cont.					tubers	0.02	0	
						<0.02	14	
						<0.02	28-76	
Switzerl'd/1975	EC	2	0.5	0.5	whole plant	5.70	0	58

Crop	Application				No. of trials if >1	Residue, mg/kg	PHI, days	Ref.
Country/year	Form	No	kg ai/ha	kg ai/hl				
						0.19	7	
					leaves	0.03	14	
						<0.01	21-113	
					tubers	0.04	14	
						<0.01	21-113	
	EC	2	0.5	0.5	whole plant	6.70	0	58
						0.56	7	
					leaves	0.09	14	
						<0.02	21-105	
					tubers	0.07	14	
						<0.02	21-85	
USA/1988	GR+EC	1	4.4		tops	0.96	7	58
		+4	0.55			0.20	14	
					root	0.02	7	
						0.02	14	
	GR+WP	1	4.4		2 trials	1.49, 0.80	7	58
		+5	0.55			0.41, 0.40	14	
					root	0.03, <0.01	7	
						0.02, <0.01	14	
	EC	1	4.4		3 trials tops	1.60, 1.85, 4.00	7	58
		+5	0.55			0.52, 0.31, 2.51	14	
					root	0.01, 0.01, <0.01	7	
						0.10, <0.01, <0.01	14	
	EC+WP	1	4.4		3 trials tops	1.12, 1.10, 2.05	7	58
		+5	0.55			0.17, 0.40, 0.51	14	
					root	<0.01, 0.02, <0.01	7	
						0.04, <0.01, <0.01	14	
USA cont.	GR+EC	1	5.5		tops	4.40	7	58
		+4	0.55			1.20	14	
					root	0.01	7	
						<0.01	14	
	GR+WP	1	5.5		tops	2.45	7	58
		+5	0.55			0.57	14	
					root	0.01	7	

Crop	Application				No. of trials if >1	Residue, mg/kg	PHI, days	Ref.
Country/year	Form	No	kg ai/ha	kg ai/hl				
						<0.01	14	
				Processed fractions:				
					root	<0.01	7	
					cossettes	<0.01	7	
					molasses	<0.01	7	
					sugar	<0.01	7	
					pulp	0.01	7	

[1] All US trials 1 pre-plant + 3, 4 or 5 foliar

Table 16. Residues from supervised trials on stalk and stesm vegetables.

Crop Country/year	Application				Sample analysed	Residue, mg/kg	PHI, days	Ref.
	Form	No	kg ai/ha	kg ai/hl				
Artichoke								
Spain/1987	EC	1	1.512	0.072	fruit	3.40	0	54
						1.70	3	
						0.96	7	
						0.50	14	
						0.30	21	
	EC	1	1.512	0.072	fruit	3.05	0	54
						1.70	3	
						1.02	7	
						0.76	14	
						0.21	21	
	EC	1	1.512	0.072	fruit	3.40	0	54
						2.70	3	
						1.28	7	
						0.48	14	
						0.18	21	
Witloof Nthlds/1983	TB	1	3.3 g/cell (glass)			<0.01	14	53

Table 17. Residues from supervised trials on cereals.

Crop Country/year	Application				Sample analysed	Residue, mg/kg	PHI, days	Ref.
	Form	No	kg ai/ha	kg ai/hl				
Maize/field corn								
Germany/1974	EC	1	0.94	0.164	plant	<0.02	66	58
						<0.02	97	
					grain	<0.02	142	
					plant	<0.02	142	

Crop Country/year	Application				Sample analysed	Residue, mg/kg	PHI, days	Ref.
	Form	No	kg ai/ha	kg ai/hl				
Germany/1975	DS	1	0.5 kg/		plant	<0.02	85	58
			100 kg		cobs	<0.02	119	
			seed		stems	<0.02	119	
					grain	<0.02	146	
	DS	1	0.5 kg/		plant	<0.02	99	58
			100 kg			<0.02	127	
			seed		grain	<0.02	156	
	DS	1	0.5 kg/		plant	<0.02	80	58
			100 kg		cobs	<0.02	114	
			seed		stems	<0.02	114	
					grain	<0.02	140	
Switzerl'd/1974	EC	1	1	0.1	plant	<0.02	58-120	58
					grain	<0.02	154	
					plant	<0.02	154	
	EC	1	1	0.1	plant	<0.02	58-120	58
					grain	<0.02	148	
					plant	<0.02	148	
USA/1988	GR	4	1.1		forage	3.95	7	58
					silage	5.3	10	
					fodder	3.15	45	
					grain	<0.01	45	
	GR	4	1.1		forage	2.5	7	58
					silage	2.7	10	
					fodder	0.29	43	
					grain	<0.01	43	
	GR	4	1.1		forage	1.07	6	58
					silage	1.18	10	
					fodder	0.39	45	
					grain	<0.01	45	
	GR	4	1.1		forage	1.8	7	46
					silage	5.4	10	
					fodder	1.12	45	
USA cont.					grain	<0.01	45	
				Processed fractions				
				dry milled:				
				kernel, whole		0.01	45	46
				broken		0.04		
				grits, small		0.01		
				meal		0.01		
				flour		<0.01		
				hulls		0.01		
				crude oil expeller		0.01		

Crop Country/year	Application				Sample analysed	Residue, mg/kg	PHI, days	Ref.
	Form	No	kg ai/ha	kg ai/hl				
				presscake solvent		<0.01		
				crude oil solvent		0.02		
				refined oil		<0.01		
				refined oil, bleached		<0.01		
				refined oil, bleached & deodorized		<0.01		
				wet milled:				
				kernel, whole		0.08		
				steepwater concentrate		<0.01		
				hulls		0.05		
				gluten		<0.01		
				starch		<0.01		
				gluten starch		<0.01		
				crude oil expeller		0.08		
				presscake solvent		0.01		
				refined oil		0.14		
				refined oil, bleached		0.06		
				refined oil, bleached & deodorized		<0.01		
Rice								
India/1974	GR	3	1.5		grain	<0.02	59	58
					straw	0.04	59	
India/1975	GR	3	1.0		unhusked grain	<0.02	44	58
					husks	<0.02	44	
	GR	1	1.0		unhusked grain	<0.02	44	58
		+2	1.5		husks	<0.02	44	
	EC	3	0.375	0.075	grain	<0.02	23	58
					straw	0.04	23	
	EC	1	0.125	0.05	grain	<0.02	54	58
		+2	0.15	0.05	straw	0.04	54	
Indonesia/1979	EC	5	0.6	0.12	unhusked grain	<0.02	33	58
Pakistan/1971	GR	1	1.68		unhusked grain	<0.03	45	58

Table 18. Residues from supervised trials on tree nuts in California (USA), 1988. All trials with 1 application at dormant stage + 3 foliar, all at 3.3 kg ai/ha. Underlined residues are from treatments according to GAP. All ref. 58.

Crop Country/year	Application	Sample analysed	Residue, mg/kg	PHI, days

	Form	No	kg ai/hl			
Almonds						
	EC	4	0.35	nuts	<0.01	45
				hulls	0.05	45
	EC+WP	4	0.35	nuts	<0.01	45
				hulls	0.05	45
	WP+EC	4	0.35	nuts	<0.01	45
				hulls	0.05	45
	WP	4	0.35	nuts	<0.01	45
				hulls	0.06	45
	*EC+WP	4	1.77	nuts	<0.01	45
				hulls	0.05	45
	EC	4	0.18	nuts	0.08	0
					0.01	14
					0.01	28
				hulls	16	0
					3.8	14
					4.35	28
	EC+WP	4	0.18	nuts	0.04	0
					0.01	14
					<0.01	28
				hulls	17	0
					3.8	14
					3.5	28
	WP+EC	4	0.18	nuts	0.04	0
					0.03	14
					0.01	28
				hulls	17	0
					5.0	14
					4.35	28
	WP	4	0.18	nuts	0.03	0
					<0.01	14
				hulls	8.5	0
					2.0	14
	EC	4	0.24	nuts	<0.01	45
				hulls	0.09	45
	EC+WP	4	0.24	nuts	<0.01	45
				hulls	0.06	45
	WP+EC	4	0.24	nuts	<0.01	45

Crop Country/year	Application			Sample analysed	Residue, mg/kg	PHI, days
	Form	No	kg ai/hl			
				hulls	0.04	45
	WP	4	0.24	nuts	<0.01	45
				hulls	0.04	45
	*EC+WP	4	1.77	nuts	<0.01	45
				hulls	0.02	45
	EC	1+3	0.33	nuts	<0.01	45
			0.75	hulls	1.05	45
	EC+WP	1+3	0.33	nuts	<0.01	45
			0.75	hulls	0.1	45
	WP+EC	1+3	0.33	nuts	0.01	45
			0.75	hulls	3.10	45
	WP	1+3	0.33	nuts	0.02	45
			0.75	hulls	3.2	45
Walnuts						
	EC	4	0.18	nuts	<0.01	0, 14, 28
					<0.01	45
	EC+WP	4	0.18	nuts	<0.01	0, 14, 28
					<0.01	45
	WP+EC	4	0.18	nuts	<0.01	0, 14, 28
					<0.01	45
	WP	4	0.18	nuts	<0.01	0, 14, 28
					<0.01	45
	*EC	1+3	0.35+1.77	nuts	<0.01	45
	*EC+WP	1+3	0.35+1.77	nuts	<0.01	45
	*WP+EC	1+3	0.35+1.77	nuts	<0.01	45
	*WP	1+3	0.35+1.77	nuts	<0.01	45
	*EC	1+3	0.24+1.77	nuts	<0.01	45
	*EC+WP	1+3	0.24+1.77	nuts	<0.01	45
	*WP+EC	1+3	0.24+1.77	nuts	<0.01	45
	*WP	1+3	0.24+1.77	nuts	<0.01	45
	EC	4	0.24+1.77	nuts	<0.01	45
	EC+WP	4	0.24+1.77	nuts	<0.01	45
	WP+EC	4	0.24+1.77	nuts	<0.01	45
	WP	4	0.24+1.77	nuts	<0.01	45
	EC	1+3	0.15+0.14	nuts	<0.01	45
	EC+WP	1+3	0.15+0.14	nuts	<0.01	45
	WP+EC	1+3	0.15+0.14	nuts	<0.01	45

Crop Country/year	Application			Sample analysed	Residue, mg/kg	PHI, days
	Form	No	kg ai/hl			
	WP	1+3	0.15+0.14	nuts	<0.01	45
	EC	1+3	0.12+1.05	nuts	<0.01	45
	EC+WP	1+3	0.12+1.05	nuts	<0.01	45
	WP+EC	1+3	0.12+1.05	nuts	<0.01	45
	WP	1+3	0.12+1.05	nuts	<0.01	45

*First treatment ground, others aerial

Table 19. Residues from supervised trials on cotton seed in India, 1975. All EC. All ref. 58.

Application			Residue, mg/kg	PHI, days
No	kg ai/ha	kg ai/hl		
2	0.75	0.12	<0.05	7
			<0.05	21
5	0.75		<0.05	93
2	0.40	0.064	<0.05	7
			<0.05	21
5	0.40		<0.05	93

Animal commodities

Cattle. The Canadian government reported the residues in milk and tissues of cattle resulting from applying ear tags to the animals.

Two tags (6% cypermethrin, 11% diazinon), one per ear, were attached to each of three Holstein dairy cows. The animals were maintained on a total mixed ration of corn silage, haylage, high moisture corn, soya bean meal, and mineral supplement. Daily milk production ranged from 12 to 15 kg/cow. Milk samples were taken five hours before application, and five hours, one day, 3, 7, 14, 21, and 28 days after application.

Residues of diazinon were not detectable (<0.0005 mg/kg) in milk samples until three days after tag application. The diazinon residues remaind consistently less than 0.002 mg/kg for the entire residue study. Animals were in good health and no apparent ill effects were observed throughout the trial (Surgeoner ***et al.***, 1987a).

Three Hereford ste ers treated with two ear tags (6% cypermethrin, 9.6% diazinon) were maintained in an indoor/outdoor pen under normal feedlot conditions. After 14 days, one animal was slaughtered and samples of blood, liver, muscle, tongue, omental fat and perirenal fat were taken for analysis; after 100 days the remaining two treated animals were killed and sampled similarly.

Diazinon was found on the hair of the coat, but in the tissues it was detectable only in the omental and perirenal fat of the animal killed days after tag attachement. The levels in the fat were low at 0.032 and 0.035 mg/kg respectively. No residues (< 0.01 mg/kg) were found in the tissues of animals 100 days after treatment, indicating that there was no

accumulation. Analysis of hair samples showed that the concentrations were approximately equal on the neck and side at the end of the season, while slightly less diazinon was found on the face. (Surgeoner ***et al.***, 1987b)

Four Hereford steers were treated with two ear tags (20% diazinon) and maintained in an indoor/outdoor pen under normal feedlot conditions. The steers were slaughtered after 7, 14 and 28 days, (2 at 28 days) and samples of blood, liver, muscle tongue, subcutaneous fat and perirenal fat were taken for analysis.

Diazinon residues were found on the hair of the neck at levels from 11 to 55 mg/kg on all three sample dates, and on the tongue of two animals (less than 0.02 mg/kg) presumably owing to licking. Residues were found in the subcutaneous and perirenal fat of all animals. The highest levels were 0.045 and 0.041 mg/kg respectively on day 14. There were still detectable residues in the subcutaneous and perirenal fat on day 28 at approximately 0.02-0.03 mg/kg. No residues were dectectable (< 0.01 mg/kg) in any other samples. Cholinesterase levels were not affected by the tags. (Surgeoner ***et al.***, 1989)

FATE OF RESIDUES

In animals

Several studies with radiolabelled material on farm animals are now available. Studies with unlabelled active ingredient were also carried out in connection with the registration of diazinon as an animal health product, giving information on residues in animal tissues upon dermal exposure.

The fate of diazinon was studied after oral and t opical administration of unlabelled and radiolabelled diazinon in various mammalian species including the rat, mouse, guinea pig, dog, goat, sheep and cow. Additional ***in vitro*** experiments were conducted using tissue slices or cell fractions from various species.

Absorption. In all species studied, diazinon was rapidly and almost completely absorbed from the intestinal tract. Dermal absorption was also high: in rats, 73 to 81% of a dermally administered dose was recovered in the urine within 72 hours.

Elimination. In rats dosed with [2 -^{14}C]pyrimidine-diazinon, 50% of the label was eliminated within 12 hours. Within 168 hours 69-80% was excreted with the urine and 18-25% with the faeces. 48 hours after the last of ten successive daily oral doses no residual activity could be detected in any of the examined tissues.

Metabolism. Diazinon was readily degraded in all species examined. Various metabolites were identified. In mammals the cleavage of the pyrimidinyl ester bond leading to 4-hydroxy-2 -isopropyl-6-methylpyrimidine (G 27550) was found to be the main primary pathway of metabolism. Subsequent oxidation of the isopropyl substituent yields secondary and tertiary alcohols which may form glucuronide conjugates. It has been demonstrated in the rat that the pyrimidine ring is not cleaved.

Newer studies in rats (Brown and Lai, 1989), goats (Brown and Lai, 1988a), sheep (Barr and Carlin, 1990) and chickens (Brown and Lai, 1988b), which were conducted according to the latest standards, basically confirm these results. Residues of diazinon deposited in tissues were mostly insignificant: if found at all they were present in fat-containing fractions, as were traces of diazoxon (G 24576).

The radioactivity excreted with the milk was low: goats given 4 consecutive doses corresponding to an exaggerated feeding level of 100 mg/kg excreted only 0.31% of the total dose with the milk, with daily

values of approximately 0.3 to 0.5 mg/kg (Brown and Lai, 1988a).

The proposed metabolic pathway in rats, goats and hens is pres ented in Figure 1.

In recent studies diazinon residues were determined in milk, fat and tissues of cattle after exaggerated dermal exposure (spraying with 10 l of a 600 g ai/l solution) to the diazinon animal health product Neocidol 250EC.

Residues had decreased to undetectable (<0.01 mg/kg) in muscle, liver and kidney on day 14 after the treatment, while residues in omental and kidney fat decreased from levels on day 1 of 2.0 and 2.1 mg/kg respectively to 0.16 and 0.06 mg/kg on day 14. After 21 days the corresponding values were <0.01/0.05 and <0.01/0.01 mg/kg (Strong ***et al.***, 1986).

The data indicate a clear decrease of parent residues transported into fatty deposits, thus excluding a potential bioaccumulation of diazinon in these tissues. In another study, residues in milk from animals treated with the same dosage of Neocidol (spraying with 10 l of a 600 g ai/l solution) decreased from an initial average level of 0.22 mg/l measured 7 h after treatment to undetectable (<0.01 mg/l) 80 h after treatment (Bull ***et al.***, 1986).

In plants

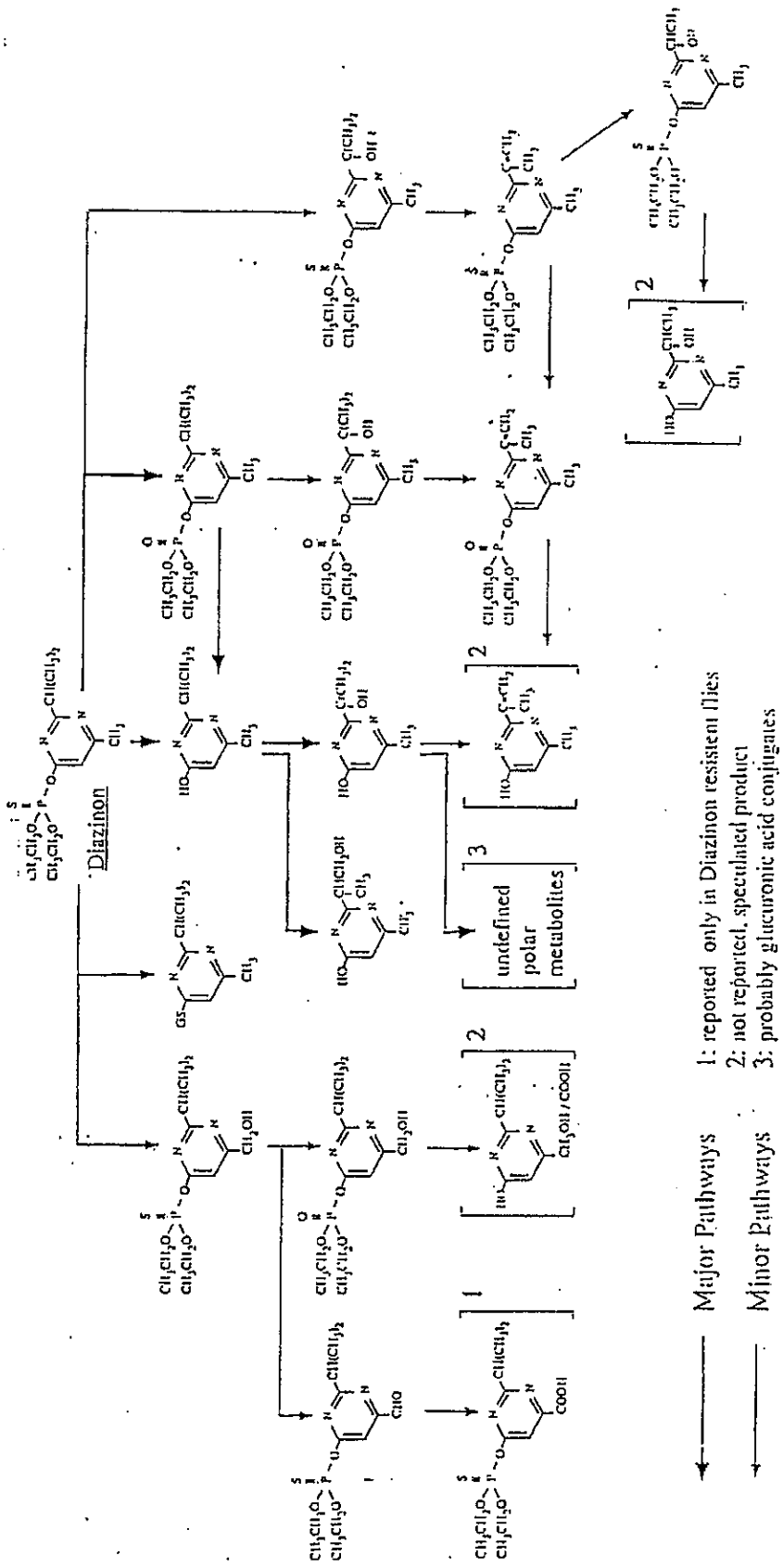
At the time of the FAO evaluation of 1973 little was known of the metabolism of diazinon in plants on the basis of available ^{14}C studies. Since then many plant metabolism and crop rotational studies have successfully addressed the characterization and identification of metabolites owing mainly to advance in methods and instruments. Details of these studies are given below.

Metabolism

Apple. One branch of an Empire hybrid apple tree (10 years old) was sprayed three times with formulated [2 -^{14}C]pyrimidine-diazinon. The initial application was a combined foliar and soil treatment, when the apple buds were in the early tight cluster stage, at a rate equivalent to 3.36 kg ai/ha. The second and third treatments were with foliar sprays when the apples were between 5 and 7.5 cm in diameter, each at a rate equivalent to 10.08 kg ai/ha. Mature apples and apple leaves were harvested 14 days after the last treatment.

Radioactive residues in mature apples and leaves at harvest were 1.29 mg/kg and 51.1 mg/kg diazinon equivalents respectively. The majority of the residue in the fruit was located in the peel (3.44 mg/kg) while the mature pulp contained 0.13 mg/kg diazinon equivalents.

Figure 1. Proposed metabolic pathways of diazinon in mammals.



By harvest, diazinon had been metabolized to a complex mixture of apolar and polar metabolites. Quantitatively, diazinon and G -27550 (4-hydroxy-2-isopropyl-6-methylpyrimidine) constituted the major residues in apples with a combined total of 85% and 77% in the peel and pulp, respectively.

The metabolism of diazinon progressed via hydrolysis of the phosphate ester linkage followed by oxidation steps to primary and tertiary alcohols. Three dihydroxy pyrimidines (C, D, E1; Figure 2) were identified in apple peel and pulp in amounts less than 3% of the total radioactive residues. Further metabolism occurred with the formation of small amounts (<4%) of five conjugates. Less than 15% of the total radioactivity in apple peel and pulp was present as bound non-extractable residues (Wong ***et al.***, 1989b).

A metabolic pathway for diazinon in plants is presented on the basis of the nine metabolites identified (Figure 2). Identical metabolites were also found in beans, maize, lettuce, potatoes and rice.

Beans. Field-grown green beans were treated with a pre -emergent soil application of [2 -^{14}C]pyrimidine-diazinon at 4.48 kg ai/ha and a further two foliar sprays, each at 1.4 kg ai/ha. The half-mature vines were sampled 32 days after planting. An intermediate harvest of vines and beans was taken 7 days after the first foliar spray (42 days after planting) and mature vine and bean samples were taken 14 days after the last application (64 days after planting).

Radioactive residues were 0.425 mg/kg diazinon equivalents in half-mature vines, increased to 4.45 mg/kg 7 days after the first foliar spray, and decreased to 3.53 mg/kg at the mature harvest. Radioactive residues in mature beans (14 days PHI) were 0.46 mg/kg. The extracted radioactivity from mature beans contained 0.01 mg/kg diazinon, while G -27550, GS -31144 and JAK -III-57 (see Figure 2) accounted for 0.12, 0.03 and 0.02 mg/kg respectively. A mixture of an additional dihydroxy metabolite, CL -XIX-29, and a glucose conjugate of GS -31144 represented 7.0% of the total radioactivity (0.03 mg/kg).

The half-mature samples and mature vines contained a mixture of metabolites qualitatively similar to those found in mature beans.

The metabolic pathway of diazinon in green beans is identical to that in apples (Wong and McFarland, 1990a).

Maize. Sweet corn grown in a greenhouse was treated with [2-^{14}C]pyrimidine-diazinon, once with pre -emergent (4.48 kg ai/ha) and twice with foliar sprays (3.5 kg ai/ha). Plant samples were taken at 50% maturity, just before the second foliar spray, and at 100% maturity.

The total radioactive residue increased from 0.810 mg/kg expressed as diazinon in 50% mature stalks to 2.07 mg/kg in immature forage and 3.89 mg/kg in mature forage. Mature cobs and grain contained 0.25 mg/kg and 0.45 mg/kg. Only 20% of the radioactivity was extractable from 50% mature stalks, and 74% from mature forage of which 77% partitioned into the aqueous phase and 23% into the organic phase. The mature grain was 26% extractable, of which 95% partitioned into the aqueous phase and 5% into the organic phase.

Diazinon accounted for 1.8% (0.07 mg/kg), G -27550 for 14.5% (0.56 mg/kg), GS-31144 for 3.0% (0.12 mg/kg) and JAK-III-57 for 5.6% (0.22 mg/kg) of the total radioactivity in mature corn forage. A mixture of an additional dihydroxy metabolite, CL -XIX-29, and a glucose conjugate of GS-31144 represented 4.0% (0.16 mg/kg) in mature forage. Diazinon was metabolized to a complex mixture of apolar and polar metabolites. The metabolism progressed via hydrolysis of the phosphate ester linkage followed by oxidation steps to primary and secondary alcohols. The metabolites in sweet corn were qualitatively the same as those in apples and potatoes (Rezaaiyan ***et al.***, 1989).

Lettuce. A field plot planted with lettuce was treated with a pre -emergent soil application of [2 -^{14}C]pyrimidine-diazinon at 4.48 kg ai/ha and two foliar sprays, each at 1.4 kg ai/ha. Immature leaves were harvested prior to the second foliar spray and mature leaves 14 days after the last application.

Radioactive residues in immature and mature leaves were 1.89 mg/kg and 0.66 mg/kg, respectively. The extractable proportions in the two samples were 87% and 78%, of which 54% and 70% respectively partitioned into the aqueous phase and 46% and 30% into the organic phase. In the mature leaves diazinon accounted for 11.8% (0.08 mg/kg), G -27550 for 17.5% (0.12 mg/kg), GS -31144 for 11.7% (0.08 mg/kg), and JAK -III-57 for 1.3% (0.01 mg/kg) of the total radioactivity. A mixture of the dihydroxy metabolite CL-XIX-29 and a glucose conjugate of GS -31144 represented 3.0% (0.02 mg/kg), while an additional dihydroxy and two resolved trihydroxy glucose conjugates represented 9.4% (0.06 mg/kg), 6.7% (0.04 mg/kg) and 2.1% (0.01 mg/kg) of the total radioactivity in mature leaves respectively. The metabolic pathway of diazinon in lettuce leaves is similar to that in apples (Wong and McFarland, 1990b).

Potatoes. Field-grown potatoes were treated in a similar manner to lettuce with [2 -^{14}C]pyrimidine-diazinon. Immature foliage and tubers were sampled before the first foliar spray and mature foliage and tubers were harvested 15 days after the last application.

Radioactive residues in foliage increased to 1.93 mg/kg at harvest while mature tuber residues reached 0.275 mg/kg. 82% of the mature foliage residue was extractable, with 25% partitioning into the organic phase and 75% into the aqueous phase. The mature tuber residue was 16% extractable with only 8% partitioning into the organic phase and 95% into the acqueous phase. Obviously the residues which were translocated into the tubers were extensively metabolized.

In mature potato foliage diazinon accounted for 14.2% (0.27 mg/kg), G-27550 for 1.3% (0.03 mg/kg), GS -31144 for 2.1% (0.04 mg/kg), JAK -III-57 for 4.3% (0.08 mg/kg), and a mixture of CL -XIX-29 and a glucose conj ugate of GS-31144 for 11.5% (0.22 mg/kg) of the total radioactivity. A mixture of additional dihydroxy and trihydroxy glucose conjugates represented 20.8% (0.4 mg/kg) and 14.1% (0.27 mg/kg) respectively.

Unextractable residues accounted for 84.2% (0.23 m g/kg) of the total radioactivity in mature tubers. These bound (unextractable with neutral solvents) residues were combined with those from maize grain (73.6% unextractable, 0.33 mg/kg) for characterization. Acetylation and acid hydrolysis indicated that these unextractable residues were also a complex mixture of sugar conjugates (Wong ***et al.***, 1989a).

Rice. The uptake of radioactivity and its distribution within the rice plant were followed after one and two applications of [2-^{14}C]pyrimidine-diazinon to th e paddy water. The results demonstrated that diazinon was rapidly absorbed by and translocated in rice plants. Less than 10% of the radioactivity remaining in the plants after 9 days was from the parent compound. About 50% of the applied radioactivity was lost within 5 days owing to volatilization from paddy water and transpiration from the leaves. A parallel experiment was also run with pea plants. The types of metabolites and the metabolic pathways in rice and peas were similar to those shown in all other crops examined (apples, beans, maize, lettuce). (Laanio ***et al.***, 1972).

Rotational crops. A total of five studies on apples, green beans, maize, lettuce and potatoes were conducted to determine the similarity of metabolites and metabolic pathways of diazinon in the target crops and to facilitate the identification of specific metabolites common to all of them. The soil from the maize study was subsequently used in a greenhouse rotational crop study while the green bean, lettuce and potato field plots

were subsequently used in a field rotational crop study.

In the greenhouse rotational crop study, rotational crops spring wheat, lettuce, sugar beet and soya beans were grown in soil which had been previously used for growing maize. The radioactive residues in mature rotational plant samples corresponded to 0.24 mg/kg in wheat grain, 0.038 mg/kg in lettuce, 0.016 mg/kg in sugar beet and 0.19 mg/kg in soya beans. The highest residues in the rotational crops were detected in mature spring wheat stalks (0.62 mg/kg). Diazinon was rapidly metabolized by the rotational crops in a similar manner as in the target crop (Rezaaiyan and McFarland, 1990).

In the field rotational study, winter wheat, lettuce, sugar beet and soya beans were grown as rotational crops in soil which had been previously used for growing green beans, lettuce and potato as target crops. Radioactive residues in the rotational crops were <0.001–0.027 mg/kg, about 1/10 of the residues in the greenhouse rotational crops (Sobralske ***et al.***, 1990).

In soil

Degradation. The degradation of diazinon in soil was investigated under laboratory conditions using ^{14}C-ring-labelled diazinon. The parent compound is very rapidly degraded under laboratory conditions (25 °C), with a half-life of approximately 11 days, to the major (transient) product 4-hydroxy-2-isopropyl-6-methylpyrimidine (G 27550) and a series of related compounds oxidized at the alkyl groups. The products are further degraded by breakdown of the pyrimidine ring with the formation of $^{14}CO_2$ in quantities >55% of the activity after 166 days (Keller, 1981).

In a study conducted at 10 °C diazinon was degraded with a half-life of 3–5 and 16 weeks in a loam soil and a humic sandy soil, respectively (Vonk and de Jong, 1987).

Mobility. Diazinon is fairly strongly adsorbed to soil particles, as shown by a laboratory study. An average adsorption constant K_{om} = 332 mg/g organic matter was determined. The value indicates the relatively low mobility of the insecticide in soil (Laanio ***et al.***, 1972; Guth, 1972). This was confirmed in leaching studies using soil columns. After 200 mm of simulated rainfall within 48 hours the compound penetrated 2, 4, 8 and 16 cm into a sandy loam, silty loam, loamy sand and sandy soil, respectively. On the basis of these results a relative mobility factor RMF = 0.28 was determined (monuron as standard compound with RMF = 1.0), indicating that diazinon has a low mobility (Guth, 1978).

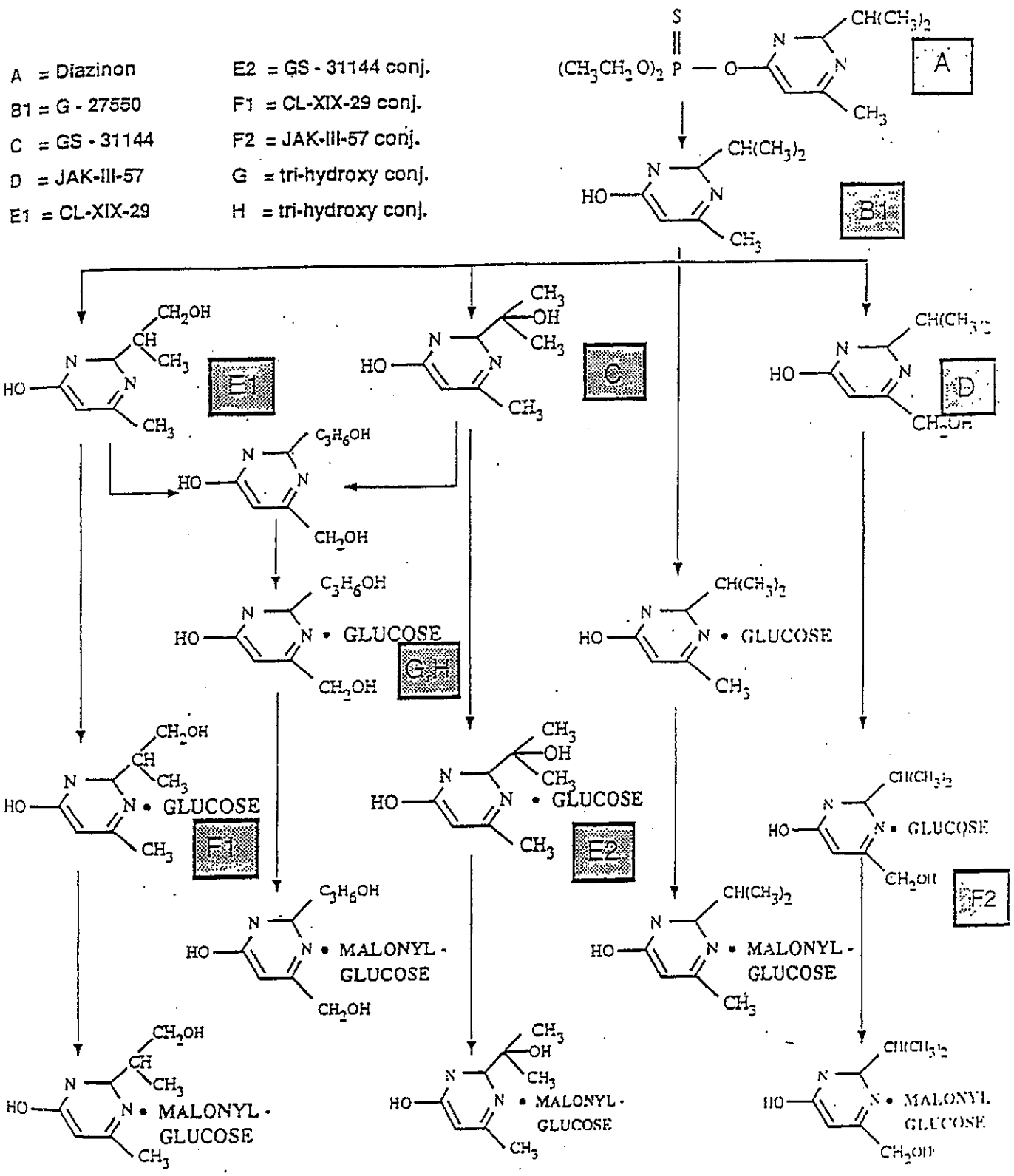
In a similar study using extreme rain conditions (508 mm within approximately 24 hours), 6, 16, 18 and 81% of the applied radioactivity was found in the leachate of silt loam, clay, sandy loam and sand soils respectively. Characterization showed that 83–94% of the radioactivity in the eluate was the pyrimidinol G 27550. The majority of radioactivity in the silt loam remained in the top 5, 7.5 and 22.5 cm of the soil column, respectively (Spare, 1987).

Leaching. The leaching characteristics of aged residues of ^{14}C-labelled diazinon were studied in a sandy loam, silty loam and sandy soil. After 30 days ageing followed by a period of 45 days with 571 mm artificial rain the leachates and soil layers were analysed. Approximately 32 and 25% of the applied radioactivity were found in the eluates of the sand and silty loam soil columns with approximately one third being the major soil degradation product G 27550 and half tentatively identified as the 2-ethyl derivative. No parent was detectable (Burkhard, 1980).

After a longer ageing period of 84 days in sandy loam and a raining period of 16 days (200 mm rain) approximately 31% of the applied radioactivity was found in the eluate, consisting almost exclusively of the major soil product G 27550 (Keller, 1983).

Photodegradation. The photodegradation of diazinon was investigated under natural and artificial sunlight. Half-lives corresponding to 17.3 hours and 5.5 days respectively were determined for these light conditions at temperatures ranging between 16 and 31 °C (Mortison, 1985). In an earlier study 72% and 77% breakdown of diazinon was found within 24 h in moist and dry soil respectively. The major breakdown product was identified as G 27550 (Burkhard, 1978).

Figure 2 . Metabolic pathways of diazinon in plants.



Volatilization. The volatilization of diazinon from soil surfaces is moderate. During the first 24 hours after application, 3.0 -10.4% of the applied product was calculated to be volatilized in various soil types (Burkhard, 1977).

Field dissipation . Field dissipation studies on different soil types confirmed the rapid degradation observed in the laboratory tests. The field half-life was approximately one week. No carry-over of residues was found in repeated treatments (Spare, 1987).

In processing

Processed fractions were prepared from a number of crops: apples, grapes, maize, pineapples, potatoes, sugar beet and tomatoes. Most of the samples were analyzed for diazinon and the two metabolites diazoxon (G -24576) and hydroxydiazinon (CGA 14128). Wine was prepared in some cases from harvest grapes and olive oil (crude) was prepared from olives in one trial. No measurable metabolite residues were found in these fractions.

Apples. Apples containing 0.98 mg diazinon/kg immediately after treatment at the normal recommended rate were processed into culls, wet pomace, dry pomace, fresh juice, canned juice, canned slices, frozen slices, and apple sauce. The results are shown in Table 3 (Ross and Gold, 1989a).

No residues were detectable in canned juice, canned slices, frozen slices or apple sauce, and only a low residue in the fresh juice (0.02 mg/kg). While residues were reduced by a factor of about 2.5 in dry pomace (0.4 mg/kg), an accumulation was observed in culls (2.2 mg/kg) and wet pomace (1.4 mg/kg).

Grapes. Grapes containing 0.06 mg diazinon/kg at harvest 7 days after treatment at the normal recommended rate were processed into wash water, stems, destemmed grapes, wet pomace, dry pomace, unclarified juice, filter cake, "Agrol" settling, clarified juice and canned juice (Gold, 1990a). See Table 5.

With the exception of pomace, residues in the processed fractions did not exceed the residues in grapes (wash water <0.01 mg/kg; stems 0.04 mg/kg; destemmed grapes 0.06 mg/kg; unclarified juice 0.02 mg/kg; filter cake <0.01 mg/kg; "Agrol" settling <0.01 mg/kg; clarified juice <0.01 mg/kg; canned juice <0.01 mg/kg). In wet pomace residues accumulated to a level of 0.13 mg/kg with further accumulation by drying up to 0.36 mg/kg in dry pomace.

In several cases wine was produced from harvest grapes (Table 5). Diazinon residues were not detectable in any of the analysed samples (<0.02 or <0.001 mg/l).

Leafy vegetables . The Netherlands government reported the results of washing head lettuce and endive (Table 13). Residues of 0.32 mg/kg in lettuce and 0.55 mg/kg in endive two days after application were reduced by washing by one third to 0.21 and 0.33 mg/kg respectively. Cooking reduced the residue further to 0.02 mg/kg (Ministry of Welfare, Health, and Cultural Affairs, 1993).

Maize/corn. Whole kernels from harvest samples after treatment at the normal recommended rate were processed by dry and wet milling. Whole kernels, broken kernels, grits, meal, flour, hulls, presscake solvent, oil (crude, refined, and refined, bleached and deodorized) and starch were analysed (Gold, 1990b, Table 17).

In the dry milling process residues in most fractions were at about the limit of determination (whole kernel 0.01 mg/kg; grits 0.01 mg/kg; meal 0.01 mg/kg; flour <0.01 mg/kg; hulls 0.01 mg/kg; presscake solvent <0.01 mg/kg) with a higher value in the broken kernels (0.04 mg/kg). Residues in oil were reduced from 0.02 to <0.01 mg/kg in the course of processing from

crude to refined, to refined and bleached, to refined, bleached and deodorized.

In the wet milling process the residue in the whole kernels was somewhat higher, reaching a level of 0.08 mg/kg. In hulls the residue was 0.05 mg/kg, in starch <0.01 mg/kg, and in presscake solvent 0.01 mg/kg. Residues in oil decreased from 0.14 to <0.01 mg/kg in the course of processing as above.

Commercial grade oil fit for human consumption contained no measurable residues.

Olives (Table 8). The government of Portugal reported that residues in whole fruit of 0.56 -3.0 mg/kg were increased by processing into crude oil to 1.7-9.0 mg/kg (Ministério dos Negócios Estrangeiros, 1990).

Whole olive fruit at harvest with little or no residue (<0.02 and 0.07 mg/kg) were in one case processed into crude oil containing 0.10 and 0.29 mg/kg respectively (Altenburger, 1976b).

Evidently processing olives into crude oil results in an accumulation of diazinon by a factor of 3-5.

Residues of 0.03 and 0.09 mg/kg in olives showed no decline after preservation (0.04 and 0.09 mg/kg) (Altenburger, 1976a).

Pineapples. Pineapples containing undetectable or trace residues (<0.02-0.07 mg/kg) in whole fruit harvest samples after treatment at the normal recommended rate were processed into juice and filter cake/bran (Kühne-Thu, 1989b,c, 1991, Table 8). Residues were undetectable in the juice (<0.02 or <0.03 mg/kg) and low in the filter cake/bran (<0.02 -0.03 mg/kg).

Potatoes. Tubers containing no residues (<0.01 mg/kg) at harvest after treatment at the normal recommended rate were processed into culls, wet peel, potato sliced and peeled, wash water, chips, and flakes, all with no detectable residues (<0.01 mg/kg). The only diazinon residues detected were in dry peel: 0.02 mg/kg (chips) and 0.03 mg/kg (flakes) (Ross and Gold, 1989b, Table 15).

Sugar beet . Sugar beet roots containing no residues (<0.01 mg/kg) in harvest samples after treatment at the normal recommended rate were processed (Table 15). Cossettes, molasses, and sugar contained no detectable residues (<0.01 mg/kg). The only diazinon residues detected were in pulp at 0.01 mg/kg (Ross and Gold, 1989c).

Tomatoes. Tomatoes containing 0.09 mg diazinon/kg in unwashed harvest samples after treatment at the normal recommended rate were processed (Table 12). Residues of the same order or lower were found in washed tomatoes (0.15 mg/kg), canned tomatoes (<0.01 mg/kg), canned juice (0.03 mg/kg), puree (0.13 mg/kg), paste (0.11 mg/kg), juice (<0.01 mg/kg), and ketchup (0.05 mg/kg). Residues were higher in pomace, reaching levels of 2.2 mg/kg in wet pomace and 2.6 mg/kg in dry pomace mg/kg (Ross and Gold, 1989d).

Generally residues of diazinon are reduced or not detectable in processed commodities of importance for human consumption, namely flour, juices, sugar, commercial grade oils and wine.

A concentration of residues was observed in crude olive oil and in pomace-type fractions, the latter having a potential use for animal feed.

Stability of pesticide residues in stored analytical samples

The stability of residues of diazinon and the metabolites diazoxon (G-24576) and hydroxydiazinon (CGA 14128) under freezer storage conditions was checked in a variety of crop substrates and processed commodities , namely maize grain, tomatoes, potatoes, apples, strawberries, lettuce, soya beans (dry), refined corn oil, tomato paste and sugar beet molasses.

Residues of diazinon are generally stable in crops and processed commodities for a minimum of 26 months of freezer storage. A slight decline was observed in strawberries after three months. This decline continued at a much slower rate through 26 months.

Residues of G -24576 are unstable in crop and processed fraction substrates, but are stable in corn oil.

Residues of CGA -14128 are generally stable in crop substrates and processed fractions. A decline in residues was observed in apples and strawberries after three months of storage and the decline continued at a much slower rate through twenty-six months (Beidler and Moore, 1991).

The stablity of diazinon under freezer storage conditions was further tested in the muscle, liver, kidney and fat of sheep. It was found to be stable for at least 8 months of storage (Schnabel, 1981).

Residues in the edible portion of food commodities

No data about the partition of residues between pulp and peel were available for citrus fruit, pineapple, or cantaloupe. No residues were detectable in the whole fruit, pulp or peel after applying diazinon to bananas.

When diazinon was applied to walnuts only the whole nuts were analysed. For almonds residues in the whole nuts and the hulls but not in the meal were available. Residues in the whole nuts were low, <0.01 -0.01 mg/kg, while the residues in the hulls were 0.02 -4.35 mg/kg 28 -45 days after the last application. From these results it seemed rather unlikely that residues could occur in the meal.

METHODS OF RESIDUE ANALYSIS

Methods for the determination of diazinon residues were described in the 1967 and 1970 monographs. Since then, basically all methods developed for diazinon employ gas chromatography using the very sensitive phosphorus/nitrogen-specific thermionic detector or in some cases the phosphorus-specific flame-photometric detector. Devices used during the early 1970s such as conductivity (microcoulometric) detectors or the Coulson detector were abandoned.

With t he advent of microbore and capillary columns for gas chromatography it became possible to co-determine the metabolites diazoxon (G 24576) and hydroxydiazinon (CGA 14128) by this technique, and the formerly used thin-layer chromatography with detection by cholinesterase inhibition for the determination of diazoxon was no longer applied. More recent improvements in laboratory equipment (e.g. cartridges for clean-up purposes) led to miniaturization of the methods used.

Methods for diazinon with sufficiently low limits of determination (0.02 mg/kg or lower) are available for practically all crops, processed commodities, animal tissues, human and animal body fluids, water and soil. Many are published in the literature. Diazinon is also generally determined by most of the available and published multi-residue methods.

In the older non-US trials reported here analyses were mostly by the

CIBA-GEIGY internal methods REM 7a/73 (Blass, 1973) and REM 15/82 Appendix 2 (Hohl, 1983), while more recently the method REM 119.01 (Kühne -Thu, 1989a) was used. In trials conducted in the USA samples were generally analyzed by method AG-550A (Hubbard and Todt, 1990).

Method REM 7a/73 (Blass, 1973) employs gas chromatography with a flame-ionization detector. Recoveries from apples, lettuce, and leaves of beans at levels of 0.2 and 1.0 mg/kg were 88-102% and 84-105% respectively. The limit of determination was assessed to be 0.02 mg/kg.

Method REM 15/82 Appendix 2 (Hohl, 1983) used an NP detector instead of a flame-ionization detector. The method was validated for cherries, lettuce and whole cacao seeds. When adding 0.05 and 0.5 mg/kg recoveries were 82 -98% and 91 -113% respectively. The limit of determination was assessed to be 0.02 mg/kg.

Method REM 119.01 (Kühne -Thu, 1989a) described the determination of several active ingredients using gas chromatography with either an NP or an electron-capture detector. Reported recoveries for kiwifruit and maize (whole plant) were:

kiwi/NPD: 77% (4.0 mg/kg), 80% (0.4 mg/kg), 77% (0.04 mg/kg),
kiwi/ECD: 89% (4.0 mg/kg), 110% (0.4 mg/kg), and
maize/NPD: 97% (4.0 mg/kg), 107% (0.4 mg/kg), 103% (0.04 mg/kg).

The limit of determination was assessed to be 0.01 mg/kg.

With method AG-550A (Hubbard and Todt, 1990) residues of diaz oxon (G 24576) and hydroxydiazinon (CGA 14128) as well as diazinon were determined using capillary column gas chromatography with a flame-photometric detector. The method was validated for 21 crops, almond meat and hulls, and corn oil products at levels of 0.01 and 0.05 mg/kg, for hops at 0.05 mg/kg, and for five animal tissue substrates at the US tolerance level or 1.0 mg/kg. Reported recoveries were in the range of 70 -120% for each of the substances in crops and animal tissues except diazinon in maize (128.2%) and lettuce (124.7%), diazoxon in hops (53.5%), beef liver (68.2%), beef fat (124.7%) and poultry eggs (123.2%), and hydroxydiazinon in lettuce (121.2%). With the exception of hops a limit of determination of 0.01 mg/kg was estimated for diazinon.

NATIONAL MAXIMUM RESIDUE LIMITS

The national MRLs reported below have been established on the basis of local conditions and requirements. Most of them were adopted some time ago. They all refer to the active ingredient diazinon. The Table may not completely cover all countries or commodities with officially approved MRLs.

Country	Commodity	MRL	Remarks
		mg/kg	
Australia	eggs	0.05	
	edible offal	0.7	
	meat	0.7	residues in fat
	poultry meat	0.05	
	milk	0.5	fat basis
	milk products	0.5	fat basis
	cereals	0.1	

Country	Commodity	MRL	Remarks
		mg/kg	
	citrus fruit	0.7	
	sweet corn	0.7	
	all other fruits	0.5	
	kiwi-fruit	0.5	
	nuts	0.1	
	olives, olive oil	2	
	peaches	0.7	
	sugar cane	0.5	
	vegetables	0.7	
	vegetable oil	0.1	except olive oil
Belgium	cereals	0.05	
	fruit	0.5	
	vegetables	0.5	
Brazil	beans	0.5	
	citrus fruit	0.5	
	coffee	0.05	
Brazil cont.	corn/maize	0.7	
	cotton seed	0.5	
	cucurbits	0.5	
	fruit	0.5	
	onion	0.5	
	peas	0.5	
	peanuts	0.5	
	pecan	0.5	
	rice	0.1	
	soya beans	0.5	
	sugar cane	0.7	
	tomatoes	0.5	
	vegetables, root/tuber	0.5	
	vegetables,leafy	0.7	
	wheat	0.1	
Canada	beans	0.5	
	broccoli	0.75	
	Brussels sprouts	0.5	

Country	Commodity	MRL	Remarks
		mg/kg	
	cabbage	0.75	
	carrot	0.75	
	cauliflower	0.75	
	celery	0.75	
	chard	0.25	
	collards	0.25	
	cranberry	0.25	
	cucumber	0.5	
	grapes	0.75	
	hops	0.25	
	parsley	0.25	
	kale	0.75	
	kohlrabi	0.75	
	lettuce	0.75	
	lima bean	0.25	
	onion	0.75	
	parsley	0.25	
	parsnip	0.25	
Canada cont.	peaches	0.7	
	peppers	0.75	
	pome fruit	0.75	
	radish	0.25	
	salsify	0.75	
	spinach	0.75	
	squash	0.25	
	stone fruit	0.75	except peaches
	strawberry	0.75	
	sugar beet	0.75	
	tomatoes	0.75	
	turnip	0.5	
	vegetables, root/tuber	0.5	except parsnips, radish
	vegetables, leafy	0.75	except chard
Denmark	carrots	0.5	
	cereals	0.05	

Country	Commodity	MRL	Remarks
		mg/kg	
	fruit	0.5	
	nuts	0.1	
	vegetables	0.5	
	olive oil	2	
France	asparagus	0.5	
	cabbage	0.5	
	carrots	0.5	
	cherry	0.5	
	corn (maize)	0.5	
	grapes	0.5	
	leek	0.5	
	lettuce	0.5	
	nuts	0.05	
	olives	0.5	
	onions	0.5	
	peaches	0.5	
	pome fruit	0.5	
Germany	fruits	0.5	
	vegetables	0.5	
	other plant products	0.05	
Israel	meat	0.7	
	apple	0.5	
	avocado	0.5	
	banana	0.5	
	citrus fruits	0.7	
	cole crops	0.5	
	corn/maize	0.7	
	eggplant	0.5	
	grapes	0.7	
	olives/olive oil	2	
	peanut	0.5	
	peppers	0.5	
	potatoes	0.5	
	safflower seed	0.5	

Country	Commodity	MRL	Remarks
		mg/kg	
	stone fruit	0.5	
	sugar beet	0.5	
	sunflower seeds	0.5	
	tomatoes	0.5	
	vegetable	0.5	
Italy	cereals	0.05	
	citrus fruit	0.5	
	corn (maize)	0.05	
	fruit	0.5	
	grapes	0.5	
	nuts	0.05	
	olives	0.5	
	potatoes	0.1	
	rice	0.05	
	sorghum	0.1	
	strawberries	0.5	
	sugar beet	0.1	
	sunflowers	0.5	
	tobacco	0.5	
Japan	cereals	0.1	
	fruit	0.1	
	pulses	0.1	
Japan cont.	sugar cane	0.1	
	vegetables	0.1	
Netherlands	meat	0.7	fat basis
	milk	0.02	
	cereals	0.05	
	corn (sweet)	0.7	
	other fruits	0.5	
	nuts	0.1	
	oil seeds	0.1	
	other vegetables	0.5	
	potatoes	0.02*	
	tea	0.1	

Country	Commodity	MRL	Remarks
		mg/kg	
	other food commodities	0.02*	
New Zealand	meat	0.7	
	cereals	0.1	
	fruit	0.5	
	nuts	0.1	
	oil seeds	0.1	
	tomatoes	0.5	
	vegetables	0.5	
Spain	artichokes	0.5	
	cotton seed	0.05	
	grapes	0.5	
	other fruits	0.5	
	maize	0.05	
	nuts	0.05	
	olives	1	
	olive oil	1	
	vegetables	0.5	
	other plant products	0.05	
Sweden	meat, milk	0.02	
	cereals	0.1	
	citrus fruit	0.5	
	fruit	0.3	
	potatoes	0.1	
	vegetables	0.3	
Switzerland	meat	0.2	
	milk	0.05	
	citrus fruit	0.7	
	cole crops	0.7	
	fruit	0.5	
	vegetables	0.5	except cole crops
UK	brassicae	0.5	
	carrot	0.5	
	celery	0.5	
	cucumber	0.5	

Country	Commodity	MRL	Remarks
		mg/kg	
	lettuce	0.5	
	mushroom	0.5	
	onions	0.5	
	tomatoes	0.5	
USA	meat	0.7	
	almonds	0.5	
	apple	0.5	
	apricots	0.5	
	banana	0.2	
	beans	0.5	
	beets	0.75	
	blackberry	0.5	
	blueberry	0.5	
	boysenberry	0.5	
	broccoli	0.7	
	Brussels sprouts	0.7	
	cabbage	0.7	
	carrots	0.75	
	cauliflower	0.7	
	celery	0.75	
	cherry	0.75	
	chicory	0.7	
	citrus fruit	0.7	
	coffee	0.2	
	collard	0.7	
	corn/maize	0.7	
USA cont.	cotton seed	0.2	
	cranberry	0.5	
	cucumber	0.75	
	dandelions	0.7	
	dewberry	0.5	
	endive	0.7	
	figs	0.5	
	ginseng	0.75	

Country	Commodity	MRL	Remarks
		mg/kg	
	grapes	0.75	
	hazelnuts	0.5	
	hops	0.75	
	kale	0.7	
	kiwifruit	0.75	
	lettuce	0.7	
	loganberry	0.75	
	melons	0.75	
	mushrooms	0.75	
	nectarines	0.5	
	olives	1	
	onion	0.75	
	parsley	0.75	
	parsnip	0.5	
	peaches	0.7	
	peanut	0.75	
	pears	0.5	
	peas	0.5	
	pecan	0.5	
	peppers	0.5	
	pineapple	0.5	
	plum	0.5	
	potato	0.1	
	radish	0.5	
	raspberry	0.5	
	sorghum	0.75	
	soya beans	0.1	
	spinach	0.7	
USA cont.	squash, summer	0.5	
	squash, winter	0.75	
	strawberry	0.5	
	sugarbeet	0.5	
	sugar cane	0.7	
	Swiss chard	0.7	

Country	Commodity	MRL	Remarks
		mg/kg	
	tomatoes	0.1	
	turnips	0.5	
	walnut	0.5	
	watercress	0.75	
	wheat	0.05	
	zucchini	0.5	

APPRAISAL

Diazinon, originally evaluated by the JMPR in 1967 and re-evaluated for residues several times up to 1979, is included in the CCPR periodic review programme.

The general CXLs for fruits and vegetables (0.5 mg/kg) were retained by the 1990 CCPR, to await review by the 1993 JMPR.

Information on current world-wide GAP and extensive residue data were provided by one manufacturer and several countries.

Diazinon is an organophosphorus insecticide with a broad spectrum of activity against a wide range of pests: sucking, chewing and boring insects, including soil-living insects. It is effective mainly by contact and stomach action. The product has been introduced world-wide in many countries and is used on numerous crop groups or commodities. It is generally used as a foliar or soil spray or applied as a granule to the soil.

Major target crops are leafy, fruiting, stem and root vegetables, deciduous fruit, rice and maize. Minor crops include berries, cereals, citrus, grapes, mushrooms, nut trees, olives and sugar beet. Additional uses for non-food crops are on ornamentals, grass and turf, and in nurseries.

Citrus fruits

Only limited data were available for oranges and mandarins which the Meeting felt were not sufficient to estimate a maximum residue level. Evidently the PHI (14-21 days) does not much influence residue levels. The Meeting recommended withdrawal of the CXL (0.7 mg/kg).

Pome fruits

The Meeting estimated a maximum residue level of 2 mg/kg (PHI 14 days), based on available trials from Germany, Switzerland and the USA on apples and pears.

Stone fruits

Cherries. Trials at dosage rates of 3.3 kg ai/ha applied 5 times showed residues up to 0.73 mg/kg 10 days after the last application. The Meeting recommended an MRL of 1 mg/kg.

Peaches. Taking into account data from Germany (PHI 14 days) and the USA (PHI 20 days), the Meeting estimated a maximum residue level of 0.2 mg/kg.

Plums (including Prunes). Trials at dosage rates of 3.3 kg ai/ha applied 5 times showed residues up to 0.78 mg/kg 10 days after the last application. The Meeting proposed an MRL of 1 mg/kg.

Prunes [dry]. US trials with dried prunes showed residues up to 1.9 mg/kg. The Meeting estimated a maximum residue level of 2 mg/kg.

Berries

Grapes. On the basis of the data available the Meeting felt unable to estimate a maximum residue level because the data do not match relevant GAP.

Strawberry. After observing the recommended PHI of 5 days the residues were below 0.1 mg/kg except one of 35 results, at 0.12 mg/kg. A maximum residue level of 0.1 mg/kg was estimated.

Cranberry. No results were reported within the reported GAP.

Currants, Black, Red, White. Results of trials in Germany and Switzerland showed residues up to 0.21 mg/kg at the recommended PHI of 14 days. The Meeting estimated a maximum residue level of 0.2 mg/kg.

Blackberries, Boysenberry, Raspberries. On the basis of US data and GAP for caneberries (PHI 7 days) the Meeting recommended an MRL of 0.1 mg/kg for blackberries and boysenberry, and 0.2 mg/kg for raspberries.

Olives. Because too few of the available residue data reflect current GAP the Meeting felt unable to estimate a maximum residue level on olives or olive oil although processing studies are available which indicate an accumulation of diazinon in crude oil by a factor of 3-5. The Meeting recommended withdrawal of the CXLs for olives (2 mg/kg) and olive oil, virgin (2 mg/kg).

Tropical fruits

Persimmons. Only one trial reflected the reported GAP in New Zealand.

Banana. Only two trials reflected the current GAP in Costa Rica. Although no residues were found the Meeting felt unable to estimate a maximum residue level on such limited data.

Kiwifruit. On the basis of 6 new trials from New Zealand (PHI 28 days) the Meeting estimated a maximum residue level of 0.2 mg/kg.

Pineapple. Seven days after application (the PHI in Costa Rica) trials in Honduras and Costa Rica showed residues up to 0.07 mg/kg. Reported results from the USA were not taken into account because exaggerated application rates were used. The Meeting estimated a maximum residue level of 0.1 mg/kg.

Bulb vegetables

Onion, Bulb. At the recommended rate of application and the recommended PHI of 10 days residues were below 0.05 mg/kg, which was estimated as the maximum residue level.

Spring onion. The range of residue levels found in a series of trials was wide, between <0.01 and 0.65 mg/kg. A maximum residue level of 1 mg/kg, after the recommended PHI of 10 days, was estimated.

Brassica vegetables

Broccoli. Results of 10 trials from the USA within recommended GAP (PHI 7 days) showed residues up to 0.23 mg/kg. The Meeting estimated a maximum residue level of 0.5 mg/kg.

Cabbages, Head . On the basis of several trials from the USA within the recommended GAP (PHI 7 days) the Meeting estimated a maximum residue level of 2 mg/kg.

Cauliflower. The Meeting concluded that the reported data were insufficient to estimate a maximum residue level. Most of the trials were in Germany where there is no GAP. These results could be related to GAP in Switzerland (PHI 14 days), but only three trials included this PHI.

Kohlrabi. On the basis of the available trials and GAP in Switzerland (14 days PHI) the Meeting estimated a maximum residue level of 0.2 mg/kg.

Fruiting vegetables

Cucumber. On the basis of a PHI of 7 days (GAP in the USA) the Meeting estimated an MRL of 0.1 mg/kg. One value of 0.4 mg/kg was assumed to be an outlier because in that trial only 0.2 mg/kg was found 3 days after application.

Cantaloupe. Seven days after application (GAP in he USA) residues up to 0.18 mg/kg were found in the reported USA trials. The Meeting estimated a maximum residue level of 0.2 mg/kg.

Squash, Summer. In US trials residues up to 0.05 mg/kg were found 7 days after application. An MRL of 0.05 mg/kg was recommended by the Meeting.

Mushrooms. Limited data from The Netherlands do not reflect the current PHI. The Meeting felt unable to estimate a maximum residue level.

Peppers, Sweet. Although no data were available for the recommended PHI of 5 days in the USA, the data from USA trials at PHIs of 3-7 days clearly show that 5 days after application residues would not exceed 0.05 mg/kg. The Meeting estimated a maximum residue level of 0.05 mg/kg for sweet peppers.

Sweet corn (corn-on-the-cob) . No residues of diazinon were detectable (<0.01 mg/kg) 10-14 days after the last application. Seven days after the last application residues were small, <0.01-0.02 mg/kg. The Meeting proposed an MRL of 0.02 mg/kg.

Maize forage . Residues were detectable at levels of 0.04-7.95 and <0.01-4.95 mg/kg 7 and 14 days after the last application. The Meeting estimated a maximum residue level of 10 mg/kg.

Leafy vegetables

Tomato. On the basis of reported US data and a PHI of 1 day, the Meeting estimated a maximum residue level of 0.5 mg/kg.

Chinese cabbage; Kale, Chinese . Reported trials from Thailand on Chinese cabbage did not include the national PHI of 14 days but data from Thailand on Chinese kale showed residues below 0.02 mg/kg 14 days after application. Taking all the data on both commodities into account, the Meeting felt able to estimate a maximum residue level of 0.05 mg/kg for both Chinese cabbage and kale.

Lettuce, Head; Lettuce, Leaf. No data were available covering the PHI of 10 days registered in the USA where most of the trials were done. However, the great number of results at sampling intervals of 7-14 days clearly show that 10 days after application residues will be below 0.5 mg/kg. The Meeting estimated a maximum residue level of 0.5 mg/kg for both head and leaf lettuce.

Spinach. The Meeting concluded that the residue situation is comparable to that of lettuce and proposed an MRL of 0.5 mg/kg (PHI 10 days).

Legume vegetables

Common beans (pods and/or immature seeds). At a PHI of 7 days residues were at levels up to 0.2 mg/kg, which was estimated as a maximum residue level.

Garden pea, shelled . Residue results from the USA showed a comparable situation to that of common beans. The Meeting estimated a maximum residue level of 0.2 mg/kg (PHI 7 days).

Root vegetables

Carrot. After PHIs of 7 and 14 days residues were below 0.5 and 0.3 mg/kg, respectively. A maximum residue level of 0.5 mg/kg was estimated, covering the recommended PHI of 7 days.

Celeriac; Parsley, Turnip rooted . The results of only one trial were reported for each commodity. The Meeting therefore did not propose an MRL.

Potatoes. After the recommended PHI of 14 days residues were below the reported limit of determination. A maximum residue level of 0.01* mg/kg was estimated.

Radish. After PHIs of 7 and 14 days residues were below 0.1 mg/kg. A maximum residue level of 0.1 mg/kg was estimated, covering the recommended PHI of 10 days.

Sugar beet. At a PHI of 14 days residues were at levels up to 0.1 mg/kg, which was estimated as a maximum residue level.

Sugar beet leaves or tops . On the basis of the reported data and a PHI of 14 days a maximum residue level of 5 mg/kg was estimated.

Stalk and stem vegetables

Artichoke. Limited data available from Spain (3 results, one location) were insufficient to recommend an MRL.

Witloof chicory (sprouts) . In the one trial reported no residues were detectable (<0.01 mg/kg) 14 days after application. No MRL was proposed.

Cereal grains

Maize. In all reported trials there were no detectable residues (<0.02 mg/kg) in the grain. Taking into account the reported results on sweet corn, the Meeting estimated a maximum residue level of 0.02* mg/kg.

Rice. Seven trials were conducted in India, Indonesia and Pakistan. No residues were detectable (<0.02 or <0.03 mg/kg) in the grain or the unhusked grain 23-59 days after the last application. Low residues were detectable in the straw, up to 0.04 mg/kg. Because the data did not match GAP in most of the countries the Meeting agreed not to recommend an MRL. The Meeting recommended withdrawal of the CXL for polished rice (0.1 mg/kg).

Tree nuts

Almonds. All results from the USA clearly show that normally there will be no residues in the nuts (<0.01 mg/kg). However in several cases residues up to 0.03 mg/kg occurred owing to contamination. The Meeting proposed an MRL of 0.05 mg/kg.

Almonds, hull. The available data were at PHIs of 14-45 days (and some at 0 days). The Meeting estimated a maximum residue level of 5 mg/kg (PHI 14-45 days).

Walnuts. On the basis of the available results of 24 supervised trials it is clear that no residues occur in nuts. The Meeting proposed the limit of determination as the MRL (0.01* mg/kg).

Oilseed

Cotton seed. On the basis of only four trials from one year and one country the Meeting felt unable to estimate a maximum residue level and recommended withdrawal of the CXL for cotton seed (0.1 mg/kg).

Animal products

Meat and milks . Residues in the milk and tissues of cattle were reported after applying ear tags to the animals (registered use in Canada).

Two ear tags (6% cypermethrin, 11% diazinon), one per ear, were attached to each of three Holstein dairy cows. Milk samples were taken five hours before application, and 5 h and 1, 3, 7, 14, 21, and 28 days after application.

Residues of diazinon in milk samples were not detectable (<0.0005 mg/kg) until three days after tag application. The residues remained consistently less than 0.002 mg/kg for the entire residue study.

Three Hereford steers were treated with two ear tags (6% cypermethrin, 9.6% diazinon), one per ear. After 14 days, one animal was slaughtered and samples of blood, liver, tongue, muscle, back fat, and kidney fat were analysed; after 100 days the remaining two treated animals were killed and similar samples were analysed.

Diazinon was found on the hair, but in the analysed tissues it was detectable only in the back fat and kidney fat of the animal killed 14 days after tag attachment. The levels were low, 0.032 and 0.035 mg/kg respectively. No residues (<0.01 mg/kg) were found in the back fat, kidney fat, liver, muscle and tongue 100 days after treatment, indicating that there was no accumulation.

Four Hereford steers were treated with two ear tags (20% diazinon) - one per ear. After 7 days, one animal was killed and samples of blood, liver, tongue, muscle, centre back fat, and kidney fat were analysed; a second steer was killed after 14 days and the remaining two after 28 days, all animals being sampled in the same fashion.

Residues were detectable in the centre back and kidney fat of all the animals. The highest levels were 0.045 and 0.041 mg/kg, respectively, on day 14. There were still detectable residue levels of diazinon in the centre back and kidney fat on day 28 at 0.02-0.03 mg/kg.

Data from planned animal transfer studies are not yet available. The Meeting recommended withdrawal of the established MRLs.

Withdrawal of the CXLS for Barley, Fruits (except ...), Hazelnuts, Leafy vegetables, Meat of cattle, pigs and sheep, Milks, Peanut, Pecan, Safflower seed, Sunflower seed, Vegetables (except ...), and Wheat is recommended because available residue data are insufficient although GAP is reported.

Metabolism in plants

Metabolism studies have been carried out on apples, beans, sweet corn, lettuce, potatoes and rice.

In processing

Processed fractions were prepared from apples, grapes, lettuce, endive, maize, pineapples, potatoes, sugar beet and tomatoes. Wine was made in some cases from harvest grapes and olive oil (crude) was prepared from olives in one trial. Generally residues of diazinon are reduced or not detectable in processed commodities with importance for human consumption: juice, sugar, and wine.

A concentration of residues was observed in crude olive oil and in pomace-type fractions, wi th the latter having a potential use as animal feed.

Residues in the edible portion of food commodities

No information was available about the partition of residues between the pulp and peel in citrus fruit, pineapple or cantaloupe.

No residues were detectable in the whole fruit or in separate samples of pulp and peel when diazinon was applied to bananas.

Residues in almonds (kernels) were low, ranging from <0.01 to 0.03 mg/kg 28-45 days after the last application.

Stability of pesticide residues in stored analytical samples

The stability of diazinon and the metabolites diazoxon (G-24576) and hydroxydiazinon (CGA 14128) under freezer storage conditions was determined in maize, tomatoes, potatoes, apples, strawberries, lettuce, soya beans (dry), refined corn oil, tomato paste and sugar beet molasses.

Residues of diazinon are generally stable in crops and processed commodities for a minimum of twenty-six months of freezer storage. A slight decline was observed in strawberries after three months storage, which continued at a much slower rate through twenty-six months.

Residues of diazoxon are unstable in crop and processed fraction substrates, but are stable in maize oil.

Residues of hydroxydiazinon are generally stable in crop substrates and processed fractions. A decline in residues was observed in apples and strawberries after three months of storage and continued at a much slower rate through twenty-six months.

The stability of diazinon under freezer storage conditions was further tested in some animal tissues, namely muscle, liver, kidney and fat of sheep. Diazinon was found to be stable for at least 8 months of storage.

Methods of residue analysis have been described for commodities of plant and animal origin with a limit of determination of 0.01 mg/kg in most commodities. Diazinon is classified as fat-soluble (octanol/water partition coefficient 3.95). In a large number of studies residues of the potential metabolites diazoxon (G 24576) and hydroxydiazinon (CGA 14128) were determined besides parent diazinon. Since these two compounds were practically not found in crops at harvest or in processed commodities it can be concluded that the use of the product according to GAP may be reliably monitored by determining the parent compound alone.

The data now available from the various metabolism studies show that

major metabolites identified in plant and soil metabolism, namely 4-hydroxy-2-isopropyl-6-methylpyrimidine (G 27550), 4-hydroxy-2-(2-hydroxyprop-2-yl)-6-methylpyrimidine (GS 31144), and diazoxon (G 24576), also occur in animal metabolism (rat). Thus there is no need to include compounds other than diazinon in the residue definition.

RECOMMENDATIONS

On the basis of the data on residues from supervised trials the Meeting concluded that the residue levels listed below (next page) are suitable for establishing maximum residue limits.

Definition of the residue: diazinon (fat-soluble)

Commodity		Recommended MRL (mg/kg)		PHI, days
CCN	Name	New	Previous	
	Almond, hull	5	-	14-45
TN 0660	Almonds	0.05	0.1	14
GC 0640	Barley	W	0.1	
FB 0264	Blackberries	0.1	0.5[1]	7
FB 4079	Boysenberry	0.1	0.5[1]	7
VB 0400	Broccoli	0.5	0.5[2]	7
VB 0041	Cabbages, Head	2	0.5[2]	7
VC 4199	Cantaloupe	0.2	0.5[2]	7
VR 0577	Carrot	0.5	0.5[2]	7
TS 0013	Cherries	1	0.5[1]	10
VL 0467	Chinese cabbage	0.05	0.7[3]	14
FC 0001	Citrus fruits	W	0.7	
VP 0526	Common bean (pods and/or immature seeds)	0.2	0.5[2]	7
SO 0691	Cotton seed	W	0.1	
VC 0424	Cucumber	0.1	0.5[2]	7
FB 0021	Currants, Black, Red, White	0.2	0.5[1]	14
A02 0002	Fruits (except as otherwise listed)	W	0.5	
VP 0529	Garden pea, shelled	0.2	0.5[2]	7
TN 0666	Hazelnuts	W	0.1	
VL 0480	Kale	0.05	0.7[3]	14
FT 0341	Kiwifruit	0.2	0.5[1]	28
VB 0405	Kohlrabi	0.2	0.5[2]	14
VL 0053	Leafy vegetables	W	0.7	
VL 0482	Lettuce, Head	0.5	0.7[3]	10
VL 0483	Lettuce, Leaf	0.5	0.7[3]	10
AF 0645	Maize forage	10	-	7-14
GC 0645	Maize	0.02*	-	45-150
MM 0097	Meat of cattle, pigs and sheep	W	0.7 (fat)V	
ML 0106	Milks	W	0.02 FV	
OC 0305	Olive oil, virgin	W	2	
FT 0305	Olives	W	2	

Commodity		Recommended MRL (mg/kg)		PHI, days
CCN	Name	New	Previous	
VA 0385	Onion, Bulb	0.05	0.5[2]	10
FS 0247	Peach	0.2	0.7	14-20
SO 0697	Peanut	W	0.1	
TN 0672	Pecan	W	0.1	
VO 0445	Peppers, Sweet	0.05	0.5[2]	5
FT 0353	Pineapple	0.1	0.5[1]	7
FS 0014	Plums (including Prunes)	1	0.5[1]	10
FP 0009	Pome fruits	2	0.5[1]	14
VR 0589	Potato	0.01*	0.5[2]	14
DF 0014	Prunes	2	0.5[1]	10
VR 0494	Radish	0.1	0.5[2]	10
FB 0272	Raspberries, Red and Black	0.2	0.5[1]	7
CM 1205	Rice, polished	W	0.1	
SO 0699	Safflower seed	W	0.1	
VL 0502	Spinach	0.5	0.7[3]	10
VA 0389	Spring onion	1	0.5[2]	10
VC 0431	Squash, Summer	0.05	0.5[2]	7
FB 0275	Strawberry	0.1	0.5[1]	5
AV 0596	Sugar beet leaves or tops	5	-	14
VR 0596	Sugar beet	0.1	0.5[2]	14
SO 0702	Sunflower seed	W	0.1	
VO 0447	Sweet corn (corn-on-the-cob)	0.02	0.7	7-14
VO 0448	Tomato	0.5	0.5[2]	1
A01 0002	Vegetables (except as otherwise listed)	W	0.5	
TN 0678	Walnuts	0.01*	0.1	14-45
GC 0654	Wheat	W	0.1	

W: The previous recommendation is withdrawn
[1] Fruits (except as otherwise listed)
[2] Vegetables (except as otherwise listed)
[3] Leafy vegetables

FURTHER WORK OR INFORMATION

Desirable

1. Results of ongoing residue studies on citrus fruits, hazelnuts, hops, pecans and peanuts.

2. Data from dairy cattle transfer studies reported to be completed.

3. Additional information on registered veterinary use including residue data.

REFERENCES

References are listed both alphabetically and numerically. The numerical list gives full references. The alphabetical list is intended as an aid to cross reference and gives author, date and reference number from the numerical list.

1. Numerical list

1. Laanio, T.L., Dupuis, G. and Esser, H.O. 1972. Fate of ^{14}C-labelled Diazinon in Rice, Paddy Soil and Pea Plants. J. Agr. Food Chem. 20, 1213-1219.
2. Guth, J.A. 1972. Adsorption and Leaching Behaviour of Diazinon in various Soils. Ciba -Geigy AG, Basel, Switzerland, Report SPR 46/72. Unpublished.
3. Blass, W. 1973. Diazinon - Gas Chromatographic Determination of Residues in Plant Material. Ciba -Geigy AG, Basel, Switzerland, REM 7a/73. Unpublished.
4. Altenburger, E. 1976a. Diazinon (G -24480) and Methidathion (GS -13005) Residues in preserved Olives after Application of Diazinon/Methidathion 2:1 (Tankmix) and Ultracide (R) 40E, respectively. Ciba -Geigy AG, Basel, Switzerland, RVA 235/76. Unpublished.
5. Altenburger, E. 1976b. Diazinon (G -24480) and Methidathion (GS -13005) Residues in Olives and Olive Oil after Application of Diazinon/Methidathion 2:1 (Tankmix) and Ultracide (R) 40E, respectively. Ciba -Geigy AG, Basel, Switzerland, RVA 234/76. Unpublished.
6. Burkhard, N. 1977. Volatilization of Diazinon from Soil under Laboratory Conditions. Ciba -Geigy AG, Basel, Switze rland, Project Report 57/77. Unpublished.
7. Burkhard, N. 1978. Photolysis of Diazinon on Soil Surfaces under artificial Sunlight Conditions. Ciba -Geigy AG, Basel, Switzerland, Project Report 2/78. Unpublished.
8. Guth, J.A. 1978. Leaching Model Study with the Insecticide Diazinon in four Standard Soils. Ciba -Geigy AG, Basel, Switzerland, Project Report 29/78. Unpublished.
9. Burkhard, N. 1980. Leaching Characteristics of aged ^{14}C-Diazinon Residues in two Standard Soils. Ciba -Geigy AG, Basel, Switzerland, P roject Report 9/80. Unpublished.
10. Schnabel, D. 1981. Diazinon; Deep Freeze Stability Study of Residues in Musle, Liver, Kidney and Fat of Sheep. Ciba -Geigy AG, Basel, Switzerland, Report SPR 19/81. Unpublished.
11. Keller, A. 1981. Degradation of Diazinon in aerobic Soil. Ciba -Geigy AG, Basel, Switzerland, Project Report 37/81. Unpublished.
12. Keller, A. 1983. Leaching Characteristics of aged ^{14}C-Diazinon Residues in a sandy loam soil. Ciba -Geigy AG, Basel, Switzerland, Project Report 8/83. Unpublished.
13. Hohl, J. 1983. Gas Chromatographic Determination of Organophosphorous Esters in Plant Material (including Diazinon). Ciba -Geigy AG, Basel, Switzerland, REM 15/82 Appendix 2. Unpublished.
14. Mortison, J.P. 1985. Photolysis of Diazinon on Soil. Biospherics USA, Project No. 85-E-044 SP. Unpublished.
15. Strong, M.B., Wiggins, D. and Bull, M.S. 1986. Diazinon Residues in Fat and Tissues of Cattle sprayed with an Emulsion of Neocidol EC250. Ciba-Geigy AUSTRALIA, Ltd., Technical Report No 86/6/1075. Unpublished.

16. Bull, M.S., Adams, S. and Strong, M.B. 1986. Diazinon Residues in Milk of Dairy Cattle following Treatment by Spraying with an Emulsion of Neocidol EC250. Ciba-Geigy AUSTRALIA, Ltd., Technical Report No 86/6/1076. Unpublished.
17. Vonk, J.W. and de Jong, P.W.J. 1987. Degradation of Diazinon in Soil at 10°C. TNO, The Netherlands, Ref. Nr. 87/0801/3606-14943/HV/JB. Unpublished.
18. Spare, W.C. 1987. Leaching Characteristics of Diazinon. Agrisearch Inc., USA, Project No. 1293. Unpublished.
19. Szeto, S. and Clements, S.J. 1987. Residues of Chlorpyrifos and Diazinon in Cranberry. Rapport de recherche sur les pesticides - Pesticide Research Report, 359.
20. Surgeoner, G.A., Flannigan, G. and Braun, H.E. 1987a. Cypermethrin and Diazinon Residues in Milk Associated with Combination Insectizide Ear Tags. Rapport de recherche sur les pesticides - Pesticide Research Report, 352.
21. Surgeoner, G.A., Flannigan, G., Braun, H.E. and Purdy, J. 1987b. Cypermethrin/Diazinon Residues in Meat Associated with Ear Tags. Rapport de recherche sur les pesticides - Pesticide Research Report, 346 - 347.
22. Brown, K. and Lai, K. 1988a. Characterisation of ^{14}C-Diazinon Metabolites in Goats. Ciba -Geigy Corp., USA, Project No. ABR -88118. Unpublished.
23. Brown, K. and Lai, K. 1988b. Characterisation of ^{14}C-Diazinon Metabolites in Chicken. Ciba -Geigy Corp., USA, Project No. ABR -88119. Unpublished.
24. Prestidge, R. 1988. Residue Report for Diazinon, Trial Ref. 4041:88.01. MAF Technology, Ruakura Agricultural Research Centre, Hamilton, New Zealand. Unpublished.
25. Ritcey, G., McIver, S. and McEwen, F.L. 1988. Pesticide Residue in Beans after Culinary procedures. Rapport de recherche sur les pesticides - Pesticide Research Report, 299.
26. Rezaaiyan, R., Cross, C. and McFarland, J. 1989. Uptake and Metabolism of 2D-^{14}C-Diazinon in Greenhouse grown Sweet Corn. Ciba -Geigy Corp., USA, Project No. ABR-89057. Unpublished.
27. Wong, A., McDonald, J. and McFarland, J. 1989a. Uptake and Metabolism of 2D-^{14}C-Diazinon in Field grown Potatoes. Ciba -Geigy Corp., USA, Project No. ABR-89059. Unpublished.
28. Wong, A., Rezaaiyan, R. and McFarland, J. 1989b. Uptake and Metabolism of 2D-^{14}C-Diazinon in Field grown Apples . Ciba -Geigy Corp., USA, Project No. ABR-89058. Unpublished.
29. Kühne -Thu, H. 1989a. Gas Chromatographic Determination of Unpolar Pesticides in Plant Material - General Method (includes Diazinon). Ciba-Geigy AG, Basel, Switzerland, REM 119.01. Unpublished.
30. Brown, K. and Lai, K. 1989. Characterisation and Identification of Diazinon Metabolites in Rat. Ciba -Geigy Corp., USA, Project No. ABR -88164. Unpublished.
31. Ross, J.A. and Gold, B. 1989a. Ciba-Geigy Residue Chemistry Test Report OW-IR-618-88. Ciba-Geigy Corp., USA, Project Number 302204. Unpublished.
32. Kühne-Thu, H. 1989b. Determination of residues of parent compound and one metabolite in pineapples after treatment with insectizide/acaricide 'Diazinon 600 EC' - field experiment. Ciba -Geigy AG, Bas el, Switzerland, Residue report 1058/88. Unpublished.
33. Kühne-Thu, H. 1989c. Determination of residues of parent compound and one metabolite in pineapples after treatment with insecticide/acarizide 'Diazinon 600 EC' - field experiment. Ciba -Geigy AG, Bas el, Switzerland, Residue report 1059/88. Unpublished.
34. Ross, J.A. and Gold, B. 1989b. Ciba-Geigy Residue Chemistry Test Report OW-IR-607-88. Ciba-Geigy Corp., USA, Project Number 302191. Unpublished.
35. Ross, J.A. and Gold, B. 1989c. Ciba-Geigy Residue Chemistry Test Report MW-IR-502-88. Ciba-Geigy Corp., USA, Project Number 302191. Unpublished.
36. Ross, J.A. and Gold, B. 1989d. Ciba-Geigy Residue Chemistry Test Report OW-IR-203-88. Ciba-Geigy Corp., USA, Project Number 302195. Unpublished.
37. Ritcey, G. and McEwen, F.L. 1989. Pesticide Residue in Tomatoes. Rapport de recherche sur les pesticides - Pesticide Research Report, 292.
38. Surgeoner, G.A., Sach, B., Ewing, R. and Braun, H.E. 1989. Residues of Diazinon and Diazinon Metabolites in Meat Associated with 20% Diazinon Ear Tags. Rapport de recherche sur les pesticides - Pesticide Research Report, 275 - 276.

39. Wong, A. and McFarland, J. 1990a. Uptake and Metabolism of 2D-^{14}C-Diazinon in Field grown Green Beans. Ciba -Geigy Corp., USA, Project No. ABR-90040. Unpublished.
40. Wong, A. and McFarland, J. 1990b. Uptake and Metabolism of 2D-^{14}C-Diazinon in Field grown Lettuce. Ciba -Geigy Corp., USA, Project No. ABR-90039. Unpublished.
41. Rezaaiyan, R. and McFarland, J. 1990. Uptake and Metabolism of ^{14}C-Diazinon in Greenhouse Rotational Crops grown in Soil previously used for growing Corn. Ciba -Geigy Corp., USA, Project No. ABR -90064. Unpublished.
42. Sobralske, M., Wong, A. and McFarland, J. 1990. Uptake and Metabolism of 2D-^{14}C-Diazinon in Field Rotation al Crops grown in Soil previously used for growing Target Crops. Ciba -Geigy Corp., USA, Project No. ABR -90065. Unpublished.
43. Hubbard, L. and Todt, J. 1990. Determination of Diazinon, Diazoxon and CGA-14128 Residues in Crops, Crop Fractions, and Animal T issues using Gas Chromatography. Ciba -Geigy Corp., USA, Analytical Method AG -550A. Unpublished.
44. Barr, H.P. and Carlin, T.J. 1990. Characterisation and Identification of Major Metabolites in Tissues of Sheep treated dermally with ^{14}C-Diazinon. Ciba-Geigy Corp., USA, Project No. ABR-90014. Unpublished.
45. Gold, B. 1990a. Ciba -Geigy Residue Chemistry Test Report 02 -IR-023-88. Ciba-Geigy Corp., USA, Project Number 302198. Unpublished.
46. Gold, B. 1990b. Ciba -Geigy Residue Chemistry Test Report MW -IR-603-88. Ciba-Geigy Corp., USA, Project Number 302201. Unpublished.
47. Ministério dos Negócios Estrangeiros 1990. Residues of diazinon resulting from supervised trials carried out in Portugal. Ministerio dos Negocios Estrangeiros, Lisboa, Portugal. Unpublished.
48. Beidler, W.T. and Moore, L. 1991. Residue Stability of Diazinon and Metabolites in Crop Substrates and Processed Commodities under Freezer Storage Conditions. Ciba -Geigy Corp., USA, Summary Report ABR -91015. Unpublished.
49. Kühne -Thu, H. 1991. De termination of residues of parent compund and metabolite CGA 14128 (hydroxydiazinon) in pineapples (fruits, juice, remainder) after treatment with 'Diazinon 600 EC - field experiment. Ciba-Geigy AG, Basel, Switzerland, Report of residue studies 1050 + 1051/88. Unpublished.
50. Patterson, T. 1991. Residue Report for Diazinon, Trial ref. 4240:91.1165. Ciba Geigy and MAF Technology, Ruakura Agricultural Research Centre, Hamilton, New Zealand. Unpublished.
51. Ministry of Industry 1991. Residues resulting from supervised trials. Ministry of Industry, Thai Industrial Standards Institute, Bangkok, Thailand. Unpublished.
52. Ministry of Trade and Industry 1992. Diazinon residues in or on Chinese cabbage and onions. Ministry of Trade and Industry, Advisory Committee on Foodstuffs, Helsinki, Finland. Unpublished.
53. Ministry of Welfare, Health, and Cultural Affairs 1993. Residue Data Summary from Supervised Trials. Ministry of Wellfare Health and Cultural Affairs, Division of Food and Product Safety, Rijswijk, Netherlands. Unpublished.
54. Subdireción General de Sanidad Vegetal (M.A.P.A.) 1993. Residue Data Summary from Supervised Trials. Subdireccon General de Sanidad Vegetal (M.A.P.A.), Spain. Unpublished.
55. Federal Biological Research Centre for Agriculture and Forestry 1993. Residue Data Summary from Supervised Trials. Federal Biological Research Centre for Agriculture and Forestry, Braunschweig, Germany. Unpublished.
56. Bodzian, F. 1993a. Report on Pesticide Residue Trials with Diazinon on Yardlong Bean. Deutsche Gesellschaft für Technische Zusammenarbeit, Eschborn, Germany. Unpublished.
57. Bodzian, F. 1993b. Report on Pesticide Residue Trials with Diazinon on Chinese Kale. Deutsche Gesellschaft für Technische Zusammenarbeit, Eschborn, Germany. Unpublished.
58. Ciba-Geigy 1973-1991. 373 residue reports 1973-1991. Unpublished.
59. Prestidge, R.A., Holland, P.T., Clarke, A.D. and Malcolm, C.P. Pesticides for Use Close to and during Harvest of Persimmons. Proc. 42nd N.Z. Weed and Pest Control Conf., 195-199.

2. Alphabetical list

Reference	No.
Altenburger, E. 1976a	4
Altenburger, E. 1976b	5
Barr, H.P. and Carlin, T.J. 1990	44
Beidler, W.T. and Moore, L. 1991	48
Blass, W. 1973	3
Bodzian, F. 1993a	56
Bodzian, F. 1993b	57
Brown, K. and Lai, K. 1988a	22
Brown, K. and Lai, K. 1988b	23
Brown, K. and Lai, K. 1989	30
Bull, M.S., Adams, S. and Strong, M.B. 1986	16
Burkhard, N. 1977	6
Burkhard, N. 1978	7
Burkhard, N. 1980	9
Ciba-Geigy 1973-1991. 373 residue reports 1973-1991. Unpublished	58
Federal Biological Research Centre for Agriculture and Forestry 1993.	55
Gold, B. 1990a	45
Gold, B. 1990b	46
Guth, J.A. 1972	2
Guth, J.A. 1978	8
Hohl, J. 1983	13
Hubbard, L. and Todt, J. 1990	43
Keller, A. 1981	11
Keller, A. 1983	12
Kühne-Thu, H. 1989a	29
Kühne-Thu, H. 1989b	32
Kühne-Thu, H. 1989c	33
Kühne-Thu, H. 1991	49
Laanio, T.L., Dupuis, G. and Esser, H.O. 1972	1
Ministério dos Negócios Estrangeiros 1990.	47
Ministry of Industry 1991	51
Ministry of Trade and Industry 1992.	52
Ministry of Welfare, Health, and Cultural Affairs 1993.	53
Mortison, J.P. 1985	14
Patterson, T. 1991	50
Prestidge, R. 1988	24
Prestidge, R.A., Holland, P.T., Clarke, A.D. and Malcolm, C.P. (undated)	59
Rezaaiyan, R., Cross, C. and McFarland, J. 1989	26
Rezaaiyan, R. and McFarland, J. 1990	41
Ritcey, G. and McEwen, F.L. 1989	37
Ritcey, G., McIver, S. and McEwen, F.L. 1988	25
Ross, J.A. and Gold, B. 1989a	31
Ross, J.A. and Gold, B. 1989b	34
Ross, J.A. and Gold, B. 1989c	35
Ross, J.A. and Gold, B. 1989d	36
Schnabel, D. 1981	10
Sobralske, M., Wong, A. and McFarland, J. 1990	42
Spare, W.C. 1987	18
Strong, M.B., Wiggins, D. and Bull, M.S. 1986	15
Subdirección General de Sanidad Vegetal (M.A.P.A.) 1993.	54
Surgeoner, G.A., Flannigan, G. and Braun, H.E. 1987a	20
Surgeoner, G.A., Flannigan, G., Braun, H.E. and Purdy, J. 1987b	21
Surgeoner, G.A., Sach, B., Ewing, R. and Braun, H.E. 1989	38
Szeto, S. and Clements, S.J. 1987	19
Vonk, J.W. and de Jong, P.W.J. 1987	17
Wong, A., McDonald, J. and McFarland, J. 1989a	27
Wong, A., Rezaaiyan, R. and McFarland, J. 1989b	28
Wong, A. and McFarland, J. 1990a	39
Wong, A. and McFarland, J. 1990b	40

DICHLORVOS (025)

EXPLANATION

Dichlorvos was previously evaluated at the 1965, 1966, 1967, 1969, 1970 and 1974 Meetings and is included in the CCPR periodic review programme (ALINORM 89/24A Appendix V.; ALINORM 91/24 para 360; ALINORM 91/24A para 316). MRLs have been recommended for dichlorvos in a wide range of food commodities.

The compound was first described as an insecticide by CIBA in 1951. It was later introduced by Ciba ("Nogos"/"Nuvan"), Shell ("Vapona") and Bayer ("Dedevap"). It is now manufactured by at least 14 basic producers.

During the past 19 years (since the 1974 residue submission) the use of the compound has been extended to other crops or the recommendations have been adapted to current needs and in some cases uses were discontinued. Consequently additional residue studies have been carried out either to support existing MRLs or to provide a basis for new ones.

The residue studies conducted between 1974 and 1992, as well as the currently recommended uses, are reviewed in this monograph.

USE PATTERN

Dichlorvos is an organophosphate insecticide. It combines both contact and stomach action and has a marked vapour action. It is effective against a broad spectrum of insect pests in the field and in stored products, and is often used in public health vector control and in animal health for ectoparasite control.

In vegetables, dichlorvos provides control of aphids, white flies, thrips, leafhoppers, diamond back moth, whites, armyworms, snout moths, leaf and flea beetles and root maggots. In deciduous fruits, it has good activity against leafroller moths, apple leaf miner, aphids, psyllids, blossom weevil, saw flies, tiger moth, plum curculio and gelechiid moth. In plantation crops (cotton, coffee, tea, cocoa etc.) it is used for the control of leafrollers, hairy caterpillars, thrips, mealybugs, jassids, bollworms, aphids, psyllids and white flies. Dichlorvos is also effective against planthoppers, grasshoppers, leafminers, roller moths, beetles, rice bugs, caterpillars, rice hispa, armyworms and cutworm in rice. Other uses include pest control in ornamentals, oil crops, citrus, tropical fruits, mushooms and greenhouses.

Dichlorvos is officially registered and/or approved for use all over the world. Details of the local use recommendations in the various crops and commodities are summarized in Table 1. The number of applications is often not specified as this depends on pest incidence.

Table 1. Registered or approved uses of dichlorvos. For animals/cattle see Indoor uses at end of Table.

Crop	Country	Application				PHI[2], days
		Formulation[1], type	No.	g ai/hl	kg ai/ha	
Apple/pome fruits	Austria	EC; foliar	>1	75		14
	Greece	EC; foliar	>1	50-75		7
	India	EC; foliar	1-2	20-50		
	Italy	EC; foliar	>1	45.5		7
	Jordan	EC; foliar	weekly		0.3-0.5	2-3
	Lebanon	Ec; foliar	>1	63		2
	Portugal	EC; foliar	>1	75-100	0.75-1.0	7
	Spain	EC; fly trap				
	Switzerland	EC; foliar	>1	50		
Avocado	Columbia	EC; foliar	>1	16		7
	Switzerland	EC; foliar	>1	50		7
Beans	Jordan	EC; foliar	>1		0.4-0.6	2-3
	Lebanon	EC; foliar	1	63		2
	Malaysia	EC; knapsack	>1	111-166		7
	South Africa	EC; full cover	>1	100		3
	Switzerland	EC; foliar	>1	50		7
	Venezuela	EC; foliar	>1		0.75-1.0	20
Berries (small fruits)	Netherlands	EC; foliar EC; space treatment	>1 >1	50 60	0.3-1.2	F4 G10-14
	New Zealand	EC; broadcast LV or HV	>1	60	0.5-0.8	2
	South Africa	EC; full cover	>1	50		2
	Switzerland	EC; foliar	>1	50		7
Brassica (excl cabbage)	Jordan	EC; foliar	>1		0.3-0.5	2-3
	Malaysia	EC; knapsack	>1	111-166		7
	Netherlands	EC; foliar	2-6		0.6-1.0	4
	New Zealand	EC; broadcast	1-2		0.3-0.75	3
	Peru	EC; HV	2-3		0.25-0.5	2-3
	Switzerland	EC; foliar	>1	50		7
Cabbage	Brazil	EC; spray	>1	70		7
	France	EC; foliar	5-8	125	 1.0	5
	Peru	EC; HV	2-3		0.37-0.5	2-3
	Portugal	EC; foliar	>1	75-100	0.75-1.0	7
	South Africa	EC; full cover		100		2
	Venezuela	EC; foliar	>1		1.0-1.5	20
Cereal grains	Argentina	EC; spray		10-20 mg/kg		
	Australia	EC; spray		500 g/hl 6-12 mg/kg		7-28
	Austria	OL; fogging		14-21 mg/kg		28
	Chile	EC; fumigation		1-2%		1

Crop	Country	Application				PHI[2], days
		Formulation[1], type	No.	g ai/hl	kg ai/ha	
	Czechoslovakia	EC; fumigation		0.5-1 %		
	France	EC; fumigation KN; fumigation		19 q 3.5 mg/kg		
	India	EC; foliar spray	2		0.5	F
	Italy	SO; spray/aerosol		2.2-10.2 mg/kg		1
	Netherlands	UL; spray		7-14 mg/kg		14-28
	New Zealand	EC; broadcast			0.35-0.75	F 3
	Nigeria	EC; fogging		20 mg/kg		7
	Poland	AE; fumigation		7-21 mg/kg		3 mo.
	Spain	OL; atomizer		2.1-4.9 mg/kg		15
	Switzerland	AE; spray				
Cocoa	Columbia	EC; foliar	>1	10-40		21
	Netherlands	UL; spray		7-14 mg/kg		14-21
	Switzerland	AE; spray				
Coffee	Columbia	EC; foliar EC; spray		30 10 g/m^3		21
	Indonesia	EC; foliar		100-150		21
	Netherlands	UL; spray		7-14 mg/kg		14-28
Cotton	Columbia	EC; foliar	>1		0.4-0.6	
	Greece	EC; foliar	>1	40-60		7
	Pakistan	EC; foliar	2-3		0.625	2
	Sudan	EC; foliar			0.714	
Cucumber	Canada	EC; spray KN; fogging		6 g/1000 m^2 35 g/1000 m^3		G7
	Czechoslovakia	EC; spray	1	100-300		7
	Greece	EC; foliar	3-4	75-100		
	India	EC; foliar spray	2		0.05	
	Jordan	EC; foliar	>1		0.4-0.6	
	Lebanon	EC; foliar	>1	63		2
	Malaysia	EC; knapsack	>1	111-166		
	Netherlands	AE; aerosol EC; foliar	>1 up to 15	2.7 g/ 100 m^3 50	0.5-1.8	3
	Poland	EC; evaporation EC; spray	>1	5-6 g/100 m^3 50		7
	Switzerland	EC; foliar	>1	50		7
Dates	U.A.E.	EC; foliar/aerial EC; foliar	>1 >1	75-100 75-100		3
Egg plant	Italy	EC; foliar	>1	48.9-97		7
	Jordan	EC; foliar	>1		0.4-0.6	2-3
	Poland	EC; fogging	2	5-6 g/100 m^3		7
	Switzerland	EC; foliar	>1	50		7
Grapes	Austria	EC; foliar		75		14
	Chile	EC; foliar	>1	50-75		2
	France	EC; foliar	2-3		1.25	5
	Greece	EC; foliar	>1	50-75		7
	Lebanon	EC; foliar	>1	63		2

Crop	Country	Application				PHI[2], days
		Formulation[1], type	No.	g ai/hl	kg ai/ha	
	South Africa	EC; full cover	1-3	75		7
	Switzerland	EC; foliar	>1	50		
Indoor uses, see end of Table						
Lettuce	France	EC; foliar	>1		1.0	5
	Greece	EC; foliar	>1		0.5-0.75	7
	Netherlands	EC; foliar AE; space treatment	up to 15	50-60 2 g /100 m^3	0.5-1.8	10-14
	Turkey	EC; foliar	1-2		1.0	5
	Switzerland	EC; foliar	>1	50		7
Melon	Netherlands	EC; foliar space treatment	up to 15 >1	50-60 2 g/100 m^3	0.5-1.8	10
Mushroom	Canada	KN; fogging		10 g/1000 m^3		1
	Czechoslovakia	EC; spray		1.2 %		2-3
	Greece	EC; spray		5-10 g/100 m^3	1	
	Netherlands	EC; space treatment EC; foliar AE; aerosol		41.5 83.0 2.75 g/100 m^3		2 4 2
	New Zealand	EC; fogging		500		1-3
	Poland	EC; thermal spray	>5	8 g/100 m^3		1
	Spain	OL; fogging	1	0.7 g/100 m^3		4
Onion	Austria	EC; foliar	>1	50		14
	Brazil	EC; spray	>1	70		7
	Greece	EC: foliar	>1	40-60		7
	Jordan	EC; foliar	>1		0.4-0.8	2-3
	Switzerland	EC; foliar	>1	50		7
Pea	France	EC; foliar	>1		1.0	5
	Greece	EC; foliar	>1	40-60		7
	Switzerland	EC; foliar	>1	50		7
Peach	Austria	EC; foliar	>1	75		14
	France	EC; foliar	>1	125		5
	Greece	EC; foliar	>1	50-70		7
	Switzerland	EC; foliar	>1	50		7
Peanuts	Greece	EC; foliar	>1	40-60		7
	India	EC; foliar spray	>1		0.37	
	Venezuela	EC; foliar	>1		0.75	2
Peppers	Chile	EC; foliar	>1		0.05	2
	Czechoslovakia	EC; foliar	1	0.1-0.3%		7
	Jordan	EC; foliar	>1		0.4-0.6	2-3
	Malaysia	EC; knapsack	>1	111-166		7
	Netherlands	space treatment	>1	2 g/ 100 m^3		3
	Switzerland	EC; foliar	>1	50		7
Potato	Chile	EC; foliar	>1		0.06-0.1	2
	Switzerland	EC; foliar	>1	50		7
	Venezuela	EC; foliar			0.1-0.15	20
Rice	Bangladesh	EC; foliar	1-2		0.5	14-21

Crop	Country	Application				PHI[2], days
		Formulation[1], type	No.	g ai/hl	kg ai/ha	
	Colombia	EC; foliar	>1		0.3-0.5	7
	Ecuador	EC; foliar	>1	250	0.5	7
	India	EC; foliar spray	1-3		0.37-0.5	
	Indonesia	EC; layer treatment		0.5-1 g/m^2		
	Malaysia	EC; knapsack	>1	111-222		7
	Nigeria	EC; thermofog EC; HV		20 mg/kg	 0.75	7
Sorghum	Nigeria	EC; HV	>1		0.75	
	Indonesia	EC; layer treatment	>1	0.5-1 g/m^2		
Soya bean	Colombia	EC; foliar	>1		0.5	7
	India	EC; foliar spray	2		0.22-0.3	
	Indonesia	EC; layering	>1	0.5-1 g/m^2		
Tea	Indonesia	EC; foliar	>1	100-150	0.5-0.75	7
	Turkey	EC; foliar	>1	75		5
Tomato	Bolivia	EC; foliar	>1		0.3-0.5	2-10
	Brazil	EC; spray	>1	70		7
	Canada	EC; spray UN; fogging		6 g/100 m^2 35 g/1000 m^3		g7
	Chile	EC; foliar	>1		0.05	2
	Czechoslovakia	EC; foliar	>1	0.1-0.3%		7
	Ecuador	EC; foliar	>1	100	0.4	1
	Jordan	EC; foliar	>1		0.4-0.6	2-3
	Lebanon	EC; foliar	>1	63		7
	Malaysia	EC; knapsack	>1	111-166		7
	Netherlands	EC; foliar AE; space treatment	up to 15	50-60 2 g/100 m^3	050-1.8	3
	South Africa	EC; full cover	>1	100		2
	Switzerland	EC; foliar	>1	50		7
Indoor uses	Australia	EC; spray space treatment		3 g/m^2 7 g/m^3		
	Canada	EC; spray EC; direct spray[3]		0.25 g/m^2 0.06-0.11 g/cow		
	France	KN; aerosol	1	0.077 g/m^3		
	Netherlands	AE; space treatment		1 g/ m^3		
	Switzerland	AE; aerosol				

[1] Code for formulation: AE Aerosol dispenser; EC Emulsifiable concentrate; KN Cold-fogging concentrate; OL Oil-miscible liquid; SO Spreading oil; UL Ultra low-volume (ULV) liquid.
[2] Pre-harvest interval refers to outdoor or field use unless it is specified (F-field, G-greenhouse).
[3] Applied directly on cattle.

RESIDUES RESULTING FROM SUPERVISED TRIALS

Supervised trials have been carried out in a number of countries during the period 1973-1992. Results of these and some earlier trials are given in Tables 2-14. Samples from the trials performed after 1973 were analyzed by gas chromatography and the residues were determined as parent dichlorvos. The residue data in earlier trials were obtained by an automated cholinesterase-inhibition method.

In the Tables the countries (except the UK) are indicated by three-letter codes as follows:
ARG-Argentina, AUL-Australia, AUS-Austria, BGD-Bangladesh, BOL-Bolivia, BRA-Brazil, CAN-Canada, CHI-Chile, COL-Columbia, CZE-Czechoslovakia, ECU-Ecuador, FRA -France, GRE-Greece, IND-India, INS-Indonesia, ITA-Italy, JOR-Jordan, LEB-Lebanon, MAL-Malaysia, NET-Netherlands, NZE-New Zealand, NIR-Nigeria, PAK-Pakistan, PER-Peru, POL-Poland, POR-Portugal, SAF-South Africa, SPA-Spain, SUD-Sudan, SWI-Switzerland, TUR-Turkey, UAE-United Arab Emirates, VEN-Venezuela.

Underlined residues in the Tables are from treatments in accordance, or approximately in accordance, with GAP.

Apples. Five field trials were carried out in Switzerland by applying dichlorvos at a rate of 1.5 kg ai/ha or with a spray ai concentration of 0.05-0.1%. Residue data are summarized in Table 2. Residues decreased from 0.05-0.45 mg/kg at 0-1 day to <0.02 mg/kg within 14 days.

Cherries. Cherries were treated at the recommended and double rates (0.65-1.25 kg ai/ha) in South Africa. The residues (Table 3) were 0.05-0.39 mg/kg and <0.02 at days 2 and 8, respectively.

Peaches. Four field trials were carried out in France and Switzerland. Single applications were made on peach trees at a rate of 50 or 125 g ai/hl. The residue data are shown in Table 4. Residues in peaches decreased from 0.69 mg/kg immediately after treatment to <0.02-0.15 mg/kg in five days and <0.02-0.05 mg/kg in seven days.

Table 2. Residues in apples from supervised trials in Switzerland.

Year	Application				Residues (mg/kg) at days after last application					Ref.
	Form	No.	g ai/ha	g ai/hl	0-1	3-4	5-7	14	21	
1970	50EC	1		50	0.05	0.03	<0.03			6.
1971	50EC	1		100	0.45	0.14	0.1			7.
1971	500EC	3	1500		0.2	0.05	<0.02	<0.02	<0.02	8.
1992	500EC	3	1500				<0.02	<0.02	<0.02	9.
1992	500EC	3	1500				<0.02	<0.02	<0.02	10.

Table 3. Residues of dichlorvos in cherries and strawberries from supervised trials.

Crop/country/year	Application				Residues (mg/kg) at days after last application					Ref.
	Form	No.	g ai/ha	g ai/hl	1	2	3	4-5	7-8	
Cherry, SAF, 1979	100 EC	1	625	50	0.24	0.05	<0.02	<0.02	<0.02	18
Cherry, SAF, 1979	100 EC	1	1250	100	1.39	0.39	0.16	0.04	<0.02	19
Strawberry, SWI, 1967	50 EC	1		0.05%		0.35	0.07		<0.02	20

Table 4. Residues of dichlorvos in peaches from supervised trials.

Country, Year	Application			Residues (mg/kg) at days after last application						Ref
	Form	No.	g ai/hl	0	1	2	4	5	6-7	
FRA, 1973	50EC	1	0.125%			0.32-0.38	0.05-0.08*	0.09-0.15*	0.01-0.05*	81
FRA, 1973	50EC	1	0.125%				<0.02	<0.02	<0.02	82
SWI, 1969	50EC	1	0.05%	0.69	0.43	0.24				55
SWI, 1970	50EC	1	0.05%		0.13	0.10	0.05		0.04	56

* Replicate samples from a single trial.

Grapes. Three field trials were carried out in France and Switzerland. One or three applications were made at a rate of 1.0 -1.25 kg ai/ha. The residue data are given in Table 5. Residue values were 0.23-1.14 mg/kg, <0.03-0.11 mg/kg and <0.02-<0.03 mg/kg after pre-harvest intervals of zero, three and ten days, respectively.

Table 5. Residues of dichlorvos in grapes from supervised trials.

Country Year	Application				Residues (mg/kg) at days after last application						Ref.
	Form	No.	g ai/ha	g ai/hl	0	1	3	5-7	10-14	17	
FRA, 1971	50EC	1	1250						<0.02	<0.02	46
SWI, 1973	50EC	1	1000		0.23	0.05	<0.03	<0.03	<0.03		47
SWI, 1992	500 EC	3	1200		1.14		0.11	0.08	<0.02		48

Strawberries. A single trial was reported from Switzerland. The residues are shown in Table 3.

Dates. Two field trials were carried out in Iraq (Ref.43) according to the GAP in UAE. Date trees were sprayed once with dichlorvos at 0.95-2.0 kg ai/ha. No measurable residue (<0.03 mg/kg) was detected in mature fruits 138 days after the treatments.

Avocados. A single field trial was carried out in Australia (Ref.3). Dichlorvos was applied once to avocado trees at a rate of 50 g/hl. Results showed an initial deposit of 0.07 mg/kg and no measurable residues (<0.01/0.02 mg/kg) in whole fruit, pulp or peel of avocados at day 3.

Onions. Two field trials were carried out in Switzerland (Refs. 52-53.) with 40% higher than the recommended rate. There were three foliar applications (1 week interval) at a rate of 0.56 kg ai/ha. No measurable residues (<0.02 mg/kg) were detected in onion bulbs during the first 7 days after the last application.

Brassicas. Ten field trials were carried out with Brussels sprouts, cabbage, cauliflower and kale in India, The Netherlands and Switzerland. One to four applications were made at a rate of 0.5-0.8 kg ai/ha or as

0.05-0.075% ai spray solution. The results are summarized in Table 6. Residues were at or below 0.04 mg/kg after a pre-harvest interval of 5 days.

Table 6. Residues of dichlorvos in brassica vegetables and kale from supervised trials.

Crop, Country, Year	Application				Residues (mg/kg) at days after last application						Ref.
	Form	No.	g ai/ha	g ai/hl	0	5	7-8	10-11	14	18	
Brussels sprouts SWI, 1970	50EC	1		0.05%	1.25	<0.03	<0.03	<0.03			15
SWI, 1965	50EC	1		0.05%	1.35	<0.04					16
NEL, 1982		1	750	75		<0.01					108
NEL, 1982		1	750	75		<0.01					109
Cabbage SWI, 1992	500EC	4	800		0.66		<0.02		<0.02		17
Cabbage, red NET, 1964	50 EC	1		0.075%				<0.02			2
Cauliflower SWI, 1965	50EC	1			0.37	<0.04					16
Cauliflower IND, 1980	100EC	3	500					<0.02		<0.02	13
Cauliflower IND, 1980	100EC	3	625					<0.02		<0.02	14
Kale, NEL, 1982		1	750	75		0.04					108
NEL, 1982		1	750	75		0.03					108

Cucumbers. Two field trials were carried out in Switzerland according to GAP (Refs. 41, 42). Cucumber plants were sprayed three times with dichlorvos at a rate of 0.6 kg ai/ha. No measurable residues (<0.02 mg/kg) were detected on the day after the last treatment.

Egg plants. Two field trials were carried out in India (Ref. 45) with three foliar applications at rates of 0.37 and 0.62 kg ai/ha. Less than 0.02 mg/kg residues were measured in egg plant fruits, twenty one days after the last application.

Mushrooms. Four glasshouse trials were carried out in The Netherlands and the United Kingdom with fogging or thermal spray applications at rates between 11 and 21 g ai/100 m^3. Residues were between 0.03 and 0.11 mg/kg two days after the treatment (Table 7).

Peppers. A single residue trial was carried out in India. Three applications were made to chilli plants at a rate of 0.4 kg ai/ha. Residues were <0.04 mg/kg at seven and fourteen days after the last application (Ref. 59).

Table 7. Residues of dichlorvos in mushrooms from supervised trials.

Country, Year	Application			Residues (mg/kg) at days after last application				Ref.
	Form	No.	g ai/hl	0	1	2	3	
NET, 1964			100ml/100 m^3	<0.01	<0.01			1
UK, 1963	50 EC	1	11 g/100 m^3	0.3		0.03		88
				0.14	0.02			88
				0.04	<0.02			88

				0.06	0.12			88
UK, 1964	50 EC	1	17 g/100 m^3	25.6	0.9	0.08	0.27	99
UK, 1964	50 EC	1	21.2 g/100 m^3			0.04-0.06		100
						0.09-0.11		100

Tomatoes. Two field trials were conducted in Switzerland according to GAP. Three applications were made at a rate of 70 g ai/hl. Residues were <0.02-0.09 mg/kg, <0.02-0.03 mg/kg and <0.02 mg/kg after zero, one and three days respectively (Refs. 73-74).

Lettuce. Trials were carried out in the field and in glasshouses in Germany, Switzerland and the United Kingdom. One or three applications were made at a rate of 0.5 or 1.25 kg ai/ha, (50 g ai/hl or 0.5 or 1.0 g ai/100 m^3. Residue data are given in Table 8. In field trials, no measurable residues (<0.0002-<0.03 mg/kg) were detected within two days after the last application. In greenhouse trials, somewhat higher residue levels were detected; however, these residues decreased from 0.6-3.0 mg/kg at 1 day to 0.13-0.4 mg/kg within 4 days.

Table 8. Residues of dichlorvos in lettuce from supervised trials.

Country Year	Application				Residues (mg/kg) at days after last application					Ref.
	Form	No.	g ai/ha	g ai/hl	0	1	2	3-4	5-7	
GER, 1972	50 EC	1		0.05%	0.05	0.005	<0.0002	<0.0002	<0.0002	107
SWI, 1972	50 EC	1	500	60	0.72	<0.03	<0.03	<0.03	<0.03	49
SWI, 1992	500 EC	3	1250		0.71			<0.02	<0.02	50
SWI, 1992	500 EC	3	1250		5.94			<0.02	<0.02	51
UK, 1964	50 EC	1		1g/100 m^3		1.6-3.0	0.2-0.6	0.13-0.3		87
UK, 1964	50 EC	1		0.5g/100 m^3		0.6-2.2	0.2-0.5	0.2-0.4		87

Beans. Trials were carried out in Switzerland and the United Kingdom. After three foliar applications at a rate of 1.2 kg ai/ha, no measurable residues (<0.02 mg/kg) were detected in beans at a PHI of three days. The results are shown in Table 9, which also includes the results of post-harvest treatments (see below).

Table 9. Residues of dichlorvos in beans from supervised trials.

Crop/Year Country	Application			Residue, mg/kg, at PHI, days					Ref.
	Form	No.	g ai/ha	0-1	3-4	7	14	21	
Bean pods, SWI, 1992	500 EC	3	1250	0.07	<0.02	<0.02			12
Beans, dry, SWI, 1970	70 AE		20 mg/kg*	13.1		6.75	1.85	1.48	11
Beans, dry, UK, 1963	50 EC		1 g/1000 m^3	2.5-0.47	<LOD				86

* Treated in glass bottles

Peas. In a recent Swiss trial (Ref. 54), pea plants were treated with dichlorvos at a rate of 1.25 kg ai/ha. Three applications were made over a period of three weeks. The spray concentration was three times that given in the national recommendation. No measurable residues (<0.02 mg/kg) were detected in pods or peas four days after the last treatment. The pod:pea ratio increased from 0.7:1 at day zero to about 1:1 in seven days.

Soya beans. Two field trials (Refs. 69-72) were carried out in India.

Application rates were 0.3-0.6 kg ai/ha (foliar). No measurable residue (<0.02 mg/kg) was detected in soya beans after application (21 days PHI).

Witloof chicory. A single trial was reported from The Netherlands (Netherlands, 1993) in which dichlorvos was applied at a rate of 4 g/100 m^3. No residue (<0.01 mg/kg) was detected 14 days after treatment.

Rice. Six field trials were conducted in Bangladesh (Refs. 60-61), Columbia (Ref. 62) and India (Refs. 63-64). One or two applications were made at rates of 0.37-1.0 kg ai/ha. After foliar application no measurable residues (<0.02-<0.05 mg/kg) were detected in either rice grain or straw.

Sorghum. Two field trials were carried out in India. Two applications were made with a two-week interval at a rate of 30 or 50 g ai/hl. No measurable residues (<0.02 mg/kg) were detectable in either grain or straw thirteen days after the last treatment (Refs. 67-68).

Cotton. Field trials were carried out in India. Three to five foliar applications were made in combination with either phosphamidon or diazinon at rates of 0.20-0.37 kg ai/ha. Residues in cotton seeds were <0.01-<0.05 mg/kg at or after 23 days follwing the last application (Refs. 39-40).

Peanuts. In the field trials in India, peanut plants were treated twice with dichlorvos at a rate of 250 g ai/ha. The residue data are given in Table 10 together with the results of Swiss trials on stored peanuts. No measurable residues (<0.03 mg/kg) were detected in peanuts after foliar treatment.

Table 10. Residues of dichlorvos in peanuts from supervised trials.

Country Year	Application				Residues (mg/kg) at days after last application						Ref.
	Form	No.	g ai/ha	g ai/hl	1	7	12-14	16	21	60	
IND, 1974	100 EC	2	250	50			<0.03	<0.03		<0.03	57
		2					<0.03	<0.03		<0.03	57
		2					<0.03	<0.03		<0.03	57
SWI, 1970	7 EC	1	10 mg/kg	1%	4.7-5.0	2.5-2.7	2.1		1.5		58
SWI, 1970	7 EC	1	20 mg/kg	1%	9.1	4.5	3.1-3.3		2.5-2.6		58

Cacao beans. Foliar treatment was at a rate of 0.5 kg ai/ha in Brazil. Residue data are shown in Table 11, which also gives the results of storage trials in The Netherlands and Switzerland (see below). No measurable residue (<0.02 mg/kg) was detected in dried seeds 32 days after application.

Table 11. Residues of dichlorvos in cacao beans from supervised trials.

Country, year, crop	Application				Residues (mg/kg) at days after last application			Ref.
	Form	No.	g ai/ha	g ai/hl	0	21	>32	
BRA, 1982, seed	100 EC	1	500	3.33%			<0.02*	35
NET, 1966, seeds				20% strips**			<0.01-0.02	112
husk							n.d.-0.3	
kernels							0.01	
kernel, roasted							<0.01	
SWI, 1972, seed	50 EC	1	10 mg/kg	0.25%	4.5	<0.1		37
seed, roasted butter					<0.1 0.15	<0.1 <0.1		

SWI, 1972, seed	50 EC	1	100 mg/kg	0.1%	64	13-15		37
seed, roasted butter					0.2 0.8	0.2 0.1		
SWI, 1972, seed	50 EC	1	500 mg/kg	5%	370	50		37
seed, roasted						0.5		

* seeds were collected 32 days after foliar treatment and dried for two weeks at room temperature

** Resin strips of 6.25 x 25 x 0.5 cm containing 20% dichlorvos were placed in 3 x 4.5 m rectangle and held at room temperature.

Tea. A single field trial was reported from China (Zongmao and Haibin, 1968). The application rate was 1.0 kg/ha. The half-life was 0.2 days. After 6 days residues were below 0.1 mg/kg, the limit of determination.

Post-harvest applications

Beans. Two trials were carried out in Switzerland and the United Kingdom. Applications were at a rate of 20 mg/kg and l g/1000 m^3. In dry beans, residues decreased from 13 mg/kg immediately after treatment to less than 1.5 mg/kg at 21 days. The results are shown in Table 9.

Soya beans. A post-harvest application (La Hue *et al.*, 1973) was carried out in the USA at a rate of 20 mg/kg. The initial residues, 4.5-3.2 mg/kg immediately after treatment, decreased to <0.02 mg/kg within 21 days.

Cereals. Numerous (18) trials were carried out in Argentina, Austria, Brazil, France, Germany and Switzerland on wheat, barley and rye. Applications were made as aerosol, fogging or by hanging "Vapona" strips over the barley, rye and wheat storage areas. Application rates were between 7 and 38 mg/kg or 0.3 and 5.6 g ai/ton grain or l strip/40 m^3. The residue data are given in Table 12.

Table 12. Residues of dichlorvos in stored cereals from supervised trials.

Crop Country, Year	Application		Residues (mg/kg) at intervals (days/months) after last application						Ref
	Form	Rate, ai	0-1d	14-17d	21d	1-2m	3m	>6m	
Wheat, ARG, 1979	600 EC	3.2 mg/kg						<0.2	21
Wheat[4], AUS,1975	70 AE	14 mg/kg	5.6-14	5.0	3.5	1.9			22
Wheat, BRA, 1975	2.5 EC	1.25 mg/kg						0.05	113
Barley, FRA, 1969 Wheat	70 AE	7.4 mg/kg	0.53-0.75 1.5-2.6	 0.7					23
Wheat, FRA, 1982	mixture	3.48 mg/kg	3.4[1]-1.96[2]	0.75		0.46-0.33			84
Wheat, FRA, 1986	mixture	3.48 mg/kg	1.56[3]	1.26		0.90	0.53		85
Wheat, GER, 1977	vapona strips	1 strip/ 40 m^3					<0.01-0.02		114
Rye, GER, 1977	vapona strips	1strip/ 40 m^3					<0.01-0.02		115
Barley, SWI, 1969	70 AE	7mg/kg	1.2	0.2	0.14				24
Barley, SWI, 1972	70 AE	5.6 mg/kg	3.0	1.8	0.66-0.74	0.31-0.68			25
Barley, SWI, 1976		38.5 mg/kg	15.0	12.5-21.5	4.0-5.4	2.2-4.6			27
Wheat, SWI, 1969	70 AE	7mg/kg	1.8-2.0	0.8-0.9		0.5			28
Wheat, SWI, 1970	70AE	20 mg/kg		3.1-3.6					29
flour				0.14-0.18					
bread				<0.02					
Wheat[4], SWI, 1975	70 AE	15 mg/kg	7.8	4.8-6.0	4.4-5.2	2.1-5.0	1.5-1.9	0.2-	30

Crop Country, Year	Application		Residues (mg/kg) at intervals (days/months) after last application						Ref
	Form	Rate, ai	0-1d	14-17d	21d	1-2m	3m	>6m	
								0.4	
Wheat[4], SWI, 1975	70 AE	17.5 mg/kg	10.8-11.1	2.2-2.2	1.5-1.25	0.92-0.41			31
Wheat[4], SWI, 1976	70 AE	15 mg/kg	9.7-12.5	0.4-0.6	0.2-0.3				32
Wheat, SWI, 1977	70 AE	14 mg/kg		2.6-2.4			<0.12		33
Wheat, SWI, 1977	70 AE	29 mg/kg				2.4	0.82		34
Wheat, SWI, 1977	70 AE	28 mg/kg				0.7	0.39-0.64		34

[1]at day 0 after application
[2]at day 3 after application
[3]at day 1 after application
[4]sampled at the surface and at 2 m depth

Stored rice was treated in Switzerland with 75-150 mg ai/m^3. Residue data are shown in Table 13. Residues were between 0.12 and 0.23 mg/kg after about three weeks. After 2 months only 0.04 mg/kg was found.

Table 13. Residues of dichlorvos in stored rice from supervised trials in Switzerland in 1965.

Commodity	Application				Residues (mg/kg) at days after last application				Ref.
	Form	No.	g ai/ha	g ai/hl	10-12	17	23-24	60	
Rice	20EC	1	75 mg/m^3	spray	<0.04				65
Rice, ground					<0.04				
Rice, polished	20EC	1	75 mg/m^3	dispenser			0.12		65
Rice, ground							0.16		
Rice	2EC	1	150 mg/m^3	spray	0.5	0.2	0.23	0.04	66

Cereals and other crops. Trials were reported from India in which residues of dichlorvos in various commodities in food storage warehouses were determined at different geographical locations (Rajak, 1973). Nuvan 100 EC was applied at recommended and higher rates to the walls and the floor. The results are summarized in Table 14.

Table 14. Residues of dichlorvos in commodities stored in food storage warehouses treated with Nuvan 100 EC in India (Rajak, 1973).

Commodity, location	Application rate, g ai/100 m^2	Residues (mg/kg) at days after treatment					
		0 (1 hour)	1	2	4	8-11	16-22
Wheat, Rajasthan	100	3.6	2.5	0.88	0.6		
Wheat, Maharashtra	10*	0.13	0.06	0.04	0.04	0.03	0.08
Wheat, Assam	10*		1.0	0.58	0.39	0.1	0.004
Wheat Mixed, Assam	10*				0.05	0.007	0.002
Wheat Mixed, Assam	35				0.99	0.23	0.006
Wheat, Assam	10*				0.48	0.16	0.002
Wheat[1], Assam	10*				0.76	0.2	0.09
Wheat, Assam	35		2.4	1.9	1.5	0.67	0.09

Commodity, location	Application rate, g ai/100 m^2	Residues (mg/kg) at days after treatment					
		0 (1 hour)	1	2	4	8-11	16-22
Wheat[1], Uttar Pradesh	50		0.86	0.64	0.43	0.13	0.03
Wheat, Uttar Pradesh	50		0.66	0.47	0.23	0.1	0.03
Wheat[1], Uttar Pradesh	10*		0.49	0.28	0.18	0.03	0.02
Wheat, Uttar Pradesh	10*		0.36	0.16	0.07	0.03	0.02
Maize, Rajasthan	10*	0.75	0.44	0.24	0.14		
Paddy, Rajasthan	10*	0.51	0.35	0.15	0.1		
Rice, Tamil Nadu	10*		0.22		0.11	0.03	0.01
Coriander, Assam	10*		1.2	0.51	0.26	0.19	0.02
Coriander, Assam	35		2.3	2.1	1.5	0.57	0.09
Cumin, Assam	10*				0.89	0.37	0.1
Cumin[1], Assam	10*				1.1	0.45	0.12
Gram, Rajasthan	10*	0.61	0.33	0.14	0.11		
Gram[1], Uttar Pradesh	50		1.3	0.58	0.2	0.07	0.03
Gram, Uttar Pradesh	50		0.99	0.46	0.19	0.05	0.02
Gram[1], Uttar Pradesh	10*		0.59	0.29	0.17	0.05	0.02
Gram[1], Uttar Pradesh	10*		0.78	0.49	0.23	0.14	0.04
Gram, Uttar Pradesh	10*		0.36	0.11	0.08	0.04	0.02
Gram and Pulse, Tamil Nadu	10*		0.26		0.06	0.04	0.02
Black gram, Maharashtra	10*	0.34	0.03	0.02	0.02	0.01	0.004
Red gram, Maharashtra	10*	0.22	0.05	0.03	0.01	0.009	0.006
Groundnut[1], Uttar Pradesh	50		1.7	0.95	0.32	0.18	0.09
Groundnut, Uttar Pradesh	50		1.3	0.72	0.3	0..15	0.06
Groundnut[1], Uttar Pradesh	10*		0.78	0.49	0.23	0.14	0.04
Groundnut, Uttar Pradesh	10*		0.67	0.37	0.18	0.11	0.03
Lentil, Rajasthan	100	2.7	1.3	0.75	0.6		
Soya bean, Maharasthra	54,5	1.3	0.2	0.16	0.13	<0.04	<0.04
Linseed, Rajasthan	100	2.7	2.0	0.91	0.67		
Linseed, Maharasthra	54,5	1.1	0.49	0.29	0.22	0.07	0.02
Mustard, Assam	10*		0.81	0.62	0.43	0.25	0.09
Mustard, Assam	35		1.8	1.4	0.95	0.89	0.12
Sesame, Tamil Nadu	10*		0.18		0.11	0.06	0.04
Mahua-raisin, Maharashtra	54,5	1.6	0.63	0.16	0.15	0.05	0.01

* Treated at the recommended rate

[1] Sprayed directly on bags

Peanuts. In post-harvest trials in Switzerland, stored peanuts were sprayed with dichlorvos at a rate of 10 or 20 mg/kg. The residue data are in Table 10. Residues decreased from 4.7-9.1 mg/kg immediately after treatment to 2.5-4.5 mg/kg in 7 days and 1.5 to 2.6 mg/kg in 21 days.

Cacao beans. Trials were carried out in The Netherlands and Switzerland. Treatments were made with dichlorvos at a rate of 0.25-5.0% ai in the spray solution. In one trial 20% strips were also used. Residue data are shown in Table 11.

Coffee beans. Coffee beans were sprayed with dichlorvos at concentrations of 10 and 20 mg/kg using a 7 EC formulation. No measurable residue (<0.5 mg/kg) was detected in the beans, four months after application (Ref. 38).

ANIMAL TRANSFER STUDIES

Residues of dichlorvos in livestock (cattle, sheep, goats and pigs) and poultry were discussed in the 1970 Evaluations (FAO/WHO, 1971). Since then further trials have been carried out. The new information is discussed below.

Hens. Twelve egg-laying hens confined in batteries in an 8.6 m^3 building were sprayed with dichlorvos, applying a mist spray three times at three-day intervals (Hurt, 1964). The dosage rate was 50 mg/m^3, double the recommended rate. Six eggs were collected at random on each day. Residues of dichlorvos in the eggs were first detected 2 days after the first treatment. The residue concentration varied from <0.03 to 0.11 mg/kg during the trial. There was no increase in the concentration following consecutive treatments, and the residue declined below the limit of determination 3 days after the last application. Three birds were killed 18 hours after the 3rd treatment and the breast and leg muscles were analyzed. The residues found were between <0.01 and 0.05 mg/kg. Egg production was not affected by the treatments.

Cattle. Each of three cows was sprayed for 31 consecutive days with 59 ml of 1% dichlorvos solution, for the control of horn fly and mosquitoes. Milk samples were collected after 2 hours and 1, 2, 4, 8, 16, 24 and 31 days. Tissue and blood samples were taken one day after the final treatment. No dichlorvos was detected in any milk samples (<0.003 mg/kg) or body tissues (<0.002 mg/kg) from the treated cows (Ivey and Eschle, 1970).

In another experiment, dairy cows were dosed orally with dichlorvos at rates of 1.3, 1.8 and 2.6 mg/kg body weight in the form of polyvinyl chloride pellets. No dichlorvos residues (<0.04 mg/kg) were detected in milk samples collected at 1, 3, 7, 10 or 14 days (Lloyd and Matthysse, 1971).

In a dermal application experiment, six cattle and two dairy cows were sprayed once with 15 litres of an emulsion of "Nuvan" 100 EC at a concentration of 1500 mg/kg. Cattle were slaughtered 1, 3 and 7 days after treatment. Milk samples from the dairy cows were taken 6 h, and 1, 3 and 7 days after treatment (control 1 day before treatment). No measurable residue of dichlorvos was found in milk (<0.005 mg/kg) or in muscle, liver, kidney or fat (<0.02 mg/kg) (Ref.80).

FATE OF RESIDUES

In animals

Animal metabolism studies showed that dichlorvos is readily absorbed, hydrolysed and effectively eliminated. The metabolic pathways were identical in the mammalian species studied, including humans. The main metabolic degradation routes are by hydrolyis, oxidation and demethylation (Figure 1).

Pigs. In studies by Potter *et al.* (1973), nine pigs received a single oral dose of [1-^{14}C]vinyl-dichlorvos (ca. 40 mg dichlorvos/kg feed) formulated as slow-release PVC pellets. After treatment, groups of three pigs were slaughtered after 2, 7 and 14 days. Various tissues were analyzed at each interval. The ^{14}C content of the tissues, as dichlorvos equivalents, varied from 1.6 mg/kg in subcutaneous fat to 33 mg/kg in liver after 2 days, and from 1.9 mg/kg in brain to 9.7 mg/kg in liver after 14 days, but no dichlorvos, demethyl-dichlorvos, dichloroacetaldehyde or dichloroacetic acid could be detected. The mean residues found are given in Table 15.

Figure 1. Metabolic pathways of dichlorvos in mammals

Table 15. ^{14}C residues in tissues from pigs dosed with [^{14}C]dichlorvos.

Substrate	Mean ^{14}C expressed as dichlorvos, mgkg		
	2 days	7 days	14 days
Adrenals	8.7	6.5	4.6
Bladder	8.1	8.0	5.6
Blood	6.5	3.7	2.8
Brain	2.5	1.9	1.9
Carcase	5.2	5.1	4.2
Duodenum	12.3	5.6	3.1
Femur	11.7	7.4	5.4
Gastrocnemius muscle	6.3	4.6	4.8
Kidney	12.2	7.6	4.0
Liver	32.9	30.9	9.7
Lungs	8.6	5.3	3.8
Mesenteric fat	2.6	4.2	2.5
Pancreas	9.6	5.7	3.5
Quadriceps muscle	4.7	4.8	4.3
Salivary gland	10.0	5.6	3.7
Spiral colon	8.1	3.8	3.5
Spleen	11.5	7.0	4.3
Stomach	7.1	4.7	3.0
Subcutaneous fat	1.6	4.0	2.2
Thyroid	5.0	5.1	5.5

In the 14-day trial, the radioactivity administered was distributed as follows: 61.8% in the pellets recovered from the faeces, 5.6% in the remainder of the faeces, 3.6% in the urine, 14.1% in the expired air and 9.6% in the carcase. It was concluded that the ^{14}C present in the tissues was the result of incorporation of one- and two-carbon fragments from the vinyl moiety of dichlorvos into normal tissue constituents.

In plants

The metabolism of dichlorvos in plants was described on the basis of published ^{32}P studies in the 1970 monograph (FAO/WHO, 1971). New data including studies with ^{14}C in wheat and ^{32}P in various plants, and of dislodgeable residues in turf and grass are summarized below.

The main routes of degradation of dichlorvos in plants were found to be (Figure 2):

1. cleavage of the vinyl ester bond to form the major metabolite dimethyl phosphate;
2. demethylation to monomethyl phosphate and phosphoric acid;
3. demethylation of dichlorvos to demethyl-dichlorvos (minor pathway);
4. loss by volatilization (thermal, solar).

Cotton. Cotton plants of the Deltapine Smooth leaf variety were grown in a glasshouse and treated by injecting aqueous solutions of ^{32}P-labelled dichlorvos (100 ìg) into the petioles of individual, fully expanded leaves. Leaf samples were collected 1, 24 and 48 hours after the treatment. Dichlorvos was rapidly lost from leaf surfaces by volatilization and

hydrolysis with a half-life of only a few hours. Samples were partitioned into chloroform and aqueous fractions. Identification of the parent compound and metabolites was based mainly on co-chromatography of radioactive material with authentic compounds after two-dimensional TLC. Enzymic hydrolysis was also employed in identifying conjugates. The studies showed 81.2% loss as volatiles, 12.3% as dimethyl phosphate (major metabolite), 2.2% as phosphoric acid + methyl phosphate and 0.1% as demethyl-dichlorvos (minor metabolites) after 48 hours. No parent residue was detectable at this time (Bull and Ridgway, 1969). Unextractable residues corresponded to less than 5% of the applied radioactivity. The detailed fate of dichlorvos in cotton leaves over the 48-hour test period is shown in Table 16.

In another greenhouse study cotton plants of 15 cm height, with or without roots, were placed in a formulated 0.1-0.2% [^{32}P]dichlorvos solution. Foliar experiments were also performed by dipping plants into solutions of the labelled ai. Dichlorvos was degraded with a half-life of 4.6 hours. Dimethyl phosphate was a major metabolite detected during the 24-hour test period. Here again about 80% (78.4%) of the radioactivity was lost by volatilization, confirming the 1969 study (Dedek *et al.*, 1979).

Figure 2. Main plant metabolites of dichlorvos

Table 16. Relative concentrations of radioactive compounds obtained from individual cotton leaves after petiole injection with 100ìg ^{32}P-labelled dichlorvos.

Compound	% of dose		
	l h	24 h	48 h
H_3PO_4 + methyl phosphate	0.9	1.1	2.2
dimethyl phosphate	6.0	13.3	12.3
O-demethyl-dichlorvos	1.7	0.2	0.1
dichlorvos	36.8	0.1	0.0
unextractable	1.0	4.5	4.2
lost (volatile)	53.6	80.8	81.2

Bean, potato and tomato.The above study by Dedek *et al*. (1979) also included greenhouse-grown bean, potato, and tomato plants of 15 cm height treated in the same way. Dichlorvos was degraded with a half-life of 6.8, 4.6 and 6.8 hours in beans, tomatoes and potatoes respectively. Dimethyl phosphate was a major metabolite detected during the 24 hour test period. Volatile radioactivity accounted for about 60% of the ^{32}P in all three plant species. *In vitro* homogenate experiments with beans showed a longer half-life, 32.6 hours, than that measured when dichlorvos was applied to the leaf surface.

Wheat. The fate of [^{14}C]dichlorvos applied to stored wheat grain was investigated (Rowlands, 1970). Grains at moisture levels of 18% and 10.6% were topically treated with [^{14}C]dichlorvos at a rate of 40 ìg ai/10 g sample. Treated samples were stored in sealed glass jars in darkness at 20◦C and sampled over a period of 10 days.

The uptake of dichlorvos was rapid at the higher moisture level. Within 2 days the water-extractable bound activity reached a maximum and remained stable for 7 days, after which time the phosphorylated protein was converted to a more stable demethyl from. The uptake rate decreased once all the protein had been phosphorylated, so excess dichlorvos would be lost from the grain by volatilization.

Dichlorvos broke down rapidly on grain to give mainly dimethyl phosphate (about 2 mg/kg at day 10) and phosphorylated protein derivatives, which were mainly water-soluble. Lesser amounts of demethyl-dichlorvos (about 0.5 mg/kg at day 10), monomethyl phosphate (about 0.2-0.25 mg/kg at day 10) and traces of phosphoric acid (undetectable-0.05 mg/kg at day 10) were also found. The water content did not affect the rate of degradation.

In an another study wheat and sorghum were treated commercially with 6 mg/kg dichlorvos. The loss of dichlorvos was faster from small grain bulks and from the surface of large bulks than within large bulks. Dichlorvos had a half-life of 54 hours on wheat and 27 hours on sorghum. It was also degraded in sealed containers that were not chemically inert (Desmarchelier, 1977).

Turf and lawn. Six 0.61 x 2.44 m (1.49 m^2) plots in California, USA, were sprayed with "Dichloron" (2.6% dichlorvos + 3.0% chlorpyrifos) at the maximum recommended rate (3.8 litre of product in 605.7 litre of water for 508 m^2 of lawn). Leaf samples were taken 0, 2, 6, 10, 24, 72 and 96 hours after application.

The dislodgeable foliar residue level was calculated from the residue concentration measured on the grass surface with a regression equation established before the experiment. The surface residue was 0.10 ìg/cm^2 immediately after application. This level dropped rapidly below 0.06 ìg/cm^2 after 2 hours and the residue was undetectable (<0.12 mg/kg) after 23 hours. In the air 1.9 ± 0.5 ìg/kg dichlorvos was detected immediately after spraying (Goh *et al*., 1986a)

Similar experiments were performed in lawn plots using all and half the recommended quantity of water, and with and without post-spray irrigation. The results showed the dissipation of the dislodgeable residue to be remarkably similar to that found in the previous experiments, suggesting that the dilution factor of the spray is of minor importance in the field dissipation of dichlorvos (Goh *et al*., 1986b).

In water

The hydrolysis of dichlorvos at a concentration of 10 mg/kg in aqueous media follows first-order kinetics. The rate of hydrolysis appears to be strongly influenced by the ionic strength of the solution. Half-lives at 30◦C and pH 1, 5, 7 and 9 were found to be 74, 50, 18 and 16 hours respectively. At 20◦C and pH 13 the half-life was 1.3 x 10^{-2} hours (Suter, 1981).

In micro-ecosystems containing sediments from a recultivated gravel pit and the drainage ditch of a fruit orchard, dichlorvos was very rapidly degraded to CO_2. After 16 days incubation, 76 and 69% of the applied 1.0 mg [^{14}C]dichlorvos/litre was mineralized to $^{14}CO_2$ in the two systems, respectively. Unchanged parent compound could be detected until day 7

(0.5%) and day 3 (3.1%) respectively. The following compounds occurred as intermediate products of this mineralization.

I phosphoric acid, 2,2-dichloroethyl methyl phosphate, sodium phospate, demethyl-dichlorvos.
II 2,2-dichloroethanol.
III 2,2-dichloroacetaldehyde.
IV dichloroacetic acid.

Contamination of surface waters with dichlorvos is unlikely to be more than transient owing to the rapid and extensive metabolism (Fritz, 1987c).

The photolytic degradation of dichlorvos was tested in water and aqueous methanolic solutions at 20°C. Dichlorvos was degraded with a half-life of about 6 hours in water, but was stable in methanolic solution (Ref. 76).

In soil

The leaching characteristics of dichlorvos in aged and non-aged BBA 2.1 soil were studied. [^{14}C]dichlorvos equivalent to l kg ai/ha was applied to soil and aged for 0, 2, 8 and 12 days. In a column leaching study 35%, 10%, 2% and <0.1% of the applied radioactivity was detected in the leachate, respectively. Up to 15% of the radioactivity in the leachate was dissolved $^{14}CO_2$. 2,2-dichloroethanol and demethyl-dichlorvos corresponded to about 10% and 1% respectively. Parent dichlorvos was rapidly mineralized in the soil, and was not detectable in any leachates. Within 2 days of ageing, 60-65% of the parent compound was detected as $^{14}CO_2$ (Fritz, 1987a).

The loss of dichlorvos in soil perfusion systems of Houston black clay under sterile and non-sterile conditions was reported (Lamoreaux and Newland, 1978). The perfusion technique involved continually perfusing a column of soil with an aqueous solution containing 1000 mg/l dichlorvos. The pesticide was extracted from the perfusing water. After 10 days 70% and 50% losses of dichlorvos were observed under non-sterile and sterile conditions respectively.

Dichlorvos was converted to dichloroethanol, dichloroacetic acid and ethyl dichloroacetate by a microbial enrichment derived from sewage (Lieberman and Alexander, 1983).

The degradation of dichlorvos was investigated in sterile and non-sterile standard 2.1 soil of the BBA and in a natural biologically active soil (Fritz, 1987b). After an incubation period of 2 days 1.2% of the unchanged parent compound could be detected in soil 2.1 and none in the second system. During the same period 60-61% of the applied radioactivity was measured as $^{14}CO_2$. The decomposition of the active ingredient was slower in sterile standard soil 2.1, where the half-life was 8.7 days and less than 1% $^{14}CO_2$ was formed.

On the basis of various soil and microbial degradation studies, the pathway shown in Figure 3 has been proposed for dichlorvos breakdown.

Figure 3. Proposed pathway for the breakdown of dichlorvos in soil.

In storage and processing

The persistence of dichlorvos on stored commodities is strongly dependent on the temperature and moisture content or relative humidity. For instance, a moisture content of 12% in wheat is roughly equivalent to 60-65% relative humidity (RH).

Cereals. The reported half-lives on wheat are 10 days at 25°C and 12% moisture content, 25 days at 21°C and 9.3% moisture content, and 1.8 days

at 35°C and 13.7%. The Australian Grain Industry uses an estimated half-life of 7 days at 30°C and 50% RH, and 28 days at 20°C and 50% RH (Webley, 1993).

In Switzerland stored wheat treated with 20 mg/kg dichlorvos was processed. The results are included in Table 12. After 14-16 days, between 3.1 and 3.6 mg/kg residue remained in the whole wheat. The flour prepared from the treated wheat contained 0.14-0.18 mg/kg, and the bread no measurable residues (<0.02 mg/kg).

The rapid disappearance of dichlorvos in comparison with most other grain protectants makes it difficult to obtain accurate measurements of persistence in a commercial trial. Preliminary laboratory-scale trials were therefore undertaken to determine the persistence on cereals and milled products, although in commercial practice the milled products are not directly treated. Dichlorvos was added directly in aqueous solution followed by tumbling. One kg of each commodity was treated at 6 and 12 mg ai/kg and the commodity was stored in closed screw-top glass jars at 0, 20 and 30°C. The initial moisture content of wheat lots ranged from 13.05 to 13.47%. The results are shown in Table 17.

Table 17. Disappearance of dichlorvos from wheat, chickpeas and milled products under laboratory conditions.

Commodity	Appl. rate, g ai/t	Storage temp. °C	Residues (mg/kg) after weeks of storage				
			1	2	4	6	8
Flour	1	0 20 30	1.0 0.2 1.2	 0.3	0.9	0.9	1.0
	2	0 20 30	1.8 0.5 0.1	2.0 0.5	2.3 0.2	2.3 0.1	1.8
Bran	6	0 20 30	7.5 4.0 0.3	9,5 1.9	8.4 1.0	7.8 0.4	1.6
Germ	6	0 20 30	6.3 1.8 1.8	4.3	5.1	4.5	1.8
Wheat	6	0 20 30	5.5 2.8 0.7	5.5 1.2 0.1	4.7 0.5	4.0 0.3	4.8
Wheat	12	0 20 30	10.2 4.7 2.0	10.4 3.0 0.3	10.7 1.4	 0.5	7.3
Chickpea	6	0 20 30	3.5 1.0 0.4	3.2 0.5 0.1	2.6 0.2	2.2 0.2	2.7 0.1
Chickpeas	12	0 20 30	7.3 2.9 1.2	7.5 1.5 0.3	6.0 0.9	5.7 0.5	4.2 0.1

The distribution and stability of dichlorvos in milling fractions were studied in a laboratory-scale experiment (Webley, 1993). 3 kg samples of wheat were treated with dichlorvos at 6 and 12 g ai/ton and were milled on a Buhler mill after 3 days. Flour, bran and wheat were stored at 20 and 30°C for 8 weeks in closed screw-topped glass jars. The results are shown in Table 18. Residues on flour and bran decreased to about 1/4-1/5 of the initial concentration after 2 weeks at 30°C and had a half-life of about 7 days at 20°C.

Table 18. Residues of dichlorvos in wheat and milling fractions during storage (laboratory-scale).

Commodity, [treatment rate]	Storage temp. °C	Residues (mg/kg) after weeks of storage				
		1	2	4	6	8
Wheat [12 g/ton]		7.2	2.2	2.2	0.4	
Flour	20 30	0.4 0.2	0.3	0.1		
Bran	20 30	9.7 11.7	7.5 1.2	1.1		0.2
Wheat [6 g/ton]		4.1	5.0	1.3		
Flour	20 30	0.3 0.1	0.2 0.1	0.1		
Bran	20	6.4	4.8	0.9		0.3

Pilot-scale milling trials were carried out with commercially treated wheat (Webley, 1993a). Dichlorvos was applied at rates of 6, 12 and 20 mg/kg on wheat lots of 6 tons each. The treated wheat was bagged and that from each treatment was divided into two 3-ton lots. The initial moisture content of the wheat was approximately 11% (Webley 1993b). Two days after treatment, analyses of lots treated at 6, 12 and 20 mg/kg showed residues of 3.4, 8.3 and 14.8 mg/kg respectively. The lots were milled 10 and 90 days after treatment. Arabic flat bread, steamed bread, yellow alkaline noodles and white salted noodles were prepared from the milled products. The results are summarized in Table 19.

Table 19. Dichlorvos residues (mg/kg) in pilot milling trials.

Appl. Rate (mg/kg)	6		12		20	
Interval to milling (days)	10	90	10	90	10	90
Commodity						
Wheat	2.5		5.2	0.9	6.1	1.0
Wheat, conditioned	0.7	0.1	1.4	0.2	2.1	0.3
Bran	4.1	0.5	7.3	1	11.5	1.4
Germ	2.6	0.3	5.0	0.8	7.0	1.0
Flour	0.3	<0.1	0.4	<0.1	0.6	0.1
Wholemeal	1.3	0.1	1.8	0.2	2.1	0.3
White-bread	<0.1	<0.1	0.2	<0.1	<0.1	<0.1
Wholemeal bread	0.2	<0.1	0.3	<0.1	0.3	<0.1
Flat bread	0.3	<0.1	0.5	<0.1	0.6	<0.1
Steamed bread	0.2	<0.1	0.3	<0.1	0.2	<0.1
Yellow alkaline noodles	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
White salted noodles	0.2	<0.1	<0.1	<0.1	0.3	<0.1

The results show a loss of dichlorvos in the milling process of about 60%. The loss in processing may be largely attributable to the scouring and conditioning process with probably some additional loss in the milling itself. In calculating the reduction in residue in the cooked products the differences in moisture content were taken into account by the following factors: 1.5 for white, wholemeal and steamed bread, 1.14 for flat bread,

and 1.0 for noodles. This gives a loss of 75% of the dichlorvos residue in the production of pan breads, about 65% in the case of Arabic flat bread (this tends to retain a higher residue owing to the short, 30-second, heat treatment), and 55% in white noodles. There is a 100% loss in yellow noodles owing to the alkali (1% sodium carbonate by flour weight). There were no detectable residues in any cooked products produced from wheat stored for 3 months after treatment, even after an application of 20 g/ton.

The cold-storage stability of dichlorvos and other pesticides was reviewed by Kawar *et al.* (1973). Wheat samples with moisture contents ranging between 13% and 17% were treated with dichlorvos at a rate of 24 mg/kg and stored at -15°C. After 2 months, 15 mg/kg (62.5% of the initial concentration of dichlorvos), was lost. When stored at 5°C, 50% and 80% was lost after seven and thirty days respectively.

In an another study, 50 mg/kg dichlorvos was added to wheat samples of 9.3-13.7% moisture content and stored at -15°C for 11 months. The losses were only between 2% and 22% at different moisture levels. Even though these two experiments showed two different levels of loss, they indicate that the breakdown of dichlorvos cannot be completely prevented even under cold storage conditions.

Cacao. Cacao beans were treated with dichlorvos at a rate of 10, 100 and 500 mg/kg and stored at room temperature. After 21 days, as shown in Table 13, no residues (<0.1 mg/kg) were detected in whole or roasted cacao beans treated at 10 mg/kg. Samples treated at 100 and 500 mg/kg contained 15 and 50 mg/kg after 21 days. About 99% of these residues was lost on roasting.

Endive. Cutting, washing and cooking endive containing 0.31 mg/kg dichlorvos resulted in 6% and 84% losses of residues after washing and cooking respectively (Netherlands, 1993)

Lettuce. Washing head lettuce containing 0.75 mg/kg dichlorvos removed 24% of the residue (Netherlands, 1993).

Chickpeas. Laboratory-scale trials were undertaken to determine the persistence of dichlorvos on chickpeas. Dichlorvos was added directly in aqueous solution followed by tumbling. One-kg portions of chickpeas were treated at 6 and 12 mg/kg and stored in closed screw-top glass jars at 0, 20 and 30°C. The initial moisture content of the commodities was not reported. The results are shown in Table 17.

Potatoes. In a Japanese trial (Tsumura *et al.*, 1992) potatoes were sprayed with a formulated mixture of dichlorvos at a concentration of 0.2% ai in the spray solution. After storing at room temperature for 86 days 0.92 mg/kg was still measured in whole potatoes, but about 97% of this surface residue could be removed by washing. When the washed potatoes (containing 0.03 mg/kg dichlorvos) were processed into dry and wet starch, no residues (<0.001 mg/kg) were detected.

Soya beans. In a USA trial (La Hue *et al.*, 1973), whole soya beans containing 0.92-1.5 mg/kg residue were processed into hulls, toasted hulls, flakes, crude oil and refined oil. Residues were 5.4-6.0 mg/kg, <0.02 mg/kg, 0.2 mg/kg, 0.55 mg/kg and <0.02 mg/kg in hulls, toasted hulls, flakes, crude oil and refined oil respectively.

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

Analyses by the Food Inspection Services of The Netherlands in 1976 showed the following residues (mg/kg) in fruit and vegetables (number of samples are in parentheses): endive <0.05 (2); lettuce <0.05 (9), >0.2 (1); celery <0.05 (1); spinach <0.05 (1); cauliflower <0.05 (1); leek <0.05 (1), <0.1 (1); cucumber <0.05 (7); paprika <0.05 (4); tomato <0.05 (2); radish <0.05 (2); blackberry <0.05 (1) (Netherlands, 1993).

METHODS OF RESIDUE ANALYSIS

Dichlorvos can be determined by many published multi-residue procedures.

The samples from supervised trials before 1972 were analyzed by the

automated cholinesterase inhibition method described in the 1970 Evaluations. Since then all samples have been analyzed by gas chromatography.

Plant material is Soxhlet-extracted with methylene chloride or acetone. After evaporating the solvent hexane is added. Clean-up involves extracting the organic phase with water and the aqueous phase with ethyl acetate, followed by drying of the organic phase with sodium sulphate. The final determination is by GLC using a phosphorus-specific flame-photometric detector. The recovery ranges from 70 to 100% and the limit of determination is 0.02-0.03 mg/kg. The method is valid for commodities such as peaches, tomatoes, rice and wheat grain (Ref. 79).

With further clean-up, based on partitioning between the water phase (saturated with sodium chloride) and hexane/ether 1:1, the same procedure was adopted for meat, cheese, milk and stored cereals. The limit of determination was 0.01-0.02 mg/kg with recoveries of 68%-110% (Ref. 77).

For food and beverages, the sample homogenized with water or a portion of the beverage is shaken vigorously in an Erlenmeyer flask after adding sodium sulphate, phosphotungstic acid and ethyl acetate. The final determination of dichlorvos is by GLC as before. The same method can be used with slight modification for blood and animal tissues with a detection limit of 0.01 mg/kg (Zweig, 1972).

For the determination of dichlorvos in air, the dichlorvos is trapped in glass tubes containing potassium nitrate connected to a vacuum pump. Sampling tubes should be stored below room temperature before analysis. After eluting the trapped dichlorvos with distilled hexane, determination is by GLC with flame-photometric detection. A limit of determination of 0.2 mg/m^3 was reported. The recovery was above 80% (Bryant and Minett, 1978).

Another method involves condensing dichlorvos in a U-tube immersed in salt-ice mixture (-20°C). Air is sucked into the tube with a vacuum pump for a period of 30-60 min. After washing the tube with acetone (5 ml), dichlorvos is determined by GLC using either an FPD or AFID. The limit of determination was 0.056 mg ai/m^3) with 80-100% recovery (Ref. 78).

NATIONAL MAXIMUM RESIDUE LIMITS

The reported national MRLs are summarized below. They all refer to dichlorvos.

The three-letter country codes are as follows: ARG-Argentina, AUL-Australia, AUS-Austria, BGD-Bangladesh, BOL-Bolivia, BRA-Brazil, CAN-Canada, CHI-Chile, COL-Columbia, CZE-Czechoslovakia, ECU-Ecuador, FRA - France, GRE-Greece, IND-India, INS-Indonesia, ITA-Italy, JOR-Jordan, LEB-Lebanon, MAL-Malaysia, NET-The Netherlands, NZE-New Zealand, NIR-Nigeria, PAK-Pakistan, PER-Peru, POL-Poland, POR-Portugal, SAF-South Africa, SPA-Spain, SUD-Sudan, SWI-Switzerland, TUR-Turkey, UAE-United Arab Emirates, VEN-Venezuela.

Country → Commodity ↓	ARG	AUL	AUS	BRA	CAN	CHI	CZE	FRA	GRE	IND
Apple			0.1						0.1	
Cabbage				0.5				0.1		
Cacao beans		5								
Cereal grains	2	2	2			2	2	2		1
Coffee beans, green		2								
Cotton									0.1	
Cucumber							0.1			
Eggs		0.05								
Fruits		0.1								

Country → Commodity ↓	ARG	AUL	AUS	BRA	CAN	CHI	CZE	FRA	GRE	IND
Grapes			0.1			0.1		0.1	0.1	
Lentils		2								
Lettuce		1						0.1	0.1	
Meat		0.05								
Milk, whole		0.02								
Mushroom		0.5					0.1			
Non-perishable packaged food					0.5[1] 2.0[2]					
Nuts		2								
Onion			0.1	0.5					0.1	
Other foods[3]		0.1								
Pea								0.1	0.1	
Peach			0.1					0.1	0.1	
Peanuts		2							0.1	
Peppers						0.5	0.1			
Potato						0.5				
Poultry		0.05								
Rice										1
Soya beans		2								
Tomato		0.5		0.5	0.25	0.5	0.1			
Vegetables (except lettuce)		0.5								

National MRLs (continued)

Country/Commodity	ITA	JOR	MAL	NET	NZE	PER	POL	POR	SAF	SPA
Apple	0.1	0.1						0.1		
Bean		0.5	0.1						0.3	
Berries + small fruits				0.1	2.0				0.1	
Brassica (excl.cabbage)		0.1			2.0	0.5				
Cabbage						0.5		0.1	0.1	
Cacao beans				5						
Cereal grains	2			2	2		2			2
Chicken meat				0.05						
Coffee beans (green)				2						
Cucumber		0.5		0.1			0.3			
Eggs				0.05						
Egg plant	0.1	0.5					0.1			
Fruits				0.1						
Grapes									0.1	
Legume vegetables (fresh)				2						

Country/Commodity	ITA	JOR	MAL	NET	NZE	PER	POL	POR	SAF	SPA
Meat				0.05						
Milk				0.02						
Mushroom				0.1			0.1			0.1
Onion		0.5								
Other food commodities				<0.02[4]						
Other vegetables				0.1						
Peanuts				2						
Peppers		0.5								
Pulses				2						
Tomato		0.5							0.1	
Wheat, wholemeal				0.5						

Country/Commodity	SWI	TUR	UAE	VEN
Apple	0.1			
Avocado	0.3			
Bean				0.3
Berries + small fruits	0.3			
Brassica (excl. cabbage)	0.3			
Cabbage				0.2
Cereals	2			
Cocoa beans	2			
Cucumber	0.3			
Dates			3	
Egg plant	0.3			
Grapes	0.1			
Lettuce	0.3	0.2		
Milk	0.01			
Onion	0.3			
Pea	0.3			
Peach	0.1			
Peanuts	0.3			0.2
Peppers	0.3			
Potato	0.3			0.3
Rice	2			
Sorghum	2			
Soya bean	2.0			
Tomato	0.3			

[1] <6% fat
[2] >6% fat
[3] Other foods for which no MRL is specified
[4] Below the limit of determination

APPRAISAL

Dichlorvos was previously evaluated at the 1965, 1966, 1967, 1969, 1970 and 1974 Joint Meetings and it is included in the CCPR periodic review programme.

Dichlorvos, an organophosphorus insecticide, combines both contact and stomach action and has a marked vapour action. It is effective against a broad spectrum of insect pests in the field and in stored products. In addition to plant and stored product protection, it is often used in public health vector control and in animal health for ectoparasite control. The compound is registered for use on over 30 commodities in many countries.

Residues from supervised field trials or specific studies were evaluated for 45 commodities.

Following foliar application at recommended or double rates, the

residues in most of the plant commodities tested (apples, avocados, beans, Brussels sprouts, cacao beans, cabbages, cauliflower, chilli peppers, cotton seed, cucumbers, dates, egg plants, lettuce, onions, peanuts, peas, rice, sorghum, soya beans, strawberries, tea, tomatoes, witloof chicory) were below the limit of determination (0.01-0.04 mg/kg, except tea 0.1 mg/kg). Detectable residue were reported in four commodities with maximum values of 0.05 mg/kg in cherries, 0.15 mg/kg in peaches, 0.08 mg/kg in grapes and 0.04 mg/kg in kale.

Residues in samples of endive, celery, spinach, cauliflower, leeks, cucumbers, paprika, tomatoes, radishes and blackberries moving in commerce in The Netherlands were below the limit of determination (<0.05 mg/kg). A residue of 0.1 mg/kg was found in one of 10 lettuce samples.

Following the post-harvest or indoor application of dichlorvos, residues were present in detectable amounts in beans (1.85 and 1.48 mg/kg after 14 and 21 days), wheat (0.41-5.0 mg/kg after 1-2 months), barley (0.14-0.74 mg/kg after 21 days), rice (0.12-0.23 mg/kg after 23-24 days), lettuce (0.2-0.4 mg/kg after 3-4 days) and mushroom (0.02-0.12 mg/kg after 1 day). The results of supervised trials on barley, wheat and rice were supported by the extensive studies in India on a wide variety of stored commodities including cereals.

The very limited data base for most of the crops and/or the discrepancy between the trial conditions and current GAP for beans, cacao beans, coffee beans, cotton seed, egg plant, indoor lettuce, peanuts, peppers and soya beans did not allow the estimation of maximum residue levels, although the Meeting took into account that the results are mutually supportive for many commodities.

The Meeting was also concerned about the lack of frozen storage stability tests of residues in fruit and vegetable samples in view of the period of several months which often elapsed between sampling and analysis, and the substantial decrease of dichlorvos residues in wheat samples stored at -15^{o}C.

In the absence of this information and as a result of the limited residue data, the previous recommendations for fruits and vegetables were withdrawn.

The animal metabolism studies showed that dichlorvos is readily absorbed, hydrolysed and effectively eliminated. The metabolic pathway was identical in the mammalian species studied. The studies on laboratory animals are discussed as part of the toxicological evaluation.

Residues of dichlorvos in the eggs of hens receiving a mist spray three times at a rate of 50 mg/m^{3} were first detected 2 days after the first treatment. The residue concentration varied from <0.03 to 0.11 mg/kg during the trial. There was no increase in the concentration following consecutive treatments, and the residue declined below the limit of determination 3 days after the last application. Three birds were killed 18 hours after the 3rd treatment and the breast and leg muscle analysed. The residues found were between <0.01 and 0.05 mg/kg. Egg production was not affected by the treatments.

Various tissues of pigs which had received a single oral dose of vinyl-1-^{14}C-dichlorvos (ca. 40 mg dichlorvos/kg feed) were analysed at 2, 7 and 14 days after the treatment. The ^{14}C content of the tissues expressed as dichlorvos varied from 2.5 mg/kg (in brain) to 33 mg/kg (in liver) after 2 days, and from 1.9 mg/kg to 9.7 mg/kg after 14 days, but no dichlorvos, demethyl-dichlorvos, dichloroacetaldehyde or dichloroacetic acid could be detected.

Three cows were sprayed for 31 consecutive days with 59 ml of 1% dichlorvos solution for the control of horn fly and mosquitoes. Milk samples were collected at 2 hours and 1, 2, 4, 8, 16, 24 and 31 days. Tissue and blood samples were taken one day after the final treatment. No dichlorvos was detected in any milk samples (<0.003 mg/kg) or body tissues (<0.002 mg/kg) from the treated cows.

Dairy cows were dosed orally at rates of 1.3, 1.8 and 2.6 mg/kg body weight with dichlorvos in the form of polyvinyl chloride pellets. The PVC formulation prevents substantial absorption by the animal, but may release the compound only much later in the manure. As the absorbed amount is

unknown the non-detectable dichlorvos residues (<0.04 mg/kg) in milk samples, collected between 1 and 14 days, cannot be related to the dose.

In a dermal application experiment, six cattle and two dairy cows were sprayed once with 15 litres of an emulsion of NUVAN 100 EC at a concentration of 1500 mg/kg. Cattle were slaughtered 1, 3 and 7 days after treatment. Milk samples from two dairy cows were taken 6 h and 1, 3 and 7 days after treatment (control 1 day before treatment). No measurable residues of dichlorvos were found in milk (<0.005 mg/kg) or in muscle, liver, kidney or fat (<0.02 mg/kg).

In plants, the main routes of degradation of dichlorvos were found to be cleavage of the P-C bond to form the major metabolite dimethyl phosphate, demethylation to monomethyl phosphate and phosphoric acid, demethylation to demethyl-dichlorvos (a minor pathway), and loss by volatilization.

Cotton plants of the Deltapine Smooth leaf variety were grown in a glasshouse and treated by injecting aqueous solutions of ^{32}P-labelled dichlorvos (100 µg) into the petioles of individual, fully expanded leaves. Leaf samples were collected 1, 24 and 48 hours after the treatment. The analyses showed 81.2% loss as volatiles, 12.3% as dimethyl phosphate (the major metabolite), 2.2% as phosphoric acid + methyl phosphate and 0.1% as demethyl-dichlorvos (minor metabolites) within 48 hours of application. No parent residue was detectable at this time. Non-extractable residues corresponded to less than 5% of the applied radioactivity.

Greenhouse-grown bean, potato, and tomato plants of 15 cm height, with or without roots, were placed in formulated 0.1-0,2% ^{32}P-dichlorvos. Dichlorvos was degraded with a half-life of 6.8, 4.6 and 6.8 hours on beans, tomatoes and potatoes, respectively. Dimethyl phosphate was a major metabolite detected during the 24-hour test period. Volatile radioactivity amounted to about 60% in all three plant species.

Wheat grain at moisture levels of 18% and 10.6% was topically treated with ^{14}C-dichlorvos at a rate of 40 ìg ai/10 g sample. Treated samples were stored in sealed glass jars in darkness at 20°C and sampled over a period of 10 days. the uptake of dichlorvos was rapid at the higher moisture level. Within 2 days the aqueous-extractable bound activity reached a maximum and remained stable for 7 days, after which the phosphorylated protein was converted to a more stable demethyl form. The uptake rate decreased once all the protein had been phosphorylated, so excess dichlorvos would be lost from the grain by volatilization.

Dichlorvos broke down rapidly on grain to give mainly dimethyl phosphate (about 2 mg/kg at day 10) and phosphorylated protein derivatives, which are mainly water-soluble. Lesser amounts of demethyl-dichlorvos (about 0.5 mg/kg at day 10), monomethyl phosphate (about 0.2-0.25 mg/kg at day 10) and traces of phosphoric acid (undetectable-0.05 mg/kg at day 10) were also found.

The hydrolysis of dichlorvos at a concentration of 10 mg/l in aqueous media follows first order kinetics. The rate of hydrolysis appears to be strongly influenced by the ionic strength of the solution. Half-lives at 30°C determined at pH 1, 5, 7 and 9 were 74, 50, 18 and 16 hours respectively. At 20°C and pH 13, the half-life value is 1.3×10^{-2} hours.

In micro-ecosystems containing sediments from a recultivated gravel pit and the drainage ditch of a fruit orchard, dichlorvos was very rapidly degraded to CO_2. After 16 days incubation, 76 and 69% of the applied 1.0 mg ^{14}C-dichlorvos/l was mineralized to $^{14}CO_2$ in the two systems, respectively. Unchanged parent compound could be detected until day 7 (0.5%) and day 3 (3.1%), respectively. The following dichlorvos metabolites occurred as intermediary products: phosphoric acid, mono(2,2-dichloroethyl) monomethyl ester, sodium salt, demethyl-dichlorvos, 2,2-dichloroethanol, 2,2-dichloroacetaldehyde and dichloroacetic acid. Contamination of surface waters by dichlorvos is unlikely owing to rapid and thorough metabolism.

The photolytic degradation of dichlorvos was studied in water and aqueous methanolic solutions at 20 °C. It was degraded with a half-life of about 6 hours in water but was stable in methanolic solution.

Dichlorvos was applied to soil at a rate corresponding to l kg ai/ha and aged for 0, 2, 8 and 12 days. In a column leaching study 35%, 10%, 2%

and <0.1% of the applied radioactivity was detected in the leachate respectively. Up to 15% of the radioactivity in the leachate was dissolved $^{14}CO_2$. 2,2-dichloroethanol and demethyl-dichlorvos corresponded to about 10% and 1% respectively. Parent dichlorvos was rapidly mineralized in the soil, and it was not detectable in any leachates. Within 2 days of ageing, 60-65% of the parent compound was detected as $^{14}CO_2$.

Dichlorvos was very rapidly degraded to the final mineralization product CO_2 in non-sterile standard soil 2.1 of the BBA and in a natural biologically active soil. After an incubation period of 2 days, 1.2% of the unchanged parent compound could be detected in soil 2.1 and none in the second system. During the same period 60-61% of the radioactivity applied as the parent compound was measured as $^{14}CO_2$. The decomposition of the active ingredient was slower in sterile standard soil 2.1. The half-life was 8.7 days. Less than 1% $^{14}CO_2$ was formed.

Dichlorvos was converted to dichloroethanol, dichloroacetic acid and ethyl dichloroacetate by a microbial enrichment derived from sewage.

The persistence of dichlorvos in stored commodities is strongly dependent on the temperature and moisture content or relative humidity (RH). A moisture content of 12% in wheat is roughly equivalent to 60-65% relative humidity. Reported half-lives are 10 days at 25°C and 12% moisture content, 25 days at 21°C and 9.3% moisture content, and 1.8 days at 35°C and 13.7%. The Australian Grain Industry uses an estimated half-life of 7 days at 30°C and 50% RH, and 28 days at 20°C and 50% RH.

Stored wheat lots treated with 6, 12 and 20 mg/kg dichlorvos were processed and the residues were analysed in milling fractions and baked products. The results indicate that the loss in processing may be largely attributable to the scouring and conditioning process. The ratios of dichlorvos residues in bran, germ and flour to wheat were on average 1.5, 1.0 and 0.1, respectively. The residues (mg/kg) detected were as follows: in white bread <0.02-0.2, in wholemeal bread and steamed bread 0.2-0.3, and in Arabic flat bread 0.3-0.6. In calculating the reduction in residue in the cooked products the differences in moisture content were taken into account by the following factors: 1.5 for white, wholemeal and steamed bread, 1.14 for flat bread, and 1.0 for noodles. This gives a loss of 75% of the dichlorvos residue in the production of pan breads, about 65% in the case of Arabic flat bread, which tends to retain a higher residue owing to the short (30 second) heat treatment, and 55% in white noodles. There is a 100% loss in yellow noodles owing to the alkali treatment (1% sodium carbonate by flour weight). There were no detectable residues in any cooked products produced from wheat stored for 3 months after treatment even at an application rate of 20 g/tonne.

In cacao beans about 99% of the residue was lost after roasting. Cacao butter contained 1.3-3.3% of the residues detected in the un-roasted cacao beans.

Washing removed 97%, 24% and 6% of the initial dichlorvos residues from potatoes, lettuce and endive. Cooking endive resulted in an 84% loss of residues.

Whole soya beans containing 0.92-1.5 mg/kg residues were processed into hulls, toasted hulls, flakes, crude oil and refined oil. Residues were 5.4 - 6.0 mg/kg, <0.02 mg/kg, 0.2 mg/kg, 0.55 mg/kg and <0.02 mg/kg respectively.

Storage stability tests of dichlorvos in wheat samples with moisture contents ranging between 13% and 17% indicated that 2 months after treatment 62.5% of the initial concentration (20 mg/kg) was lost at -15°C. When stored at 5°C, 50% and 80% was lost after seven and thirty days respectively. In another study, 50 mg/kg dichlorvos was added to wheat samples of 9.3-13.7% moisture content and stored at -15°C for 11 months. The losses were between only 2% and 22% at different moisture levels.

Although these two experiments showed two different levels of loss, they indicated that the breakdown of dichlorvos cannot be completely prevented even under cold storage conditions.

Dichlorvos can be determined by many published multi-residue procedures.

The samples from supervised trials before 1972 were analysed by an automated cholinesterase-inhibition method described in the 1970 Evaluations. Since then all samples have been analysed by gas chromatography using phosphorus-specific flame-photometric detectors.

RECOMMENDATIONS

On the basis of the data on residues from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits.

Definition of the residue: dichlorvos.

Commodity		Recommended MRL (mg/kg)		
CCN	Name	New	Previous	PHI, days
VP 0061	Beans	W	0.5 for vegetables	
SB 0715	Cacao beans	W	5	
GC 0080	Cereal grains	5 (Po)	2	30
SB 0716	Coffee beans	W	2	
PE 0112	Eggs	W	0.05	
AO2 0001	Fruits	W	0.1	
MM 0814	Goat meat	W	0.05	
VD 0533	Lentil (dry)	W	2	
VL 0482	Lettuce, Head	W	1	
MM 0097	Meat of cattle, pigs & sheep	W	0.05	
MM 0095	Meat	0.05*	-	
ML 0106	Milks	0.02*	0.02	
VD 0541	Soya bean (dry)	W	2 Po	
AO1 0002	Vegetables (except..)	W	0.5 [1]	
CF 0654	Wheat bran	10		
CF 1211	Wheat flour	1		
CF 1210	Wheat germ	10		
CF 1212	Wheat wholemeal	2		

Note: W: the previous recommendation is withdrawn.
[1] Except otherwise listed.

FURTHER WORK OR INFORMATION

Desirable

1. Storage stability tests carried out on major commodities at or below -18°C.

2. Residue data on milled products of cereals other than wheat.

3. An animal transfer study on poultry.

REFERENCES

1. Bayer AG. 1964. Mushrooms, Niederlande. Project Report 248-64. Unpublished.

2. Bayer AG 1964. Rueckstandsanalyse, Rosenkohl (Rosen), Holland. Biolgisches Institut der Farbenfabriken. Project Report 203/64. Unpublished.

3. Bayer AG, 1986. Dichlorvos in Avocados. Dept. of Primary Industries, Australia. Project Report PES-2.01.05. Unpublished.

4. Bryant, R.J and Minett W., 1978. A gas chromatographic method for the determination of dichlorvos in air. Pesitc. Sci. 9, 525-528.

5. Bull D.L and Ridgway R.L. 1969. Metabolism of trichlorfon in animals and plants. J.Agric. Food Chem. 17, 837-841.

6. Ciba-Geigy Ltd., Basle 1971. NOGOS 50 EC, apples, Switzerland 1970, Project Report RVA-97-70 Unpublished.

7. Ciba-Geigy Ltd., Basle 1971. NOGOS 50 EC, apples, Switzerland Project Report RVA-100/71. Unpublished.

8. Ciba-Geigy Ltd., Basle 1992. NOGOS 500 EC, apple, Switzerland Project Report 1011-92. Unpublished.

9. Ciba-Geigy Ltd., Basle 1992. NOGOS 500 EC, apple, Switzerland Project Report 1012-92. Unpublished.

10. Ciba-Geigy Ltd., Basle 1992. NOGOS 500 EC, apple, Switzerland Project Report 1013-92. Unpublished.

11. Ciba-Geigy Ltd., Basle. 1970. NUVAN 7, dry beans, Switzerland Project Report RVA-99-70. Unpublished.

12. Ciba-Geigy Ltd., Basle. 1992. NOGOS EC 500, common beans, Switzerland. Project Report 1016-92. Unpublished.

13. Ciba-Geigy Ltd., Basle. 1981. NUVAN 100 EC, cauliflower, India. Project Report RVA-1152-80. Unpublished.

14. Ciba-Geigy Ltd., Basle. 1981. NUVAN 100 EC, cauliflower, India.Project Report RVA-1153-80. Unpublished.

15. Ciba-Geigy Ltd., Basle. 1970. NOGOS 50 EC, brussels sprouts, Switzerland. Project Report RVA-88-70. Unpublished.

16. Ciba-Geigy Ltd. Basle. 1965. NOGOS 50 EC, residues in cauliflower and brussels sprouts, Switzerland. Project Report CCI-1572-2. Unpublished.

17. Ciba-Geigy Ltd., Basle. 1992. NOGOS EC 500, cabbage, Switzerland. Project Report 1025-92. Unpublished.

18. Ciba-Geigy Ltd., Basle. 1980. NOGOS 100 EC, cherries, South Africa. Project Report RVA-1156-79. Unpublished.

19. Ciba-Geigy Ltd., Basle. 1980. NOGOS 100 EC, cherries, South Africa. Project Report RVA-1157-79. Unpublished.

20. Ciba-Geigy Ltd., Basle. 1968. NOGOS 50 EC, strawberries, Switzerland. Project Report EXP-4367. Unpublished.

21. Ciba-Geigy Ltd., Basle. 1979. Malathion, wheat, Argentina. Project Report RVA-4027-79. Unpublished.

22. Ciba-Geigy Ltd., Basle. 1975. NUVAN 7, wheat, Austria. Project Report IAR-341-75. Unpublished.

23. Ciba-Geigy Ltd., Basle. 1969. NUVAN 7, barley, France. Project Report RVA 53-69. Unpublished.

24. Ciba-Geigy Ltd., Basle. 1969. NUVAN 7, barley, Switzerland. Project Report RVA-44-69. Unpublished.

25. Ciba-Geigy Ltd., Basle. 1972. NUVAN 7, barley, Switzerland. Project Report RVA-2-72.

26. Ciba-Geigy Ltd., Basle. 1973. NUVAN 7, barley, Switzerland. Project Report RVA-16-73.

27. Ciba-Geigy Ltd., Basle. 1976. Dichlorvos, determination of dichlorvos in barley after application of NUVAN 7, trial silo Dintikon, CH, V 82.

28. Ciba-Geigy Ltd., Basle. 1969. NUVAN 7, wheat, Switzerland. Project Report RVA-38/69.

29. Ciba-Geigy Ltd., Basle. 1970. NUVAN 7, wheat, Switzerland. Project Report RVA-50-70.

30. Ciba-Geigy Ltd., Basle. 1975. NUVAN 7, wheat, Switzerland. Project Report RVA-312-75.

31. Ciba-Geigy Ltd., Basle. 1975. NUVAN 7, wheat, Switzerland. Project Report RVA-315-75.

32. Ciba-Geigy Ltd., Basle. 1976. NUVAN 7, wheat, Exp. Nr. 745/76, Switzerland. Project Report IAR 320-76.

33. Ciba-Geigy Ltd., Basle. 1977. CGA 20168 + C-177, feed-grain, Switzerland. Project Report RVA 615/77.

34. Ciba-Geigy Ltd., Basle. 1977. CGA 20168 + C-177, wheat, Switzerland. Project Report IAR 422-77.

35. Ciba-Geigy Ltd., Basle. 1984. NUVAN 100 EC, cocoa, Brazil. Project Report RVA 1259-82.

36. Ciba-Geigy Ltd., Basle. 1966. NUVAN, Rueckstaende in Kakaobohnen und deren Verarbeitungsprodukten, Switzerland. Project Report 7-10-1966. Unpublished.

37. Ciba-Geigy Ltd., Basle. 1972. NUVAN, Residues of dichlorvos in cocoa beans and processing products of cocoa beans, Switzerland. Project Report 25-4-72. Unpublished.

38. Ciba-Geigy Ltd., Basle. 1976. Curative treatment of infested rows of coffee using NUVAN 7 and CGA 29168, Switzerland. Project Report 9.25-VS-121. Unpublished.

39. Ciba-Geigy Ltd., Basle. 1976. Dichlorvos + phosphamidon, cotton seeds, India. Project Report RVA-372-76. Unpublished.

40. Ciba-Geigy Ltd., Basle. 1977. Dichlorvos + diazinon, cotton seeds, India. Project Report RVA-39-77. Unpublished.

41. Ciba-Geigy Ltd., Basle. 1992. NOGOS 500 EC, cucumbers, Switzerland. Project Report 1017-92. Unpublished.

42. Ciba-Geigy Ltd., Basle. 1992. NOGOS 500 EC, cucumbers, Switzerland. Project Report 1018-92. Unpublished.

43. Ciba-Geigy Ltd., Basle. 1972. NOGOS 50 EC/ 100 EC, dates, Iraq. Project Report RVA-115-72. Unpublished.

44. Ciba-Geigy Ltd., Basle. 1981. NUVAN 100 EC, egg plants, India. Project Report RVA-1150-80.

45. Ciba-Geigy Ltd., Basle. 1981. NUVAN 100 EC, egg plants, India. Project Report RVA-1151-80. Unpublished.

46. Ciba-Geigy Ltd., Basle. 1971. NOGOS 50 EC, grapes, France. Project Report RVA-81-71. Unpublished.

47. Ciba-Geigy Ltd., Basle. 1973. NOGOS 50 EC, grapes, Switzerland. Project Report RVA-5-73. Unpublished.

48. Ciba-Geigy Ltd., Basle. 1992. NOGOS 500 EC, grapes, Switzerland. Project Report 1014-92. Unpublished.

49. Ciba-Geigy Ltd., Basle. 1972. NOGOS 50 EC, lettuce, Switzerland. Project Report RVA-74-72. Unpublished.

50. Ciba-Geigy Ltd., Basle. 1992. NOGOS EC500, head-lettuce, Switzerland. Project Report 1019-92. Unpublished.

51. Ciba-Geigy Ltd., Basle. 1992. NOGOS EC500, head-lettuce, Switzerland. Project Report 1020-92. Unpublished.

52. Ciba-Geigy Ltd., Basle. 1992. NOGOS EC500, onion, Switzerland. Project Report 1023-92. Unpublished.

53. Ciba-Geigy Ltd., Basle. 1987. NOGOS EC500, onion, Switzerland. Project Report OGS 19851, 250 EC, onions, Italy. Project Report RR-1253-86. Unpublished.

54. Ciba-Geigy Ltd., Basle. 1992. NOGOS 500EC, peas, Switzerland. Project Report 1015-92. Unpublished.

55. Ciba-Geigy Ltd., Basle. 1969. NOGOS 50 EC, peaches, Switzerland. Project Report RVA-28-69. Unpublished.

56. Ciba-Geigy Ltd., Basle. 1970. NOGOS 50 EC, peaches, Switzerland. Project Report RVA-78-70. Unpublished.

57. Ciba-Geigy Ltd., Basle. 1974. NOGOS 100 EC, groundnut pods, India. Project Report RVA-135-74. Unpublished.

58. Ciba-Geigy Ltd., Basle. 1970. NUVAN 7, stored groundnut, Switzerland. Project Report RVA-98-70. Unpublished.

59. Ciba-Geigy Ltd., Basle. 1974. NOGOS 100 EC, chilli, India. Project Report RVA-137-74. Unpublished.

60. Ciba-Geigy Ltd., Basle. 1992. rice, Bangladesh. Project Report 1086-91. Unpublished.

61. Ciba-Geigy Ltd., Basle. 1992. rice, Bangladesh. Project Report 1087-91. Unpublished.

62. Ciba-Geigy Ltd., Basle. 1992. rice, Colombia. Project Report 1106-91. Unpublished.

63. Ciba-Geigy Ltd., Basle. 1984. NUVAN EC, rice, grain, straw, India Project Report RVA-1080-83. Unpublished.

64. Ciba-Geigy Ltd., Basle. 1984. NUVAN EC, rice, grain, straw, India Project Report RVA-1081-83. Unpublished.

65. Ciba-Geigy Ltd., Basle. 1965. NUVAN, residues in stored rice and cocoa beans, CH. Project Report 11-11-65. Unpublished.

66. Ciba-Geigy Ltd., Basle. 1966. NUVAN, residues in stored rice (Supplement 1), CH. Project Report 31-1-66. Unpublished.

67. Ciba-Geigy Ltd., Basle. 1980. NUVAN 100 EC, sorghum (grain, straw) India. Project Report RVA-1136-80. Unpublished.

68. Ciba-Geigy Ltd., Basle. 1980. NUVAN 100 EC, sorghum (grain,straw), India. Project Report RVA-1137-80.

69. Ciba-Geigy Ltd., Basle. 1984. NUVAN EC, soya beans, India. Project Report RVA-1078-83.

70. Ciba-Geigy Ltd., Basle. 1984. NUVAN EC, soya beans, India. Project Report RVA-1078-83A.

71. Ciba-Geigy Ltd., Basle. 1984. NUVAN EC, soya beans, India. Project Report RVA-1079-83.

72. Ciba-Geigy Ltd., Basle. 1984. NUVAN EC, soya beans, India. Project Report RVA-1079-83A.

73. Ciba-Geigy Ltd., Basle. 1992. NOGOS EC500, tomatoes, Switzerland Project Report 1021-92.

74. Ciba-Geigy Ltd., Basle. 1992. NOGOS EC500, tomatoes, Switzerland Project Report 1022-92.

75. Ciba-Geigy Ltd., Basle. 1986. Determination of residues of dichlorvos in fat, milk and animal tissues after single spray application of NUVAN 100 EC on dairy cows. Residue Report 4013/86.

76. Ciba-Geigy Ltd., Basle. 1970. Photodecomposition of dichlorvos, phosphamidon, dicrotophos, monocrotophos, chlorfenvinphos, dioxacarb, C-10015 and C-17018 in aqueous and methanolic solutions. Project Report SPR-09-70. Unpublished.

77. Ciba-Geigy Ltd., Basle. 1977. Dichlorvos, gas chromatographic residue method for plant material, meat, cheese, milk and stored cereals. REM-5-77. Unpublished.

78. Ciba-Geigy Ltd., Basle, 1979. Dichlorvos, gaschromatographische Betimmung in Raumluft. REM-4-79.

79. Ciba-Geigy Ltd.,Basle, 1970. NOGOS, gas chromatographic residue method for plant material. Method Description REM-04-70. Unpublished.

80. Ciba-Geigy Ltd. 1987. Determination of residues of dichlorvos in fat, milk, animal tissues after single spray application of Nuvan 100 EC, Switzerland. Project Report 4013/86.

81. Ciba-Geigy France. 1973. NOGOS 50 EC, peaches, France. Project Report 3010. Unpublished.

82. Ciba-Geigy France. 1973. NOGOS 50 EC, peaches, France. Project Report 3013, Unpublished.

83. Ciba-Geigy Ltd., France. 1974. NOGOS 50 EC, cereals, France. Project Report 74-2009. Unpublished.

84. Ciba-Geigy Ltd., France. 1982. C-177 + chlorpyriphos, wheat, France. Project Report 58-82. Unpublished.

85. Ciba-Geigy SA, France, 1986. Courbes de dissipation des m.a de NUVAN duree dans les grains de ble stockes. Project Report 16-7-86. Unpublished.

86. Ciba-Geigy UK. 1964. Dichlorvos, (DDVP), a report on glass house trials done in the United Kingdom. Project Report 0-2763/1D. Unpublished.

87. Ciba-Geigy UK. 1964. NOGOS 50 EC, lettuce, Gr. Britain. Project Report 2763/1D.

88. Ciba-Geigy UK. 1964. NOGOS 50 EC, mushrooms, Gr. Britain. Project Report 2763/1D. Unpublished.

89. Crips, S., Tarrant, K.R. 1971. The determination of residues of dichlorvos and malathion in wheat grain by gas-liquid chromatography. Analyst 96, 310-313.

90. Dedek W., George C., Grahl R. 1979. Comparative degradation and metabolism of 32P-labelled butonate, trichlorphone and dichlorvos in crop plants. Biochem. Physiol. Pflanzen 174, 707-722.

91. Desmarchelier J.M. 1977. A model of the breakdown of dichlorvos on grain. Australian J. FAO Exp.Agric. Animal Husbandry 17, 818-825.

92. FAO/WHO, 1971, 1970 Evaluations of some pesticide residues in food, FAO Rome.

93. Fritz R. 1987a. Leaching characteristics of dichlorvos aged in soil. Bayer AG. Unpublished.

94. Fritz R. 1987b. Degradation of [1-^{14}C-] dichlorvos in soils under aerobic conditions, BAYER AG, Project Report 2836. Unpublished.

95. Fritz R., Brauner A. 1987. Study on the metabolism of dichlorvos in soil under aerobic conditions BAYER AG, Project Report 2857. Unpublished.

96. Fritz R. 1987c. Aerobic metabolism of dichlorvos in an aquatic

ecosystem model BAYER AG, Project Report 2893. Unpublished.

97. Goch K.S. *et al.*, 1986. Dissipation of dislodgeable foliar residue of chlorpyrifos and dichlorvos on turf. Bull.Environ.Contam. Toxicol. 37, 27-32.

98. Goh K.S. *et al.*, 1986a. Dissipation of dislodgeable foliar residue for chlorpyrifos and dichlorvos treated lawn: Implication for safe reentry. Bull.Environ.Contam.Toxicol. 37, 33-40.

99. Hurt E. D. 1964. Studies on the toxicity of DDVP mist spray to hens in lay: l) acute toxicity, 2) residues in eggs and tissue. Ciba-Geigy Ltd., CCI No.:0-024/1. Unpublished.

100. Hussey N. W and Hughes J. T. 1964. Investigations on the use of dichlorvos in the control of mushroom, phorid, Megaselia halterata, Gr. Britain N.W. Ann. Appl. Biol. 54, 129-139.

101. Hussey N. W and Hughes J.T. 1963. Report on observation made following the application of DDVP in mushroom houses Gr. Britain Glasshouse Crops Research Institute. Project Report 0544D. Unpublished.

102. Ivey M.C and Eschle J.L. 1970. Residues of dichlorvos in milk and tissues of diary cows treated to control insect pest. J.Econ. Entomol. 63, 1729-1730.

103. Kawar N.S., de Batista, G.C. and Gunter, F.A. 1973. Pesticide stability is cold-stored plant parts, soil, and dairy products, and in cold-stored extractive solutions. Residue Review 48, 45 -77.

104. La Hue, D. W., Larry D., Kirk D and Mustakas G.C. 1973. Fate of dichlorvos residues during milling and oil extraction of soya beans, USA. Environ. Entomol. 4, 1. 11-14.

105. Lamoreaux R.J and Newland L.W. 1978. The fate of dichlorvos in soil. Chemosphere 10, 807-814.

106. Lieberman M.T and Alexander M.A. 1983. Microbial and nonenzymatic steps in the decomposition of dichlorvos (2,2-dichlorovinyl 0,0-dimethyl phosphate) J. Agric. Food Chem. 31, 265-267.

107. Lloyd J.E and Matthysse J.G. 1971. Residues of dichlorvos, diazinon and dimetilan in milk of cows fed PVC-insecticide feed additives. J.Econ.Entomol. 64, 821 -822.

108. Maier-Bode,. 1972. NOGOS 50 EC, Salat, Deutschland. Project Report 1972-23-1-7. Unpublished.

109. Netherlands, 1993, Information of The Netherlands on dichlorvos.

110. Potter, J.C. *et al*, 1973. Carbon-14 Balance and residues of dichlorvos and its metabolites in pigs dosed with Dichlorvos-^{14}C. J.Agric. Food Chem. 21, 163-166.

111. Rajak, R.L. 1973. Studies concerning the residues of dichlorvos in foodstuffs. Indian Grain Storage Institute, Hapur (U.P.), India. Progress Report of the FAO-Industry (CIBA) Cooperative programme.

112. Rowlands D.G. 1970. The metabolic fate of dichlorvos on stored wheat grain. J.Stored Prod. Res. 6, 19-32.

113. Schulten G.G.M and Kuyken W. Dichlorvos resin strips for control of cocoa moth Ephestia elutella, Literature Reference. Netherlands Int. Pest Control, 8, No. 3, 1966.

114. Shell Chimie, Berre, 1975. Residues of gardona and dichlorvos is stored wheat form Brazil. Project Report BEGR.0028.75. Unpublished.

115. Shell Chimie, 1977. Residues of dichlorvos in stored wheat from Germany. Arising from the use of VAPONA strips. Project Report BEGR.0001.77. Unpublished.

116. Shell Chimie, 1977. Residues of dichlorvos in stored rye from Germany. Arising from the use of VAPONA strips. Project Report

BEGR.0002.77. Unpublished.

117. Suter P. 1981. Rate of hydrolysis of dichlorvos (NOGOS, NUVAN) under laboratory conditions Ciba-Geigy Ltd., Basle. Project Report 29-81. Unpublished.

118. Tsumura-Hasegawa Y., Tonogai Y., Nakamura Y and Ito Y. 1992. Residue levels of dichlorvos, chlorpropham, and pyrethrines in post-harvest treated potatoes during storage or processing into starch. J. Agric. Food Chem. 40, 1240-1244.

119. Webley, D. 1993a. Dichlorvos residues in wheat and milled products after commercial treatment, milling and baking, Report No. AGT/93/1, Academy of Grain Technology.

120. Webley, D. 1993b. Personal communication 10/9/93.

121. WHO, 1989. Environmental Health Criteria 79, Dichlorvos. Ciba-Geigy Ltd., Basle. 1992. Dichlorvos, Biological Data and Toxicological Evaluation. WHO submission by, Ciba Geigy.

122. Zongmao, C and Haibin, W. 1968. Factors affecting residues of pesticides in tea. Pestic. Sci. 23, 109-118.

123. Zweig G. ed 1972. Vapona^{R} Insecticide in Analytical methods for pesticides and plant growth regulators, Vol VI, 529-541 (1972) Academic Press NY.

ETOFENPROX (185)

IDENTITY

Chemical name: 2-(4-ethoxyphenyl)-2-methylpropyl 3-phenoxybenzyl ether (IUPAC).
1-[[2-(4-ethoxyphenyl)-2-methylpropoxy]methyl]-3-phenoxybenzene (CA)

CAS No: 80844-07-1

Synonyms: MTI-500; "Trebon"

Structural formula:

Molecula formula: $C_{25}H_{28}O_3$

Molecular weight: 376.49

Physical and chemical properties

Pure active ingredient

Physical state:	white crystalline powder		
Vapour pressure:	2.4×10^{-4} mm Hg at 100°C		
Melting point:	36.4 - 38.0°C		
Octanol/water partition coefficient:	log P_{ow} = 7.05 at 25°C		
Solubility: (g ai/100 ml sovent)	water	$<10^{-7}$	at 25°C
	acetone	780	at 25°C
	ethanol	15	at 25°C
	acetonitrile	64	at 10°C
	n-hexane	270	at 25°C
	xylene	480	at 25°C
	carbon disulphide	340	at 10°C
	methanol	6.6	at 25°C
	chloroform	900	at 25°C
	dichloromethane	370	at 10°C
	benzene	240	at 10°C
	tetrahydrofuran	280	at 10°C

ethyl acetate 600 at 25°C

Specific gravity: solid (23.0°C): 1.157 g/ml
liquid (40.1°C): 1.067 g/ml

Hydrolysis: stable in aqueous 1N NaOH or 1N HCl for at least 10 days.

Stability to heat: no loss during at least 3 months storage at 80°C. Partial degradation at 100°C.

Photolysis: When [^{14}C]etofenprox was exposed to high-intensity lamps at 30000 lux, the half-life was approximately 4 days.

Technical material

Purity: Typically 96.3% etofenprox. Impurites <1%

Formulations

Wettable powder	20%
Emusifiable concentrate	20%

USE PATTERN

Etofenprox is used as an insecticide against many insect pests on a broad range of crops such as rice, fruits, vegetables, corn, soya beans and tea. The compound is active against Lepidoptera, Hemiptera, Coleoptera, Diptera, Thysanoptera and Hymenoptera at low rates. The compound is particularly effective against strains of rice green leefhopper and planthoppers resistant to organophosphorus or carbamate insecticides owing to its pyrethroid-like activity.

Etofenprox is mostly formulated as a 20% wettable powder or 20% emulsifiable concentrate for use on all types of crops, but in some countries 10 or 30% formulations are used.

Etofenprox is registered in several countries. Registered use patterns are shown in Table 1.

Table 1. Registered uses of etofenprox.

Crop	Country	Application			PHI, days
		No	Rate, kg ai/ha	Spray conc., kg ai/hl	
Apple	Hungary	1-2	0.15	0.01-0.05	14
	Japan	1-3	1.0-1.2	0.02	14
	Poland	1	0.14	0.009	14
Cabbage	Japan	1-3	0.4-0.5	0.02	3
	Spain	3-4	0.18-0.45	0.012-0.03	3
Chinese cabbage	Japan	1-3	0.4-0.8	0.02	7
Citrus	Spain	2	0.48-1.2	0.021-0.03	14
Corn - see Maize					
Cucumber	Japan	1-3	0.5	0.02	1

Crop	Country	Application			PHI, days
		No	Rate, kg ai/ha	Spray conc., kg ai/hl	
Egg plant	Japan	1-3	0.4	0.02	1
	Spain	3-4	0.18-0.45	0.012-0.03	3
Grape	Japan	2	0.4-0.6	0.02	
Maize	Japan	1-4	0.5	0.02	7
Mandarin	Japan	1-3	1.0-1.6	0.02	14
Olive	Spain	1	0.075-0.11	0.0075-0.011	14
Onion	Japan	4	0.3	0.02	
Orange	Japan	1-3	1.0-1.6	0.02	14
Pea (young pods)	Japan	1-2	0.3	0.02	1
Peach	Japan	1-3	0.8	0.02	14
Pear	Japan	1-3	0.8-1.0	0.02	14
Persimmom	Japan	1-3	1.0	0.02	30
Pome fruit	Spain	2-3	0.32-0.45	0.02-0.03	14
Potato	Hungary	1-2	0.045-0.15	0.08-0.05	3
	Japan	1-3	0.3-0.6	0.02	14
	Poland	1	0.03-0.09	0.01-0.03	14
Radish (Japanese)	Japan	1-3	0.3	0.02	21
Rape	Poland	1	0.03-0.09	0.01-0.06	14
Rice	Japan	1-3	0.4	0.02	21
	Spain	1-2	0.15-0.23		14
Soya bean (young pods)	Japan	1-2	0.3	0.02	14
Sugar beet	Japan	1-3	0.3	0.02	14
Tea	Japan	1-2	0.4	0.02	7[1]
Tomato	Japan	1-2	0.4	0.02	1
	Spain	3-4	0.18-0.45	0.012-0.03	3
Wheat	Japan	1-2	0.4	0.02	14

[1]before picking

RESIDUES RESULTING FROM SUPERVISED TRIALS

Supervised trials were carried out on several types of crop in Japan by the manufacturer, Mitsui-Toatsa Chemicals Inc., and all trials were reported by the Japan Food Research Laboratories. References in the Tables to the trials in Japan all refer to the date of the reports evaluated. All trials on crops in Japan were carried out at two experimental stations but in the same year.

Trials were also carried out on apples and potatoes in Hungary at Plant Protection and Agricultural Stations, and on potatoes and rape in Poland by the Poznan Institute of Plant Protection.

In all experiments analyses were by methods which determined the parent compound and metabolites containing the 3-phenoxybenzyl moiety. In all experiments in Japan studies were carried out to determine the stabiltiy of residues of etofenprox in collected samples.

Results in the Tables are underlined when the treatment was in accordance or aproximately in accordance with the approved use and recommended withholding period in the country in which the trial was carried out.

Citrus fruits. The only trials on citrus fruits were carried out on mandarins in Japan and with the approved use and recommended pre-harvest interval. Residues were determined in pulp and peel, and the level of residues in the whole fruit was calculated. With a pre-harvest interval of 2 weeks residues were below the limit of determination in the pulp and 6.6-11.4 mg/kg in the peel, equivalent to 1.5-1.9 mg/kg in the whole fruit (Table 2).

Pome fruits. Field trials were carried out on apples in Hungary and Japan and in both countries with treatments in conformity with approved uses, which were quite different in the two countries. Residues in apples in Japan after treatment with 1.0-1.2 kg ai/ha and a PHI of 2 weeks were 0.4-0.8 mg/kg, and residues in apples from Hungary after treatment with 0.15 kg ai/ha and the same PHI were 0.1-0.2 mg/kg (Table 2).

Trials were carried out on pears in Japan with the approved use, 0.8-1.0 kg ai/ha, giving residues of 0.2-0.5 mg/kg (Table 2).

Peaches. Trials were carried out in Japan on peaches with the approved use (0.8 kg ai/ha). Residues were determined separately in the pulp and peel with no calculation of the residues in the whole fruit and no information about the ratio of the weight of the peel to that of the pulp. After 2 weeks residues were undetectable in the pulp and 3.7-6.8 mg/kg in the peel (Table 2).

Grapes. Residues in grapes from trials in Japan were from 0.6 to 4 mg/kg from applicatin at 0.4-0.6 kg ai/ha. No information was given about the recommended pre-harvest interval in Japan (Table 2).

Persimmons. Field trials were carried out in Japan on persimmons in conformity with the approved use. After 28 days, which is the recommended PHI in Japan, residues were 0.56-0.70 mg/kg (Table 2).

Vegetables except root and tuber vegetables. Field trials were carried out in Japan on onions, cabbage, cucumbers, egg plants, tomatoes, chinese cabbage, peas and soya beans, all in accordance with approved uses.

Residues in onions in trials with 4 applications at 0.3 kg ai/ha were all below the limit of determination (Table 3).

Residues in cabbages after treatment with 0.4-0.5 kg ai/ha

and the recommended PHI of 3 days were 0.21-0.32 mg/kg (Table 3).

Residues in cucumbers after treatment with 0.5 kg ai/ha and a PHI of 1 day were 0.12-13 mg/kg (Table 3).

Residues in egg plants after treatment with 0.4 kg ai/ha and a PHI of 1 day were 0.15-0.48 mg/kg (Table 3).

Residues in tomatoes after treatment with 0.4 kg ai/ha and a PHI of 1 day were 0.35-1.9 mg/kg (Table 3).

Residues in chinese cabbage after treatment with 0.4 and 0.8 kg ai/ha and a PHI of 7 days were 0.08-0.15 mg/kg (Table 3).

Residues in peas with pods after treatment with 0.3 kg ai/ha and a PHI of 1 day were 0.34-0.79 mg/kg (Table 3).

Residues in soya beans were determined in unripe and mature beans. Soya beans were treated with 0.3 kg ai/ha and residues after a pre-harvest interval of 14 days were 1.0-1.1 mg/kg in unripe beans and below the limit of determination in mature beans (Table 3).

Potatoes. Field trials on potatoes were carried out in Hungary, Japan and Poland, all at approved application rates, but only in Japan were samples taken at the recommended pre-harvest interval.

In Hungary potatoes were treated with 0.15 kg ai/ha and samples were taken 7 days and later after the last application, whereas the recommended PHI is 3 days. Residues were below the limit of determination (<0.05 mg/kg). In Japan residues after treatment with 0.3 and 0.6 kg ai/ha and a PHI of 14 days were below the limit of determination (<0.01 mg/kg). In Poland potatoes were treated with 0.03-0.09 kg ai/ha, and residues after 74 days were <0.01 mg/kg. In Poland residues were also determined in potato haulm after treatments at 0.06 and 0.09 kg ai/ha. Residues in the haulm were 0.86 and 1.6 mg/kg respectively (Table 3).

Japanese radishes. Trials were carried out in Japan in conformity with the approved uses at 0.4 and 0.8 kg ai/ha. Residues 7 days after the last application were 0.08-0.15 mg/kg. Residues in the leaves from the same treatments were 0.03-1.2 mg/kg (Table 3).

Sugar beet. Residues were determined in the roots and leaves of sugar beets after the approved treatment of 0.3 kg ai/ha. After 14 days residues in the roots were <0.01-0.04 mg/kg, and in the leaves 1.0-1.7 mg/kg (Table 3).

Rape. Treatments of rape were carried out in Poland with the approved uses of 0.03-0.09 kg ai/ha. The recommended pre-harvest interval in Poland is 14 days, but samples were taken only at 59 and 74 days after application. Residues in rape seed and straw from the same treatments were all <0.05 mg/kg (Table 4).

Rice. Trials were carried out in Japan with treatments according to the approved use. Residues in the grain 21 days after the last application were <0.01-0.30 mg/kg. Residues in the straw from the same treatments and PHI were 0.9-5.3 mg/kg (Table 5).

Wheat. Residues in grain from wheat treated in Japan in conformity with the approved use, 0.4 kg ai/ha, and the recommended PHI of 14 days were 0.01-0.13 mg/kg (Table 5).

Maize and sweet corn. In Japan trials were carried out with the same treatment of sweet corn and maize. The approved rate is 0.5 kg ai/ha and the recommended pre-harvest interval is 7 days. Residues in sweet corn and maize were all below the limit of determination (<0.01 mg/kg, Table 4).

Tea. Field trial were carried out in Japan on tea with the approved rate of 0.4 kg ai/ha. Samples of tea leaves were taken and dried. Residues were determined in the dried leaves and in drinking tea prepared from the dried leaves by adding 540 ml of boiling water to 9 g leaves and filtering after 5 minutes. Residues in the leaves and in tea from leaves harvested 7 days after the last application, which is the recommended PHI, were 16-69 and 0.06-0.52 mg/kg respectively (Table 6).

Table 2. Residues of etofenprox in fruits from supervised trials.

Crop	Application				PHI,	Residues, mg/kg	Re-port
Country Year	No	Intv. weeks	kg ai/ha	kg ai/hl	days		
Mandarin							
Japan 1986 (Shizuoka)	3	2	1.0	0.02	14 20 28	pulp peel fruit <0.01(2) 7.2-7.6 1.5 <0.01(2) 6.6-6.3 1.4 <0.01(2) 5.2-4.8 1.1	7.1 87
(Oita)	3	2	1.6	0.02	14 21 28	<0.01(2) 11.4(2) 1.9 <0.01(2) 9.6-9.1 1.8 <0.01(2) 7.6-7.3 1.4	7.1 87
Apple							
Hungary 1987 (Zalaegersz.)	2	4	0.15	0.015	0 1 4 8 12 19	0.12-0.16-0.08 0.08-0.12-0.06 0.10-0.17-0.09 0.07-0.04-0.11 0.08-0.13-0.03 0.04-0.02-0.02	24 1.2 1
(Tiszavasv.)	5	3-4	0.15	0.01	0 1 4 6 10 14	0.46-0.36-0.18 0.46-0.40-0.09 0.28-0.20-0.10 0.30-0.06-0.05 0.20-0.20-0.10 0.23-0.15-0.11	600
Japan 1983 (Nagano)	3	1	1.2	0.02	14 21 28	0.37-0.41 0.27-0.28 0.27-0.31	27.7 83
(Toyama)	3	1	1.0	0.02	14 21 28	0.79-0.82 0.69-0.70 0.54-0.59	27.7 83
Pear							
Japan 1983 (Akita)	3	1	0.8	0.02	14 21 27 41	0.23-0.23 0.20-0.22 0.21-0.22 0.18-0.20	27.7 83

Crop / Country Year	Application: No	Intv. weeks	kg ai/ha	kg ai/hl	PHI, days	Residues, mg/kg	Report
(Nagano)	3	1	1.0	0.02	14 21 28 42	0.52-0.53 0.43-0.49 0.29-0.30 0.15-0.17	27.7 83
Peach							
Japan 1984 (Yamanashi)	3	2	0.8	0.02	14 21 28	pulp / peel <0.01-<0.01 / 2.3-3.7 <0.01-<0.01 / 4.2-4.2 <0.01-<0.01 / 1.2-1.3	20.5 85
(Niigata)	3	1	0.8	0.02	7 14 21 28	0.01- 0.01 / 5.5-5.6 <0.01-<0.01 / 6.4-6.8 0.02- 0.03 / 5.3-5.8 0.02- 0.02 / 5.3-5.5	20.5 85
Grapes							
Japan 1985 (Yamanashi)	2	2	0.4	0.02	28 42 56	4.0-3.8 2.1-2.0 1.1-1.1	27.11 85
(Kyota)	2	2	0.6	0.02	28 42 56	2.7-2.6 0.90-0.90 0.66-0.64	27.11 85
Persimmon							
Japan 1984 (Nara)	3	2	1.0	0.02	21 28 42	0.73-0.75 0.56-0.62 0.43-0.45	20.5 85
(Tokushima)	3	2	1.0	0.02	20 27 42	1.1-1.2 0.67-0.70 0.57-0.57	20.5 85

Table 3. Residues of etofenprox in vegetables from supervised trials.

Crop / Country Year	Application: No	Intv. weeks	kg ai/ha	kg ai/hl	PHI, days	Residues, mg/kg	Report
Onion							
Japan 1989 (Ibaragi)	4	2	0.3	0.02	14 21	<0.01-<0.01 <0.01-<0.01	30.3 90
(Nagamo)	4	2	0.3	0.02	14 21	<0.01-<0.01 <0.01-<0.01	30.3 90
Cabbage							
Japan 1983 (Ibaragi)	3	1	0.4	0.02	3 7 14	0.30-0.32 0.14-0.16 0.09-0.09	27.7 84
(Kanagawa)	3	1	0.5	0.02	3 7 14	0.21-0.21 0.06-0.06 0.08-0.08	27.7 84
Cucumber							
Japan 1984 (Ibaragi)	3	1	0.5	0.02	1 3 7	0.12-0.13 0.04-0.04 0.03-0.03	20.5 85
(Saitama)	3	1	0.5	0.02	1 3 7	0.13-0.13 0.04-0.04 <0.01-<0.01	20.5 85
Egg plant							
Japan 1984 (Ibaraga)	3	1	0.4	0.02	1 3 7	0.48-0.48 0.40-0.42 0.13-0.14	20.5 85

Crop Country Year	Application No	Intv. weeks	kg ai/ha	kg ai/hl	PHI, days	Residues, mg/kg	Report
(Saitama)	3	1	0.4	0.02	1 3 7	0.15-0.17 0.09-0.09 0.02-0.02	20.5 85
Tomato							
Japan 1984 (Ibaraga)	3	1	0.4	0.02	1 3 7	0.35-0.36 0.34-0.37 0.31-0.32	20.5 85
(Nagano)	3	1	0.4	0.02	1 3 7	1.8-1.9 1.8-1.8 2.0-2.0	20.5 85
Chinese cabbage							
Japan 1983 (Tochigi)	3	1	0.4	0.02	7 14 22	0.08-0.08 0.02-0.02 0.01-0.01	27.7 84
(Nagano)	3	1	0.8	0.02	7 14 21	0.14-0.15 0.02-0.02 0.07-0.07	27.7 84
Peas with pods							
Japan 1990 (Wakayama)	2	2	0.3	0.02	1 7 14 21	0.35-0.34 0.05-0.04 <0.02-0.02 <0.02-0.02	30.3 90
(Hiroshima)	2	2	0.3	0.02	1 7 14 21	0.79-0.79 0.27-0.26 0.16-0.15 <0.02-<0.02	30.3 90
Soya bean							
Japan 1984 (Fukushima)	2	1	0.3	0.02	7 14 21	unripe mature 1.6-1.7 1.1-1.1 <0.01 (2) 0.26-0.27	30.3 85
(Nagano)	2	1	0.3	0.02	7 14 21	1.5-1.5 1.0-1.0 <0.01 (2) 0.18-0.20	30.3 85
Potato							
Hungary 1981 (Gavavenc)	1		0.15	0.025	7	<0.05 (5)	602
(Nyiregyháza)	1		0.15	0.025	7	<0.05 (5)	
(Aranykalász)	2	6.5	0.15	0.05	27[1] 39[2]	<0.01 (3) [1] after 1 appl. <0.01 (7) [2] after last appl.	
Japan 1984 (Hokkaido)	3	1	0.3	0.02	3 7 14	<0.01-<0.01 <0-01-<0.01 <0.01-<0.01	20.5 85
(Nagano)	3	1	0.6	0.02	3 7 14	<0.01-<0.01 <0.01-<0.01 <0.01-<0.01	20.5 85
Poland 1986	1		0.03 0.045 0.06 0.09	0.01 0.015 0.02 0.03	74 74 74 74	tuber haulm <0.01 <0.01 <0.01 0.86 <0.01 1.6	17. 12. 86
Japanese radish							

Japan 1987						roots leaves	
(Nagano)	3	1.5	0.3	0.02	14	<0.01 (2) 0.33-0.31	1.6.
					21	<0.01 (2) 0.03 (2)	88
					30	0.01 (2) 0.03 (2)	
(Ishikawa)	3	1.5	0.3	0.02	14	0.05 (2) 2.0-1.8	1.6.
					21	0.03 (2) 1.2-1.1	88
					30	<0.01 (2) 0.3 (2)	
Sugar beet							
Japan 1984						roots leaves	
(Sapporo)	3	2	0.3	0.02	14	<0.01 (2) 1.6-1.7	20.5
					21	<0.01 (2) 0.91-0.97	85
					28	<0.01 (2) 0.38-0.43	
(Naganuma)	3	2	0.3	0.02	14	0.04 (2) 1.0-1.1	20.5
					21	0.03 (2) 0.36-0.36	85
					28	0.04 (2) 0.31-0.31	

Table 4. Residues of etofenprox in rape seed from supervised trials in Poland in 1986.

	Application				PHI,	Residues, mg/kg		Report
Poland	No	Intv. weeks	kg ai/ha	kg ai/hl	days			
(Trzebnica)						seed	straw	
	1		0.03	0.01	74	<0.05	<0.05	21.3
			0.045	0.015	74	<0.05	<0.05	87
			o.o6	0.02	74	<0.05	<0.05	
			0.09	0.03	74	<0.05	<0.05	
(Winnogora)								
	1		0.03	0.01	59	<0.05	<0.05	21.3
			0.045	0.015	59	<0.05	<0.05	87
			0.06	0.02	59	<0.05	<0.05	
			0.09	0.03	59	<0.05	<0.05	

Table 5. Residues of etofenprox in cereal grains from supervised trials.

Crop	Application				PHI,	Residues, mg/kg		Report
Country Year	No	Intv. weeks	kg ai/ha	kg ai/hl	days			
Rice								
Japan 1986						grains	straw	
(Chiba)	5	2	0.4	0.02	14	0.30-0.29	3.1-3.0	7.1.
					21	0.30-0.30	5.3-5.1	87
					28	0.06-0.05	2.5-2.4	
(Ishikawa)	5	2	0.4	0.02	14	0.02-0.02	2.0-1.9	
					21	<0.01-<0.01	0.9-0.9	7.1.
					28	<0.01-<0.01	1.4-1.3	87
Wheat								
Japan 1987								
(Chiba)	2	2	0.4	0.02	14	0.01-0.01		23.5
					21	<0.01-<0.01		88
					28	<0.01-<0.01		
(Ishikawa)	2	2	0.4	0.02	14	0.13-0.12		23.5
					21	0.06-0.06		88
					28	0.01-0.01		
Maize and sweet corn								
Japan 1984						Maize	Sweet corn	
Ibaragi	4	2	0.5	0.02	7	<0.01 (2)	<0.01 (2)	20.5
					14	<0.01 (2)	<0.01 (2)	85
Nagano	4	2	0.5	0.02	7	<0.01 (2)	0.06 (2)	20.5
					14	<0.01 (2)	<0.01-0.01	85

Table 6. Residues of etofenprox in tea from supervised trials in Japan in 1983.

Region	Application				PHI,	Residues, mg/kg	Report
	No	Intv. weeks	kg ai/ha	kg ai/hl	days		
Saga	2	1	0.4	0.02	7 14 21	tea leaves drinking tea 16-17 0.06-0.07 10-10 0.03-0.04 1.5-1.5 <0.01 (2)	7.1.87
Kagoshima	2	1	0.4	0.02	7 14 21	66-69 0.51-0.52 20-20 0.13-0.14 3.4-3.8 0.02 (2)	7.1.87

Animal feeding studies

Dairy cows were fed diets containing 10, 30 and 1000 mg etofenprox/animal/day for 28 days, the compound being incorporated with the concentrate feed. The dosage of 1000 mg/day represents a considerably higher level of intake than would occur in practice. Milk samples were taken from the daily production from 3 days before dosing until the end of the dosing period of 28 days. Samples were also taken from the group given 1000 mg/day during a withdrawal period of 14 days, so that the last milk sample from this group was taken at day 42. The cows fed with 10 and 30 mg/day and 3 cows fed with 1000 mg/day were slaughtered after 29-30 days, while 2 cows fed with 1000 mg/day were killed 14 days later at day 43. Samples of liver, kidney, skeletal muscle, peritoneal fat and subcutaneous fat were analyzed.

Residues in milk. Residues in the milk from cows fed with 10 and 30 mg/day were all below the limit of determination (<0.05 mg/kg) except 2 samples from one cow fed with 30 mg/kg, where the residues were at the limit of determination. Residues in the milk from cows fed 1000 mg/day were from 0.66 to 2.1 mg/kg during the dosing period, and from 1.66 to 0.09 mg/kg during the no-treatment withdrawal period, declining rapidly during the early part of this period but with the rate slowing over the last part (Table 7).

Residues in tissues. Residues of etofenprox in liver, kidney, and skeletal muscle from cows fed with 10 and 30 mg/day were all below or at the limit of determination (0.05 mg/kg), but were present in peritoneal and subcutaneous fat up to 1.9 mg/kg. Residues in cows fed with 1000 mg/kg and slaughtered at days 29-31 were present in liver (0.25-0.63 mg/kg), kidney (0.08-0.62 mg/kg), muscle (0.08-0.35 mg/kg), peritoneal fat (1.7-14 mg/kg) and subcutaneous fat (1.0-3.5 mg/kg). After a 14-day withdrawal period residues were still detectable in all tissues, at the same levels in peritoneal and subcutaneous fat but with some reduction in the liver, kidney and muscle (Roberts *et al.*, 1987). (Table 8).

Table 7. Residues of etofenprox in milk of cows.

Days	mg/l milk		
	Feeding level, mg etofenprox/cow/day		
	10	30	1000
2-28	<0.05-<0.05	<0.05-0.05	0.66-2.1 mean: 1.3
30-32			0.31-1.7
34-36			0.14-0.25
38-40			0.10-0.22
42			0.09-0.10

Table 8. Residues of etofenprox in tissues of cows.

Feeding level, mg/cow/day	mg/kg tissue at day 29-30				
	Liver	Kidney	Skeletal muscle	Peritoneal fat	Subcutaneous fat
10	<0.05-<0.05	<0.05-<0.05	<0.05-<0.05	0.21-0.54	0.08-0.28
30	<0.05-<0.05	<0.05-0.05	<0.05-0.05	0.84-2.0	0.07-0.50
1000	0.25-0.63	0.08-0.16	0.08-0.35	1.8-14	1.0-3.5
1000 (day 42)	<0.05-0.05	0.23-0.23	0.05-0.05	4.2-12	0.33-3.0

FATE OF RESIDUES

General

The fate of etofenprox was studied in plants (beans and rice), soil and water. The metabolic pathways of etofenprox in animals were also proposed, but with references only in the toxicological information available to the Meeting.

Etofenprox is mainly metabolized by desethylation of the ethoxyphenyl group, hydroxylation of the phenoxy ring and oxidation of the benzyl moiety with subsequent cleavage of the ether linkage to form polar compounds, and in animals to form conjugates.

In animals

According to the summarized information avilable to the Meeting etofenprox was rapidly excreted when given to male and female rats, with more than 80% of the administered dose excreted within 48 hours in faeces and urine and with the major route via faeces. The compound was metabolized by desethylation of the ethoxyphenyl group, hydroxylation of the phenoxy ring and oxidation of the benzyl methylene group. The same metabolic

pattern and a similar profile of metabolites were observed in dogs. Proposed metabolic pathways of etofenprox in animals are shown in Figure 1.

In plants

Beans. A study was carried out by applying [^{14}C]etofenprox to bean leaves under laboratory conditions. Two radiolabelled compounds were used: [á-^{14}C]etofenprox, where the labelled carbon was in the benzyl methylene group and [1-^{14}C]propyl-etofenprox where one propyl carbon was labelled. Two primary leaves of the plant were treated with the labelled preparations and treated plants were harvested 1, 2 and 3 weeks after application and divided into treated leaves, other leaves, shoots and roots. The parent compound and its metabolites were identified using three different TLC systems, and the identity of each fraction was confirmed by co-chromatography with unlabelled compounds. The determination of the quantity of the parent compound and the metabolites was carried out by radioanalysis using X-ray film.

The radioactivity in and on the treated leaves decreased with time, but that in other parts of the plants (leaves, shoots and roots) was less than 1% of the total radioactivity. After 3 weeks the parent compound amounted to 0.26% in the other leaves and shoots and to 0.015% in the roots. No significant difference between the two labelled forms was found in the proportion of metabolites after 1 and 3 weeks. Etofenprox was gradually decomposed to less than 75% of the original quantity after 1 week and to less than 50% after 3 weeks. The main metabolite was 2-(4-ethoxyphenyl)-2-methylpropyl 3-phenoxybenzoate (compound IV, Figure 2), which was present at 8% after 1 week and 13% after 3 weeks. Residues of the more polar metabolites (mainly 3-phenoxybenzoic acid and 2-(4-ethoxyphenyl)-2-methylpropan-1-ol) were increased after 3 weeks.

The half-life of etofenprox on bean leaves was determined to be 3 weeks with both labelled forms. At that time the main metabolite (IV) accounted for 11-15% and unrecovered compounds for 14-18% of the radioactivity. Figure 2
shows the proposed pathways, which are mainly oxidation at both carbons of the ether linkage, desethylation and acetylation, dephenylation of the phenoxy group and *para*-hydroxylation of the phenoxy group. Some of the more polar metabolites were conjugated with glucose.

Experiments have shown that all metabolites except the conjugates are very similar to photodegradation products, implying that the formation of these metabolites is probably affected by light (Tomoda *et al.*, 1985b).

Rice. A similar study to that on bean plants was carried out on rice. The experimental conditions were the same except that seeds from the rice were also investigated. The half-life of etofenprox on rice plants was determined to be 1 week, which is less than on beans, and after 20 days 90% of the original etofenprox was degraded. As with beans very little radioactivity was transferred to other parts of the plant. Residues of the parent compound in seeds after 2, 4 and 6 weeks were beween 0.01

and 0.04% of the radioactivity in the plant. The degradation pathways were very similar to those in bean plants (Tomoda *et al.*, 1985a).

In soil

Laboratory studies. Three experiments were carried out on the degradation of etofenprox in soil under laboratory conditions. Three different types of soil were used: Yamanashi sandy soil containing 78% sand, 11% silt and 11% clay; Chiba light clay containing 28% sand, 39% silt and 32% clay; and Shizuoka light clay containing 43% sand, 26% silt and 31% clay. The same two labelled compounds that were used in the degradation studies on plants were applied to the soils.

In one experiment samples of the three soil types were pre-incubated for 2 weeks, ^{14}C-labelled etofenprox was added and the incubation continued at 25°C in the dark, and the moisture content was maintained. Soil samples were extracted and analyzed after the incubation. The total radioactivity decreased gradually, and 2 weeks after application it amounted to 60-70% of the original. The half-life of etofenprox in soil was determnined to be 6-9 days with only small differences between soil types or the position of labelling. The amount of etofenprox decreased to 15% after 3 weeks. Etofenprox was degraded mainly by oxidation to 2-(4-ethoxyphenyl)-2-methylpropyl 3-phenoxybenzoate and to 2-(4-ethoxyphenyl)-2-methylpropyl 3-hydroxybenzyl ether (Figure 3).

The proportions of these products were 2.6-7.1% and 1.4-4.0% of the radioactivity, respectively, after 1 week and 1.4-4.2% and 1.3-3.0% after 2 weeks. Other, mainly polar products were present in minor quantities. The ^{14}C bound in the soil increased with time and was 25-44% 2 weeks after application.

Figure 1. Proposed metabolic pathways of etofenprox in animals.

*Postulated intermidiate

Figure 2. Proposed metabolic pathways of etofenprox in plants.

*Postulated intermidiate

Figure 3. Proposed degradation pathways of etofenprox in soil.

In the second experiment the liberation of $^{14}CO_2$ was determined. Chiba light clay was incubated with etofenprox at 25◦C in the dark. The evolved $^{14}CO_2$ was trapped, and residues of the parent compound and degradation products were determined. After 2 weeks 8-12% of the radioactivity was present as $^{14}CO_2$, and after 8 weeks the figure was 32-44%.

In the third experiment the influence of micro-organisms was investigated. The two ^{14}C-labelled forms of etofenprox were applied to sterilized and non-sterilized Yamanashi sandy loam samples, which were incubated at 25◦C for 2 weeks under light and dark conditions. After 2 weeks 60-80% of the applied etofenprox had been decomposed in the unsterilized soil, both in darkness and in light, but no degradation occurred in the sterilized samples (Tomoda *et al*., 1985c).

Field studies. The rate of degradation of etofenprox was examined in paddy and upland soils. In the paddy soil experiment two soil types were used: loam with 8.2% clay and 7.5% organic carbon and clayish loam with 21% clay and 2.4% organic carbon. Etofenprox was applied at the rate of 0.4 kg ai/ha and 7 days later at 0.9 kg ai/ha. Samples were taken from the upper 10 cm of the soil 0, 1, 3, 7, 14, 28, 56 and 98 or 105 days after the second application. The half-life was determined to be approximately 79 days in loam soil and 62 days in clayish loam (Ishii *et al*., 1985a).

The investigation on upland soil was carried out with volcanic ash loam containing 10% clay and 6.2% organic carbon, and alluvial clayish loam containing 2% clay and 2.8% organic carbon. Etofenprox was applied 3 times at the rate of 0.16-0.20 kg or 0.5 kg ai/ha. Samples were taken 0, 1, 3, 7, 14, 28 and 56 days after the last application. The half-life was determined to be 39 days in volcanic ash loam and only 9 days in alluvial clay loam (Ishii *et al*, 1985b).

Adsorption and leaching. The adsorption and leaching of etofenprox was studied in three soil types: Yamanashi sandy loam, Chiba light clay and Shizuoka light clay (the compositions were given in "Laboratory studies"). Both [1-^{14}C]propyl- and [á-^{14}C]benzyl-labelled etofenprox were used. In two experiments glass columns were packed with soil, incubated for 2 weeks at 25◦C in darkness, and labelled etofenprox mixed with soil was applied as a 5 cm layer at the top of the columns. In one experiment the two labelled compounds were mixed with the soil just before applications to the columns, in the other the soil was treated and pre-incubated 2 weeks before application. The columns were eluted gently with water equivalent to 3-5 times the maximum water-holding capacity of each soil.

In the experiment where the soil was treated just before application to the columns approximately 74% of the radoactivity remained in the top 5 cm layer, and the parent compound accounted for most of it. Translocation was very low, and unchanged parent compound was not detected in the deeper layers. In the experiment with pre-incubated soil at least 90% of the radioactivity remained in the top 5 cm layer. The radioactivity

recovered from eluted solutions was less than 4 and 5% of the applied radioactivity for direct-packed and pre-incubated soils respectively, and unchanged parent compound was not detected in the eluates (Tomoda *et al.*, 1985d).

In water

The stability of etofenprox in water was investigated under laboratory conditions at various pH values in darkness at 25◦C. Etofenprox was stable under neutral (pH 7.0) and acidic (pH 5.0) conditions. The estimated half-life was more than one year in the range of pH values tested (Asari, 1992).

In another experiment 200 mg etofenprox/l city water was incubated in a beaker covered with polythene in a greenhouse at 23 ± 5◦C. After 1 and 3 weeks 70% and 93% of the etofenprox had decomposed. This relatively rapid degradation compared with the degradation under laboratory conditions was mainly due to sunlight (Udagawa *et al.*, 1986).

Photodegradation

The half-life and degradation pathways of [1-^{14}C]propyl- and [á-^{14}C]benzyl-labelled etofenprox on the surface of glass discs were studied. The compound was exposed to artificial light of 30,000 lux for 13 hours per day at 25-30◦C. The half-life was approximately 4 days and the major degradation product was 2-(4-ethoxyphenyl)-2-methylpropyl 3-phenoxybenzoate, which accounted for 25% of the radioactivity after 7 days of exposure. The degradation pathways did not show any differences from those observed in the metabolism study on bean leaves. The degradation which occurred on the surface of bean leaves was therefore considered to be strongly linked to photodegradation.

The degradation of etofenprox applied as a film to the bottom of a silica flask was also studied. The compound was exposed to high-intensity light from a xenon lamp (550 ìw/cm^2) for about 10 hours. Several degradation products were identified, including the main product found in the experiment with glass discs (Tomoda *et al.* 1985e).

METHODS OF RESIDUE ANALYSIS

Methods have been developed to determine etofenprox in plant material, soil milk and animal tissues.

The same method with some modifications is used for plant material and soil. Residues are extracted with acetone and the extract is cleaned up by partition between n-hexane and water. Crop extracts are further cleaned up by Florisil column chromatography and soil extracts by column chromatogrphy on alumina and silica gel. For some crops it is possible to analyse the chromatographed extract directly for residues. In most cases the purified extract is reacted with trimethylsilyl iodide to form 3-phenoxybenzyl iodide and the reaction mixture partitioned with n-hexane. The derivative is determined by GLC with an EC

detector. The limit of determination is in the range of 0.01-0.02 mg/kg. The recovery is more than 73%.

Residues in milk and animal tissues are extracted into ethyl acetate/hexane. The solvent is removed by rotary evaporation and the residue cleaned up by Florisil chromatography. The concentrated eluate is derivatized with trimethylsilyl iodide to form 3-phenoxybenzyl iodide, which is cleaned up on a silica "Sep-pak" and determined by GLC with an EC detector. The limit of determination is 0.05 mg/kg.

NATIONAL MAXIMUM RESIDUE LIMITS

National MRLs reported to the Meeting are shown below.

Country	Crop	MRL, mg/kg
Italy	Apple	0.23
	Cabbage	0.96
	Egg plant	<0.05
	Peach	0,16
	Tomato	0.23
Hungary	Apple	1
	Cereals	0.1
	Corn	0.05
	Grape	5

Japan	Apple	2
	Azuki beans	0.1
	Cabbage	2
	Cereals	0.5
	Chestnut	2
	Chinese cabbage	2
	Citrus (pulp)	2
	Citrus (peel)	10
	Cucumber	2
	Egg plant	0.2
	Lettuce	2
	Melon	2
	Pea, podded	2
	Peach	2
	Pear	2
	Persimmom	2
	Potato	0.1
	Radish, Japanese	2
	Rice grains (unpolished)	0.5
	Soya beans	0.1
	Soya beans (prematured)	2
	Sugar beets	0.5
	Sweet potato	0.1
	Tea leaves (open field)	10
	Tea leaves (under coverage)	10
	Tomato	0.5
	Water melon	2
	Welsh onion	2
Spain	Cabbage	0.2
	Citrus	1
	Egg plant	0.2
	Fruit trees	1
	Rice	1
	Tomato	0.5

APPRAISAL

Degradation studies were carried out on etofenprox in plants (beans and rice) and soil. Metabolic studies were also carried out in animals (rats and dogs), but information about these was only available to the Meeting in a summarized form.

The metabolism of etofenprox in bean and rice plants was examined by applying á-^{14}C-benzyl-labelled and 1-^{14}C-propyl-labelled etofenprox to leaves of the plants under laboratory conditions. There was very limited translocation of the parent compound and its metabolites to other parts of the plants, including the seeds in rice. Etofenprox was gradually decomposed on and in the treated leaves and was reduced to approximately 50% after 3 weeks. The main metabolite from the oxidization of etofenprox was 2-(4-ethoxyphenyl)-2-methylpropyl 3-phenoxybenzoate. Residues of other metabolites, mainly 3-phenoxybenzoic acid and 2-(4-ethoxyphenyl)-2-methylpropan-1-ol, were also present but in small quantities. The half-life of etofenprox on beans was determined to be 3 weeks for both labelled forms. At that time the main metabolite accounted for 11-15% and unrecovered compounds for 14-18% of the radioactivity applied. Experiments have shown that all metabolites observed on the bean leaves, except conjugates, were very similar to products formed by photodegradation, implying that the metabolism on plant leaves is affected by light.

Degradation studies on etofenprox in soil were carried out with three different soil types using the same two ^{14}C-labelled forms as in the experiments on plants. The half-life of etofenprox in soil was determined to be 6-9 days and largely independent of the soil types and labelled forms used. The main products formed after oxidation were 2-(4-ethoxyphenyl)-2-methylpropyl 3-phenoxybenzoate and 2-(4-ethoxyphenyl)-2-methylpropyl 3-hydroxybenzyl ether. The liberation of $^{14}CO_2$ from the degradation of ^{14}C-labelled etofenprox was examined. After 2 and 8 weeks the radioactivity originating from CO_2 was 8-12 and 32-44%, respectively. The degradation of etofenprox in soil was evidently caused by micro-organisms as no degradation occurred in sterilized soil.

Field studies were carried out to examine the rate of degradation of etofenprox in paddy and upland soils. The half-lives in the two paddy soil types examined were 79 and 62 days, while the half-lives in the two upland soils were 39 and 9 days. Etofenprox is strongly adsorbed to soil, and little leaching takes place. No residues of the parent compound and only small quantities of metabolites were detectable in the effluents from three soil types after 2 weeks of leaching.

Supervised trials were carried out on several kinds of fruits and vegetables in Japan, on apples and potatoes in Hungary, and on potatoes and rape in Poland. In Japan trials were also carried out on rice, wheat, corn and tea. The application rates were different in the three countries. In Japan the rates were generally of the order of 0.5-1.5 kg/ha,

while the highest rate in Hungary was 0.15 kg ai/ha and in Poland 0.09 kg ai/ha. Residues in apples were 0.4-0.8 mg/kg in Japan and 0.1-0.2 mg/kg in Hungary. Residues in potatoes were below the limit of determination in all the trials, including those in Japan at the highest dose rate.

Residues were determined in animal products after feeding experiments on dairy cows. Cows were fed with etofenprox at levels of 10, 30 and 1000 mg/animal/day, where 1000 mg/day represents a considerably higher level of intake than would occur in practice. After a feeding period of 28 days residues in milk from cows fed with 10 and 30 mg/day were at or below the limit of determination (0.05 mg/kg), but residues from 1000 mg/day were up to 2 mg/kg. Residues in tissues were also examined after the feeding period. Residues in liver, kidney and skeletal muscle from 10 and 30 mg/day were at or below the limit of determination, but in the peritoneal and subcutaneous fat were quite high and up to 0.84 mg/kg. For cows fed with 1000 mg/day, residues were up to 14 mg/kg in peritoneal fat and up to 3.5 mg/kg in subcutaneous fat, and were also present in kidney, liver and muscle.

Residues of etofenprox in plant material and soil are determined by gas chromatography with an electron capture detector after extraction with acetone and clean-up by partitioning with water/n-hexane and by column chromatography on Florisil or alumina/silica gel. For most crops and soil the purified extract is reacted with trimethylsilyl iodide to form 3-phenoxybenzyl iodide. The limit of determination is 0.01 mg/kg. For milk and animal tissues the method is similar, but ethyl acetate/hexane is used for the extraction and a silica sep-pak is used for the chromatographic clean-up. The limit of determination for residues in animal products is 0.05 mg/kg.

The manufacturer informed the Meeting that the analytical methods described, including the chromatographic clean-up step, are specific for the parent compound etofenprox and do not determine other compounds containing the 3-phenoxybenzyl moiety.

Supervised trials for most crops were carried out in only one country, Japan, and although they were at two sites they took place within the same year. The Meeting was therefore able to propose maximum residue limits for etofenprox in only two crops.

RECOMMENDATIONS

On the basis of the data on residues from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits.

Definition of the residue: etofenprox (fat-soluble).

Commodity	Recommended MRL (mg/kg)	PHI on which based, days

CCN	Name		
FP 0009	Pome fruits	1	14
VR 0859	Potato	0.01*	14

FURTHER WORK OR INFORMATION

Desirable

1. Submission of documentation for the specificity of the analytical methods for the determination of etofenprox.

2. Supervised trials on crops from more than one year and trials carried out in more than one country.

3. Studies on the processing of crops containing residues of etofenprox

4. Residues in straw from wheat and other crops used as animal feedingstuffs.

REFERENCES

(All unpublished reports)

1. Asari, K. 1992. Stability of etofenprox in water. Report from Mitsui-Toatsu Chemicals, Inc.

2. Ishii, M. *et al.*, 1985a. Residue Determination in Paddy Soil treated with Ethofenprox 20% E C and 1.5% G. Japan Analytical Chemistry Consult. Co. Ltd.

3. Ishii, M. *et al.*, 1985b. Residue Determination in Upland Soil treated with Ethofenprox 20% Wettable Powder. Japan Analytical Chemistry Consult. Co. Ltd.

4. Roberts, N.L., Cameron, D.M., Emerson, E.S. and Carter, J.N. 1987. Residues of Ethofenprox in the Milk and Tissues of Dairy Cows. Huntingdon Research Centre Ltd. Rep. MTC 132/87587.

5. Tomoda, K. *et al.*, 1985a. Metabolism Study of Ethofenprox-2. Meabolism in Rice Plants. Mitsui-Toatsu Chemicals, Inc.

6. Tomoda, K. *et al.*, 1985b. Metabolism Study of Ethofenprox-1. Meabolism in Bean Plants. Mitsui-Toatsu Chemicals, Inc.

7. Tomoda, K. *et al.*, 1985c. Metabolism Study of Ethofenprox-3. Meabolism in Soil. Mitsui-Toatsu Chemicals, Inc.

8. Tomoda, K. *et al.*, 1985d. Metabolism Study of Ethofenprox-4. Soil Leaching Test. Mitsui-Toatsu Chemicals, Inc.

9. Tomoda, K. *et al.*, 1985e. Metabolism Study of Ethofenprox- 5. Photodegradation on Glass Surface. Mitsui-Toatsu Chemicals, Inc.

10. Udagawa, T. *et al.*, 1986. Degradation of Ethofenprox in Water. Mitsui-Toatsu Chemicals, Inc.

ENDOSULFAN (032)

EXPLANATION

Endosulfan has been reviewed many times from 1967 to 1989, at which time a full re-evaluation was carried out. As a result of information that proposed MRLs for endosulfan on several brassica crops did not reflect GAP in the USA, the 1992 CCPR placed the recommendations in step 7B, pending review by the JMPR. Additional GAP and residue data have now become available and are reviewed below.

USE PATTERN

Endosulfan is widely used as an insecticide on fruits, vegetables, cereals and oilseed. GAP information from 21 countries was provided by the main manufacturer, Hoechst AG; this is summarised in Table 1.

Table 1. Endosulfan - registered use rates and patterns

Country	Crop	EC/WP /DP	Conc	Applic. (kg ai/ha)	No.of app.	PHI (days)	Comments
Argentina	Vegetables	EC	35%	0.60	1	7	Post-Flowering
	Soya bean	EC	35%	0.45	1	14	Post-Flowering
	Sunflower	EC	35%	0.50	1	14	Flowering
Australia	Pineapple	EC	35%	[0.56-0.7 g/l]	1	-	Post-harvest dip
Belgium	Fruits	EC	35%	0.35-1.75	-	28	4 wks pre-harvest
	Berries	EC	35%		-	42	
	Strawberry & small fruits	EC	35%	0.525	-	28	Before flowering
	Mushrooms	EC	35%	14	-	21	3 wks pre-harvest
	Potato	EC	35%	0.175-0.525	-	28	4 wks pre-harvest
	Rape	EC	35%	0.6-0.7	-	28	
Brazil	Soya bean	EC	35%	0.26	1	30	
	Cotton	EC	35%	0.525	1	30	
	Coffee	EC	35%	0.525	1	70	
Canada	Apple & Pear	WP	50%	1.6-3.4	Sev.	15	
	Apricot, Cherry, Peach & Plum	EC/WP	40/50%	0.48-0.76	2-3	15	Avoid spray on fruit
	Grapes	WP	50%	1.25-1.5	2	30	
	Strawberry	EC	40%	1.0-5.4	1-2	7	Pre-bud & after harvest
		WP	50%	1.0-16	1-2	7	Pre-bud & after harvest
	Broccoli, Brussels sprouts, Cabbage & Cauliflower	EC/WP	40/50%	0.55-0.88	Sev.	7	7 day intervals
		DP	2%	0.8-1.0	Sev.	7	7-10 day intervals
	Cucumber, Melon, Pumpkin & Squash	EC/WP	40/50%	0.5-0.6	1-4	2	7 day intervals
		DP	2%	0.8-1.0	4	5	7-10 day intervals
	Egg plant, Peppers & Tomato	EC	40%	0.5-0.6	Sev.	2	7 day intervals
		DP	2%	1.0-1.1	Sev.	5	7-10 day intervals
	Sweet Corn	EC/WP	40/50%	1.0-1.7	1-2	50	
	Lettuce	EC/WP	40/50%	0.8-0.88	1-3	14	

Country	Crop	EC/WP /DP	Conc	Applic. (kg ai/ha)	No.of app.	PHI (days)	Comments
		DP	2%	0.8-1.0	1-3	14	7-10 day intervals
	Spinach	EC/WP	40/50%	0.8-0.88	1	14	
	Beans	EC	40%	0.56-1.0	Sev.	2	Not Lima beans
		WP	50%	0.55-0.75	Sev.	2	Not Lima beans
	Peas (canning)	EC/WP	40/50%	0.55-0.88	1-2	7	
	(seed)	EC/WP	40/50%	0.55-0.88	Sev.	7	
	Potato	EC/WP	40/50%	0.56-0.84	Sev.	1	As necessary
		DP	2%	0.81-1.1	Sev.	1	7-10 day intervals
	Rutabaga	EC/WP	40/50%	0.80-0.87	1-2	45	
	Turnip	EC/WP	40/50%	0.80-0.87	1-2	45	
	Sugar beet	EC	40%	0.6-1.1	1	45	
	Celery	EC/WP	40/50%	0.80-0.88	1-3	14	
		DP	2%	1.0-1.2	Sev.	14	7-10 day intervals
	Maize	EC/WP	40/50%	1.0-1.7	1-2	50	
	Sunflower	EC	40%	0.56-0.6	1	60	
	Alfalfa	EC	40%	0.3	1	30	
	Clover	EC	40%	0.3	1	30	
Colombia	Strawberry	EC	35%	0.56-0.7	-	-	
	Beans	EC	35%	0.525-0.7	1-3	-	
	Cruciferae	EC	35%	0.7-1.05	-		
	Soya bean	EC	35%	0.35-0.7	1-2	-	
	Potato	EC	35%	-	-	-	
	Rice	EC	35%	1.2-1.4	-	-	
	Cotton	EC	35%	0.525	2-5	-	
	Sesame	EC	35%	1.05-1.4	-	-	
	Coffee	EC	35%	0.525-0.7	-	-	
Ecuador	Vegetables	EC	35%	0.35	1-3	10	
	Rice	EC	35%	0.525	1-2	-	
	Cotton	EC	35%	0.17-0.35	1	-	
	Palm nut	EC	35%	0.43	1	-	
	Coffee	EC	35%	0.63	1-2	-	
Finland	Currant, Black	EC	35%	0.375-1.5	1-2	-	Pre-flowering
	Strawberry	EC	35%	0.75-1.5	1	-	Post-harvest only
Greece	Apple	EC/WP	35/47%	All EC appl.	1	30	
	Pear	EC/WP	35/47%	are at	1	30	
	Cherry	EC/WP	35/47%	0.074-0.098	1	30	
	Grapes	EC/WP	35/47%	kg ai/hl.	1-2	30	
	Strawberry	EC/WP	35/47%	(High vol.)	1-2	-	Pre-flower/ after harvest
	Olives	EC/WP	35/47%		1	-	Pre-flowering
	Cucumber	EC/WP	35/47%		1-3	7	
	Melon	EC/WP	35/47%		1-3	7	
	Squash, Summer	EC/WP	35/47%	All WP appl.	1-3	7	
	Watermelon	EC/WP	35/47%	are at	1-3	7	
	Egg plant	EC/WP	35/47%	0.075-0.094	1-3	-	
	Peppers	EC/WP	35/47%	kg ai/hl	1-3	-	
	Tomato	EC/WP	35/47%	(High vol.)	1-3	4	
	Potato	EC/WP	35/47%		1-3	21	
	Cotton	EC/WP	35/47%		1-2	-	
	Alfalfa (seed)	EC/WP	35/47%		1	-	
	Clover (seed)	EC/WP	35/47%		1	-	
Guatemala	Cruciferae	EC	35%	0.595	1	40	
	Melon	EC	35%	0.525	1-2	30	
	Tomato	EC	35%	0.595	3	20	
	Coffee	EC	35%	0.595	1	135-165	
Indonesia	Shallot	EC	35%	0.25-0.45	5	-	

Country	Crop	EC/WP /DP	Conc	Applic. (kg ai/ha)	No.of app.	PHI (days)	Comments
	Peppers, Chili	EC	35%	0.25-0.45	8	-	
	Rice	EC	35%	0.28-0.42	2	-	
	Soya bean	EC	35%	0.21-0.31	4	-	
Ireland	Apple & Pear	WP	50%	0.625-0.85	-	7-21	
	Currants, Red, Black & White	WP	50%	0.625-0.85	-	7-21	
	Brassicas	WP	50%	0.625-0.85	-	7-21	
	Beans (dwarf)	WP	50%	0.625-0.85	-	7-21	
	Cucumber	WP	50%	0.625-0.85	-	7-21	
	Tomato & Peppers	WP	50%	0.625-0.85	-	7-21	
	Carrot, Potato & Sugar beet	WP	50%	0.625-0.85	-	7-21	
	Celery	WP	50%	0.625-0.85	-	7-21	
	Rape	WP	50%	0.625-0.85	-	7-21	
Italy	Citrus fruits	EC	32.9%	1.0	1	25	
	Peach	EC	32.9%	0.75	1	25	
	Grapes	EC	32.9%	0.5	1-2	25	
	Vegetables	EC	32.9%	0.5	1-2	25	
	Tomato	EC	32.9%	0.5	1-2	25	
	Potato	EC/WP	32.9%	0.5	1-2	25	
	Sugar beet	EC/WP	32.9%	0.5	1-2	25	
	Tree nuts	EC	32.9%	0.5	1-2	25	
Republic of Korea	Mulberry	DP	3%	1.2	1	-	After summer logging
	Chinese cabbage	DP	3%	0.9-1.8	4	30	
Philippines	Citrus fruits	EC	35%	1-3	-	7	
	Perennial fruits	EC	35%	1-3	-	7	
	Mango	EC	35%	1-3	-	7	
	Pineapple	EC	35%	1-3	-	7	
	Cruciferae	EC	35%	1-3	-	7-14	
	Egg plant	EC	35%	1-3	-	7-14	
	Tomato	EC	35%	1-3	-	1	
	Legumes	EC	35%	1-3	-	3	
	Maize	EC	35%	1.5-3	-	10	
	Rice	EC	35%	1.5-2	-	10	
	Cotton	EC	35%	1.5-4	-	-	
Portugal	Apple & Pear	WP	35%	1.2-2.45	-	28	
	Brassicas	EC	38%	1.2-2.45	1-3	28	
		WP	35%	1.2-2.45	1-3	28	
		WP	80%	1.2-2.45	1-3	28	
	Cabbage	WP	35%	1.2-2.45	1-3	28	
	Tomato	WP	35%	1.2-2.45	-	28	
	Tomato	DP	3%	0.75	-	3-28	
	Maize	WP	35%	1.2-2.45	-	28	
	Sugar cane	WP	35%	1.2-2.45	-	28	
Spain	Citrus fruits	EC	35%	2.1-6.3	1-3	15	
	Stone fruits	EC	30%	0.675-1.35	-	15	
	Grapes	EC	35%	0.32-0.63	1-3	15	
	Olives	EC	35%	0.53-1.05	-	15	
	Brassicas	EC	35%	0.53-1.58	-	15	
	Cucurbits	EC	35%	0.53-1.58	-	7	
	Egg Plant	EC	35%	0.53-1.58	-	7	
	Peppers	EC	35%	0.53-1.58	-	7	
	Tomato	EC	35%	0.53-1.58	-	7	
	Potato	EC	35%	0.53-1.58	-	15	
	Asparagus	EC	35%	0.53-1.58	-	15	
	Hazelnuts	EC	35%	0.8-1.6	1-3	15	
	Cotton	EC	35%	0.53.1.05	1-3	15	

Country	Crop	EC/WP /DP	Conc	Applic. (kg ai/ha)	No.of app.	PHI (days)	Comments
Sweden	Currants, Black	EC	35%	0.35-0.7	1	-	
	Strawberry	EC	35%	0.35-1.05	1-2	-	
Thailand	Citrus fruits	EC	35%	-	10	7-14	
	Grapes	EC	35%	-	3	7-14	
	Rambutan	EC	35%	-	5	7-14	
	Vegetables	EC	35%	0.26-0.35	5	7-14	
	Onion	EC	35%	0.52-0.7	8	7-14	
	Tomato	EC	35%	0.26-0.35	5	7-14	
	Beans	EC	35%	0.35-0.44	3	7-14	
	Rice	EC	35%	0.26-0.35	4	7-14	
	Cotton	EC	35%	0.35-0.7	5	7-14	
	Coffee	EC	35%	0.88-1.3	5	7-14	
UK	Blackberries	EC	20%	0.5	3	-	
	Currants, Black	EC	20%	0.6	3	-	First flower
	Currants, Black	EC	20%	0.9	3	-	Fruit set
	Strawberry	EC	20%	0.5	1	-	
USA	Citrus fruits	EC/WP	35/50%	0.55-2.75	1-2	365	Max. 3.3 kg/ha/year (Non-bearing trees)
	Apple	EC/WP	35/50%	0.55-2.75	1-3	21	Max. 3.3 kg/ha/year
	Pear	EC/WP	35/50%	0.28-2.75	1-2	7	Max. 3.3 kg/ha/year
	Apricot	EC/WP	35/50%	0.55-2.75	1-2	21	Max. 3.3 kg/ha/year
	Cherry	EC/WP	35/50%	0.55-2.75	1-2	21	
	Nectarine	EC/WP	35/50%	0.55-2.75	1-2	21	Max. 3.3 kg/ha/year
	Peach	EC/WP	35/50%	0.55-2.75	1-2	21	Max. 3.3 kg/ha/year
	Plum & Prune	EC/WP	35/50%	0.55-2.75	1-2	7	Max. 3.3 kg/ha/year
	Blueberries	EC/WP	35/50%	1.6	1-2	-	Max. 3.3 kg/ha/year After harvest &+6-8weeks
	Grapes	EC/WP	35/50%	0.55-1.6	1-3	7	Max. 3.3 kg/ha/year
	Strawberry	EC/WP	35/50%	1.1-2.2	1-3	4	Max. 3.3 kg/ha/year
	Pineapple	EC	35%	1.6-2.1	1-2	7	Max. 3.3 kg/ha/year
	Broccoli	EC/WP	35/50%	0.8-1.1	1-4	7	Max. 3.3 kg/ha/year
	Brussels sprouts	EC/WP	35/50%	0.8-1.1	1-4	14	Max. 3.3 kg/ha/year
	Cabbage, Head	EC/WP	35/50%	0.8-1.1	1-4	7	Max. 3.3 kg/ha/year
	Cauliflower	EC/WP	35/50%	0.8-1.1	1-4	14	Max. 3.3 kg/ha/year
	Cucumber, Melon, Pumpkin & Squash (Summer & Winter)	EC/WP	35/50%	0.55-1.1	1-6	2	Max. 3.3 kg/ha/year
	Peppers	EC/WP	35/50%	0.8-1.1	1-2	1-4	Max. 2.2 kg/ha/year
	Egg plant	EC/WP	35/50%	1.1	1-2	1	Max. 1.1 kg/ha/year
	Sweet corn	EC/WP	35/50%	1.1-2.2	1-3	1	Max. 3.3 kg/ha/year
	Tomato	EC/WP	35/50%	0.55-1.1	1-6	2	Max. 3.3 kg/ha/year
	Collards	EC/WP	35/50%	0.8-1.1	1	21	Max. 1.1 kg/ha/year
	Kale	EC/WP	35/50%	0.8	1	21	Max. 0.8 kg/ha/year
	Lettuce, Head	EC/WP	35/50%	0.8-1.1	1-3	14	Max. 3 app. post-thinning
	Lettuce, Leaf	EC/WP	35/50%	0.8-1.1	1-2	14	Max. 2 app./year
	Mustard greens	EC/WP	35/50%	0.8-1.1	1	21	Max. 1.1 kg/ha/year
	Spinach	EC/WP	35/50%	0.8-1.1	1	21	Max. 1.1 kg/ha/year
	Beans	EC/WP	35/50%	0.55-1.1	1-2	3	Max. 1.1 kg/ha/year Pre-bud forming
	Peas (seed crop)	EC/WP	35/50%	0.55-1.1	1-2	1	Max. 1.6 kg/ha/year
	Carrot	EC/WP	35/50%	0.55-1.1	1	7	Max. 1.1 kg/ha/year
	Potato	EC/WP	35/50%	0.55-1.1	1-6	1	Max. 3.3 kg/ha/year
	Sugar beet	EC/WP	35/50%	0.55-1.1	1-2	30	Max. 2.2 kg/ha/year
	Sweet potato	EC/WP	35/50%	0.55-2.2	1-3	1	Max. 3.3 kg/ha/year
	Artichoke	EC/WP	35/50%	0.8-1.1	1-2	7	Max. 2.2 kg/ha/year
	Celery	EC/WP	35/50%	0.55-1.1	1	4-7	Max. 1.1 kg/ha/year
	Barley, Oats, Rye & Wheat	EC/WP	35/50%	0.28-0.8	1-2	-	Max. 1.1 kg/ha/year
	Pecan	EC/WP	35/50%	0.55-0.8	1-2	-	Max. 3.3 kg/ha/year Pre shuck split

Country	Crop	EC/WP /DP	Conc	Applic. (kg ai/ha)	No.of app.	PHI (days)	Comments
	Walnuts	EC/WP	35/50%	1.6-2.75	1-2	-	Max. 3.3 kg/ha/year
	Cotton	EC/WP	35/50%	0.4-1.6	-	-	Max. 3.3 kg/ha/year Pre boll opening
	Safflower	EC/WP	35/50%	1.1	1-2	1	Max. 2.2 kg/ha/year Pre bud opening
	Sunflower	EC/WP	35/50%	1.1	1-3	1	Max. 3.3 kg/ha/year
	Alfalfa	EC/WP	35/50%	0.28	1-3	21	Max. 0.82 kg/ha/year

RESIDUES RESULTING FROM SUPERVISED TRIALS

Fruits - see Table 2.

Citrus fruits

Clementine. In one trial in Spain in 1992, residues were below 0.1 mg/kg 14 days after application.

Lemon. Residues in lemons were up to 0.16 mg/kg at 7 days and 0.12 mg/kg at 14 days after treatment.

Oranges. Residues from three trials in Spain at GAP rates did not exceed 0.35 mg/kg at 15 days PHI. In five other trials at a slightly excessive application rate, one residue of 0.54 mg/kg was observed at 14 days; all other determinations were below 0.4 mg/kg.

Pome fruits

Apple. From five trials on apples in Germany, a maximum residue of 0.63 mg/kg was found at 21 days PHI. The current CXL for pome fruits is 1 mg/kg.

Stone fruits

Cherry, Sour. In four trials on Morello cherries in Germany in 1983 the maximum residue found at 21 days PHI was 0.03 mg/kg, well within the current CXL of 1 mg/kg.

Peach. Use of a 2.8% dusting powder in five trials on peaches in Germany resulted in residues up to 0.5 mg/kg at 14 or 21 days. Two trials in Spain using 35% EC gave a maximum residue of 0.73 mg/kg at 15 days. None of the trials was within current GAP in these countries.

Plums. In seven trials in Germany, from 1983 to 1989, a maximum residue of 0.38 mg/kg was found at 21 days PHI.

Grapes. Data were provided on 15 trials on grapes in Germany from 1974 to 1987. At a PHI of 60 to 62 days, residues were in the range 0.49-0.6 mg/kg.

Strawberry. Three trials on strawberries in Spain in 1985 showed residues up to 4 mg/kg at 3 days, 2.5 mg/kg at 4 days and 0.84 mg/kg at 13 days after application. This treatment is not GAP in Spain.

Pineapple. Endosulfan is used in Australia for quarantine purposes as a post-harvest dip. Residues up to 2 mg/kg can be expected from this procedure which would be within the existing CXL of 2 mg/kg for "Fruits".

Table 2. Residues of endosulfan in fruit from supervised trials.

Crop/Country/Year	EC/WP/DP/%	Appl.		No. of trials	Residues (mg/kg) at intervals (days) after last appl.	Ref.
		kg/ha	no.			
Clementine						
Spain '92	EC 35	2.1	1	1	0.03-0.22 (7), 0.05-0.10 (14), 0.05-0.09 (28)	Spain 1993
Lemon						
Spain '86	EC 35	4.55	1	1	0.15-0.16 (7), 0.11-0.12 (14), 0.04 (30)	Spain 1993
Orange						
Spain '91	EC 35	7.7	1	5	0.52-0.92 (7), 0.24-0.54 (14),0.12-0.44 (21)	A49713-7
'92		3.7	1	1	0.21-0.29 (7), 0.03-0.06 (14), 0.02-0.05 (28)	Spain 1993
		5.93	1	1	0.78 (3), 0.65 (7), 0.33 (15)	A49712
		6.3	1	2	0.82, 0.88 (3), 0.55,0.64 (7), 0.27, 0.35 (15)	A49710-1
Apple						
Germany'85	EC 35	0.53	4	3	0.46-0.77 (7), 0.48-0.77 (14),0.44-0.63 (21)	A33345-7
'89		0.4-0.47	4	1	0.42 (0), 0.55 (21)	A47390
		0.53	4	1	0.76 (0), 0.11 (21)	A47389
UK '80	SC 43	0.85	2	4	0.045, 0.19 (20), 0.015, 0.095 (21)	A21279-82
Cherry, sour						
Germany'83	DP 2.8	0.71	3	4	0.015-0.21 (7), 0.015-0.02 (14), 0.015-0.03 (21)	A28378-81
Peach						
Germany'83	DP 2.8	0.71	3	5	0.2-1.5 (70, 0.1-0.51 (14), 0.06-0.49 (21)	A28382-6
Spain '92	EC 35	1.68	1	1	1.0 (3), 0.98 (7), 0.73 (15)	A49700
		1.94	1	1	0.88 (3), 0.5 (7), 0.31 (15)	A49001
Plums						
Germany'83	DP 2.8	0.71	5	3	0.15-0.26 (7), 0.05-0.15 (14),0.07-0.09 (21)	A28391-3
'84		0.71	5	2	0.1-0.3 (7), 0.12-0.28 (14), 0.1-0.38 (21)	A30124-5
'89	EC 35	0.26-0.29	5	1	0.22 (0), 0.10 (21)	A46682
		0.53	5	1	0.52 (0), 0.16 (21)	A46681
Grapes						
Germany' 74	EC 35	2.8	1	2	0.7, 0.9 (7), 0.24, 0.3 (14),	A02889,2893
					0.20, 0.25 (21), 0.16, 0.18 (28)	
		2.8	2	2	0.6, 1.2 (7), 0.4, 0.6 (14),	A02887,2891
					0.26, 0.39 (21), 0.16, 0.28 (28), 0.14 (42)	
	WP 35	2.8	1	1	0.4 (7), 0.3 (14), 0.26 (21), 0.18 (28)	A02894

Crop/Country/Year	EC/WP/DP/%	Appl.		No. of trials	Residues (mg/kg) at intervals (days) after last appl.	Ref.
		kg/ha	no.			
			2	2	1.1, 1.2 (7), 0.6, 0.7 (14), 0.4, 0.4 (21),	A02888,2892
					0.26, 0.37 (28), 0.15, 0.19 (42)	
'84	WP 33	0.6+1.2	2	1	1.9 (14), 0.68 (35), 0.55 (60)	A030914
		1.2	2	1	1.3 (19, 0.7 (35), 0.49 (62)	A030915
	CS 35*	1.26	2	1	2.5 19), 1.4 (35), 0.59 (62)	A30909
		0.6+1.3	2	1	1.8 (14), 0.82 (35), 0.6 (60)	A30910
'87	EC 35	0.56	1	4	<0.015 (77-162)	A38806-9
Strawberry						
Spain '85	EC 35	2.1	1	1	0.77, 0.9 (2), 0.42, 0.72 (5), 0.36, 0.65 (8),	Spain 1993
					0.22, 0.36 (13)	
			1	1	2.5, 4.0 (3), 0.8, 1.7 (7), 0.16, 0.52 (14),	Spain 1993
					0.13, 0.14 (21)	
			1	1	2.5, 2.5 (4), 1.8, 1.8 (7), 0.84, 0.84 (13),	Spain 1993
					0.25, 0.25 (21)	
Pineapple						
Australia'90	EC 35	Dip [0.56 g/l]	1	1	1.9 (0), 1.7 (3), 1.4 (7)	Australia, 1993

* CS = Suspension of Microcapsules

Brassica vegetables - see Table 3.

The only residue data from the USA that were presented were from two trials on Brussels sprouts carried out in 1964. At 14 days PHI, residues reached 1.2 mg/kg. From one trial on Brussels sprouts in the UK in 1976, residues reached 0.1 mg/kg at 14 days and 0.06 mg/kg at 21 days PHI. One trial on broccoli at the GAP rate in Portugal gave 0.44 mg/kg at 28 days PHI, just in concordance with the existing CXL of 0.5 mg/kg.

Residues from six trials on head cabbage in Germany and Portugal showed maximum residues of 0.73 mg/kg at 27 days; the current CXL is 1 mg/kg. Data from 26 trials on Savoy cabbage in Germany showed no residues above 0.42 mg/kg at 14 days; the current CXL is 2 mg/kg. On cauliflower, four trials in Germany showed residues below 0.2 mg/kg at 10 days and 0.1 mg/kg at 14 days PHI.

Information from Canada on Chinese broccoli and mustard cabbage showed residues up to 1.3 mg/kg at 7 days.

Table 3. Residues of endosulfan in brassica vegetables from supervised trials.

Crop/Country/Year	EC/WP/DP/%	Appl.		No.of trials	Residues (mg/kg) at intervals (days) after last application	Ref.
		kg/ha	No.			
Broccoli						

Crop/Country/Year	EC/WP/DP/%	Appl.		No.of trials	Residues (mg/kg) at intervals (days) after last application	Ref.
		kg/ha	No.			
Portugal'86	EC 38	0.95	1	1	3 (7), 1.7 (14), 0.71 (21), 0.44 (28)	Portugal, 1993
		3.7	1	1	22 (7), 11 (14), 2.6 (21), 2.0 (28)	Portugal, 1993
Brussels sprouts						
UK '76	EC 35	0.3	1	1	0.80 (7), 0.10 (14), 0.06 (21)	A10195
USA '64	EC 24	0.84	14	2	0.68, 2.8 (7), 0.45,1.8 (10), 1.2 (14)	A48555
Cabbage, Head (White & Red)						
Germany'88	EC 35	0.21	1	4	<0.015 (42-94)	A40703-6
Portugal'85	EC 38	2.5	1	1	3.1 (6), 1.8 (13), 0.9 (20), 0.73 (27)	Portugal, 1993
'90	EC 38	1.1+0.9	2	1	4.3 (7), 2.7 (14), 1.3 (21), 0.62 (28)	Portugal, 1993
Cabbage, Savoy						
Germany'74	EC 35	0.21	1	2	<0.02 (54, 112)	A40701-2
			3	2	0.1, 0.2 (7), 0.1, 0.2 (14), 0.01, 0.03 (21)	A08860-1
		0.35	1	5	0.07-2.2 (7), <0.02-0.4 (14),	A02452,2456,
					<0.08-0.28 (21), <0.08-0.14 (28)	2463,3350,3352
		0.53	1	4	0.05-1.2 (7), <0.08-0.13 (14),	A02460,2464,
					<0.08 (21), <0.18-0.14 (28)	3351,3353
	WP 35	0.35	1	2	0.04, 0.7 (7), <0.08 (14-28)	A02465,3315
		0.53	1	5	0.06-0.7 (7), <0.05-0.18 (14),	A02454,2462,
					<0.08-0.07 (21), <0.08-0.06 (28)	2466,3316-7
'83	WP 33	0.2	3	3	0.1-1.2 (5), 0.25-0.09 (10), 0.02-0.34 (14)	A028372-4
	DP 2.8	0.71	3	3	0.19-1.9 (5), 0.02-0.15 (10), 0.02-0.42 (14)	A028919-21
Cauliflower						
Germany'83	DP 2.8	0.71	3	4	0.02-0.54 (5), 0.02-0.16 (10), 0.02-0.06(14)	A28593-6
Chinese broccoli (Guy Lon)						
Canada '91	EC 40	0.8	1	1	3.5 (0), 0.8 (3), 0.35 (7), 0.05 (13)	Canada, 1993
Mustard cabbage (Bok Choi)						
Canada '91	EC 40	0.8	1	1	5.9 (0), 2,4 (3), 1.3 (7), 0.55 (15), 0.07 (21)	Canada, 1993

Other vegetables - see Table 4.

Cucurbits. At a PHI of 7 days, five trials on cucumbers in Canada showed a maximum residue of 0.17 mg/kg from treatments at slightly above the current GAP rate. Greenhouse cucumbers treated in Spain in 1992 showed a residue of about 0.1 mg/kg at 7 days PHI.

Greenhouse-grown melons in Spain showed residues up to 0.5 mg/kg after 7 days. Residues from trials on squash in Canada and Spain were up to 0.23 mg/kg at 7 days PHI.

Peppers. Two trials on greenhouse peppers in Spain showed residues up to about 0.5 mg/kg at 7 days.

Tomato. Data were available from 28 field trials in Germany and one in Canada. At a PHI of 7 days residues were generally below 0.2 mg/kg, although in one trial a level of 1 mg/kg was found.

Tomatoes treated in greenhouses in Spain showed residues up to 1 mg/kg at 7 and 15 days PHI, but at rates of application that were above the quoted GAP.

Lettuce. Two trials in Canada gave residues up to 0.15 mg/kg at 14 days, well within the current CXL of 1 mg/kg.

Common bean. From 20 trials on common beans in Canada, Germany and Spain, the maximum residue observed at a PHI of 14 days was 0.59 mg/kg. At 3 days PHI, up to 0.97 mg/kg was found.

Broad beans. Twelve trials on broad beans in Germany gave residues up to 1.9 mg/kg at 7 days and 0.04 mg/kg at 21 days. No data were provided at the GAP PHI of 14 days.

Soya beans. Data were supplied from Australia, 2 trials, and Brazil, 42 trials. At the recommended PHI of 30 days, the maximum residue was 0.6 mg/kg; all of the other findings were below this at PHIs ranging from 13 to 101 days.

Potato. Residues from seven trials in Germany were all below 0.015 mg/kg at PHIs from 0 to 28 days.

Celery. Two trials in Canada showed residues well below the existing CXL of 2 mg/kg at 0 to 35 days after treatment.

Table 4. Residues of endosulfan in other vegetables from supervised trials.

Crop Country/Year	EC/WP/ DP/%	Appl.			Residues (mg/kg) at intervals (days) after last application	Ref.
		kg/ha	No.	No of trials		
Cucumber						
Canada' 89	EC 40	0.6	1	1	0.08 (1),0.03 (3),0.09 (7),0.037 (14),0.013 (21)	Canada, 1993
Germany'83	EC 35	0.21	3	1	0.16 (0),0.11 (3),0.07 (5),0.08 (7)	A28059
		0.32	3	3	0.12-0.2 (0), 0.08-0.11 (3), 0.08-0.17 (5), 0.06-0.17 (7)	A28056-8
Cucumber (greenhouse)						
Spain '92	EC 35	1.58	1	1	0.18-0.25 (2), 0.07-0.11 (7), 0.05-0.09 (10), 0.03-0.09 (15)	Spain 1993
Melon (Greenhouse)						
Spain '92	EC 35	0.71	1	1	0.38 (0), 0.05 (3), 0.02 (7), 0.02 (15)	A49703
		0.76	1	1	0.97 (0), 0.63 (3), 0.50 (7), 0.22 (15)	A49704
		0.82	1	1	0.81 (0), 0.28 (3), 0.23 (7), 0.11 (15)	A49702
		0.87	1	1	0.09 (0), <0.01 (3), <0.01 (7), 0.04 (15)	A49705
Squash, Summer ('fuzzy')						
Canada '91	EC 40	0.8	1	1	0.18 (0), 0.09 (3), 0.08 (7), 0.049 (13)	Canada, 1993

Crop Country/Year	EC/WP/ DP/%	Appl.			Residues (mg/kg) at intervals (days) after last application	Ref.
		kg/ha	No.	No of trials		
Squash (Greenhouse)						
Spain '92	EC 35	1.02	1	1	0.32 (0), 0.13 (3), 0.02 (7), 0.02 (15)	A49709
		1.09	1	1	1.1 (0), 0.46 (3), 0.23 (7), 0.03 (15)	A49706
		1.21	1	1	1.0 (0), 0.53 (3), 0.05 (7), 0.04 (15)	A49702
		1.37	1	1	0.11 (0), <0.01 (3), 0.05 (7), 0.02 (15)	A49705
Peppers (Greenhouse)						
Spain '88	EC 35	0.962	1	1	0.54-1.46 (2), 0.08-0.45 (7), 0.03-0.22 (11), 0.03-0.09 (15)	Spain 1993
'89	EC 35	0.962	1	1	0.73-0.93, 0.36-0.54 (7), 0.04-0.57 (10), 0.20-0.48 (15)	Spain 1993
Tomato						
Canada '89	EC 40	0.56	1	1	0.07 (1), 0.03 (3), 0.03 (7), 0.01 (14), <0.01 (21)	Canada, 1993
Germany'74	EC 35	0.21	1	3	0.6-0.9 (0), 0.4-0.5 (1), 0.2-1 (2), 0.03-0.7 (4)	A02392,4,6
			3	1	0.4 (0), 0.2 (1), 0.07 (2), 0.03 (4)	A08854
		0.53	1	2	0.04 (0), 0.02-0.03 (1), 0.03-0.05 (2), 0.03 (4)	A02605,7
		0.70	1	1	1 (0), 0.24 (1), 0.12 (2), 0.11 (4)	A03081
	WP 35	0.21	1	3	0.6 (0), 0.3-0.41 (1), 0.09-0.2 (2), 0.05-0.1 (4)	A02393,5,7
			3	1	0.5 (0), 0.2 (7), 0.06 (10), 0.03 (14)	A08853
		0.53	1	2	0.04-0.1 (0), 0.03-0.04 (1), 0.01-0.03 (2), 0.0.12-0.15 (4)	A02604,6
		0.70	1	1	0.36 (0), 0.12 (1), 0.09 (2), 0.08 (4)	A03082
'76	EC 35	0.21/0.28	3	1	0.1 (0), 0.09 (1), 0.05 (2), 0.02 (4)	A08855
	WP 35	0.21	3	1	0.5 (0), 0.2 (1), 0.06 (2), 0.03 (4)	A08853
		0.21/0.28	3	1	0.05 (0), 0.04 (1), 0.015 (2), 0.015 (4)	A08852
'82	EC 35	0.85	1	1	0.2 (0), 0.055 (3), 0.045 (5), 0.035 (7), 0.045 (10)	A24861
'83	EC 35	0.21	3	1	0.19 (0), 0.085 (3), 0.045 (5), 0.085 (7)	A28257
		0.35	3	1	0.50 (0), 0.92 (3), 0.72 (5), 1.0 (7)	A28256
	DP 2.8	0.71	3	3	0.3-0.3 (0), 0.21 (5), 0.1-0.12 (10), 0.075-0.13 (14)	A28922-4
'85	EC 35	0.32	3	3	0.24-0.46 (0), 0.07-0.15 (30), 0.06-0.16 (5), 0.06-0.21 (7)	A33348-50
'89	EC 35	0.21-0.42	4	2	0.6,0.72 (0), 0.035, 0.095 (7)	A43307-8
Tomato (Greenhouse)						
Spain '92	EC 35	2.10	1	1	1.4 (0), 1.3 (3), 0.43 (7), 0.36 (15)	A49690
		2.26	1	1	2.2 (0), 1.8 (3), 1.0 (7), 1.0 (15)	A49688
		2.31	1	1	1.5 (0), 1.3 (3), 0.43 (7), 0.36 (15)	A49691
		2.63	1	1	0.92 (0), 1.1 (3), 0.42 (7), 0.18 (15)	A49689
Lettuce 1						
Canada '89	EC 40	0.6		2	0.4 (3),0.06 (7),0.12,0.15 (14),0.01,0.1 (21)	Canada, 1993
Common bean						

Crop Country/Year	EC/WP/ DP/%	Appl.			Residues (mg/kg) at intervals (days) after last application	Ref.
		kg/ha	No.	No of trials		
Canada '81	EC 40	0.5	2	1	0.65 (3), 0.43 (7), 0.41 (14)	Canada, 1993
			2	1	0.46 (3), 0.33 (7), 0.24 (14)	Canada, 1993
Germany'74	DP 3.0	0.9	1	6	0.04-2.3 (7), 0.02-0.59 (14), 0.02-0.22 (21), 0.016-0.08 (28)	A05393-8
'83	DP 2.8	0.71	3	4	0.18-1.1 (0), 0.06-0.19 (5), 0.03-0.12 (11), 0.025-0.055 (14)	A28387-90
Spain '92	EC 35	1.21	1	1	2.7 (0), 0.25 (3), 0.04 (7), 0.01 (14)	A49694
		1.34	1	1	2.7 (0), 0.16 (3), 0.04 (7), 0.01 (14)	A49695
		1.79	1	1	2.0 (0), 0.42 (3), 0.05 (7), 0.03 (14)	A49693
		1.89	1	1	2.8 (0), 0.97 (3), 0.10 (7), 0.01 (14)	A49692
		2.00	1	1	2.7 (0), 0.33 (3), 0.08 (7), 0.02 (14)	A49697
		2.10	1	3	1.7-2.2 (0), 0.09-0.28 (4), 0.03-0.23 (7), 0.01-0.05 (14)	A49696, 98,99
Broad beans						
Germany'75	DP 3.0	0.75	1	6	0.01-0.02 (0), 0.02-0.09 (7), 0.015-0.038 (21), 0.015-0.023 (28)	A04910-1, 06758, 06760,2,4
			2	4	0.083 (0), 0.028-0.13 (7), 0.018-0.021 (21), 0.018 (28)	A06759,61,63, 65
'76	WP 35	0.32	3	1	9.3 (0), 1.9 (7), 0.3 (21)	A10200
	DP 3.0	0.75	3	1	11 (0), 1.9 (7), 0.2 (21)	A10199
Soya beans						
Australia'81	EC 35	0.74	1	1	0.015 (21), 0.03 (28)	A30088
		1.47	1	1	0.04 (21), 0.16 (28)	A30088
Brazil '74	EC 35	0.42	3	1	0.22 (62)	A01813
			4	1	0.17 (13)	A01812
'75		0.53	1	1	0.23 (21)	A07560
'77			1	4	0.09 (22), 0.15 (36), 0.33 (66), 0.09 (103)	A13738,0,4,2
			2	3	0.25 (22), 0.42 (36), 0.33 (66)	A13736,1,3
			3	2	0.31 (22), 0.45 (36)	A13737,5
'78			1	6	0.08 (29), 0.1 (31), 0.2 (61,62), 0.03,0.05 (90)	A06115-7,06122-4
			2	6	0.2,0.2 (29), 0.1,0.2,0.3 (31), 0.2 (61)	A16112-4,6119-21
			3	2	0.3 (29), 0.4 (31)	A016111,6118
'79			1	6	0.06 (30), 0.27 (41), 0.11 (62), 0.31 (71), 0.015 (91), 0.11 (101)	A017983-5,91-93
			2	6	0.015,0.3 (30), 0.28,0.56 (42), 0.26 (62), 0.35 (71)	A017980-2,88-90
			2	4	0.60 (30), 0.34 (41)	A017979,17986
Potato						
Germany'76	WP 35	0.21	2	4	0.01-0.015 (13-28)	A08862-5
	DP 2.8	0.71	2	3	0.015 (0-14)	A28588-90
Celery						
Canada '89	EC 40	0.8	1	1	0.38 (0), 0.18 (7), 0.14 (14), 0.11,0.24 (21)	Canada, 1993
			1	1	0.82 (0), 0.24 (21), 0.15 (28), 0.09 (35)	Canada, 1993

Cereals - see Table 5.

Maize. Seventeen trials were carried out in France and Germany. The maximum residue was 0.085 mg/kg after 91 days.

Wheat. Trials on wheat in France (3) and Germany (9) gave residues under 0.11 mg/kg at 14 to 57 days after application.

Table 5. Residues of endosulfan in cereals from supervised trials.

Crop/Country /Year	EC/WP/ DP/%	Appl.		No. of trials	Residues (mg/kg) at intervals (days) after last application	Ref.
		kg/ha	No.			
Maize (grain)						
France '74	EC 35	1.23	1	2	0.015 (91)	A04510,5767
	FG 5	1.0	1	2	0.015, 0.085 (91)	A04509,5766
Germany'75	EC 35	1.05	1	1	0.05 (36)	A06757
'76		0.70	2	4	0.015 (63), 0.010-0.015 (70)	A10205-6,10211-2
		1.05	1	4	0.02 (70), 0.01 (83)	A10201,3,7,9
'83		0.70	2	2	0.015 (54,70)	A28765-6
	WP 33	0.66	2	2	0.015 (54,70)	A28763-4
(FG = Fine Granule)						
Wheat						
France '76	EC 35	0.53	1	3	0.03 (34), 0.10 (36), 0.09 (53)	A08496-8
Germany'80	EC 35	0.21	3	5	0.015 (28-29)	A21772,4,6,8,80
	WP 35	0.18	2	2	0.015 (19)	A24253,5
'82		0.18	2	2	0.06,0.11 (14)	A26338-9

Oilseed - see Table 6.

Cotton seed. From eight trials in Spain the maximum residue at a PHI of 15 days was 0.25 mg/kg.

Rape seed. Results from 24 trials on rape seed in Germany showed residues up to 0.30 mg/kg at 56 days PHI when GAP application rates were used. A level of 0.57 mg/kg was found when over twice the recommended rate was applied.

Sunflower seed. In 5 trials in the USA in 1965 residues up to 0.6 mg/kg were found 63 to 69 days after treatment. ULV application in the Sudan in 1985 gave residues of 0.008-0.036 mg/kg after 109 days.

Table 6. Residues of endosulfan in oilseed from supervised trials.

Crop/Country /Year	EC/WP/ DP/%	Appl.		No. of trials	Residues (mg/kg) at intervals (days) after last application	Ref.
		kg/ha	No.			
Cotton seed						

Crop/Country /Year	EC/WP/ DP/%	Appl.		No. of trials	Residues (mg/kg) at intervals (days) after last application	Ref.
		kg/ha	No.			
Aus'lia'74	EC 35	0.74	13	1	0.01 (44)	A02015
			15	1	0.035 (25)	A02016
Spain '92	EC 35	0.63	1	2	0.35,0.78 (3), 0.27,0.3 (7), 0.05 (15)	A49593-4
		1.0	1	4	0.1-0.62 (3), 0.1-0.25 (7), 0.005-0.25 (15)	A49545-8
		1.11	1	2	0.4,0.4 (3), 0.11,0.11 (7), 0.07,0.10 (15)	A49599-60
Rape seed						
Germany'74	DP 3.0	0.9	1	9	0.24-0.4 (7), 0.09-0.67 (14), 0.14-0.34 (21),	A02610-1,2467,
					0.09-0.33 (28), 0.03-0.07 (53-70)	A024670,73-77
			2	5	0.07 (42), 0.03,0.09 (47), 0.50,0.57 (54)	A02469,72,
						A02616,2895-6
			4	2	0.06 (55), 0.09 (56)	A013208-9
'84	EC 35	0.42	2	3	0.03 (55), 0.30 (39), 0.30 (56)	A12344,13206-7
		0.21+0.42	2	5	0.015 (56,70), 0.02 (76), 0.06 (67), 0.11 (56)	A30122-3,31482-4
Sunflower seed						
Sudan '88	ULV 52	0.84	1	2	0.008-0.036 (109)	A41153-4
USA '65	EC 24	1.12	1	1	0.09 (88)	A38684
			2	2	0.43-0.61 (69), 0.04 (81)	A38683-4
			3	2	0.44-0.60 (63), 0.39 (74)	A38683-4

Beverage seeds - see Table 7.

Cacao. Trials were carried out in Brazil (4) and the Ivory Coast (3). All residues were below 0.02 mg/kg at 28 to 45 days after treatment.

Coffee. Trials on coffee were carried out in Brazil (6), Cameroon (60) and Guatemala (4). Results were all expressed separately as on the surface and in the interior of the green beans. All residues were below 0.05 mg/kg at PHIs ranging from 30 to 205 days.

Table 7. Residues of endosulfan in seeds for beverages from supervised trials.

Crop/ Country/Year	EC/WP/ DP/%	Appl.		No. of trials	Residues (mg/kg) at intervals (days) after last application	Ref.
		kg/ha	No.			
Cacao						
Brazil '82	EC 35	0.35	2	1	0.015(30,45)	A025749
			3	1	0.015(30), 0.02(45)	A025747
		0.7	2	1	0.015(30,45)	A025748
			3	1	0.015(30,45)	A025746
Ivory Coast'83	EC 35	0.25	2	3	0.02,0.15(2), 0.015,0.02(10), 0.015(28)	A028024-6
Coffee						
Brazil '74	EC 35	0.53	2	1	0.05,0.07(45-140)	A04394

Crop/ Country/Year	EC/WP/ DP/%	Appl.		No. of trials	Residues (mg/kg) at intervals (days) after last application	Ref.
		kg/ha	No.			
		0.70	1	1	0.025,0.035(65)	A04396
			2	1	0.025,0.04(100-200)	A04391
			3	1	0.035,0.017(90-180)	A04395
		1.05	1	1	0.035,0.05(135-165)	A04392
			4	1	0.025,0.035(60-90)	A04393
Cameroon'74	EC 35	0.88	1	3	0.028-0.12(180-205)	A05785-7
		1.05	1	2	0.028-0.044(180)	A04383-4
			2	1	0.028-0.044(150)	A05788
Guatemala '74	EC 35	0.76	2	2	0.028,0.041(36,39)	A04381-2
		0.81	1	2	0.028,0.041(30,34)	A04379-80

FATE OF RESIDUES

In storage and processing

Apples. In two trials on apples in Germany in 1989, fruit with residue levels of 0.055 and 0.11 mg/kg gave apple juice containing less than 0.006 mg/kg, while the respective pomaces held 0.08 and 0.18 mg/kg. (Hoechst, A47389-90).

Grapes. Grapes treated in Germany in 1984 which contained residues of endosulfan at levels of 0.49 and 0.55 mg/kg were used to make wines. These contained less than 0.006 mg/kg, while the musts yielded 0.03 and 0.04 mg/kg respectively (Hoechst, A30914-5).

Common beans. In Canada, in 1988, a study was made of the effect of various culinary procedures on residues in beans. The measures used included trimming, water rinsing, boiling, blanching, microwave cooking and freezing. The results obtained are summarized below and show that boiling and trimming off the ends are the major causes of residue loss (Canada, 1993).

Fresh beans (untrimmed)	(a)	0.63 mg/kg	(b)	0.24 mg/kg
Trimmed ends	(a)	2.1 mg/kg	(b)	0.92 mg/kg

Treatment	% Endosulfan removed (a)	(b)
None	0.0	0.0
Water rinse, 30 sec	0.0	17.7
Boiling 10 min	49.1	34.4
Boiling 10 min (French style)	28.5	66.1
Microwave	0.0	1.0
Frozen, 2 months	0.0	0.0
Blanched, frozen	14.3	0.0
Trimmed	13.9	3.1

Wheat. Bread was made from wheat containing residues of endosulfan at 0.19 mg/kg. Residues in wholemeal bread were 0.13 mg/kg, in refined meal bread 0.049 mg/kg and in the bran 0.28 mg/kg.

NATIONAL MAXIMUM RESIDUE LIMITS

The Meeting received information on the following national MRLs.

Commodity	MRL, mg/kg					
	Austr[1]	Belg[2]	Can[3]	Den[4]	Ger[5]	Spain
Alfalfa			0.1			
Apple			2			
Asparagus						1
Barley					0.1	
Berries, etc.		1		2		
Blackberry					1	
Brassicae						1
Broccoli			2			
Brussels sprouts			2			
Cabbages			2		1	
Carrot	0.2					
Cauliflower			1		1	
Celery			1			
Cereals	0.2					
Citrus fruits						1
Clover			0.1			
Common bean			1		1	
Cotton					1	
Cotton seed oil (crude)	0.5					
Cucumbers			1			
Cucurbits						1
Currants, Black				2		
Currants, Red & White					1	
Dairy products			0.1			
Egg plant			1			1
Eggs	0.05					
Fat of meat of cattle	0.2					

Commodity	MRL, mg/kg					
	Austr[1]	Belg[2]	Can[3]	Den[4]	Ger[5]	Spain
Fat of meat of goats	0.2					
Fat of meat of sheep	0.2					
Fruit	2					
Goat milk (fat basis)	0.5					
Grapes			1			1
Hazlenut						1
Hops					10	
Lettuce, Head			2			
Lupins	1					
Meat			0.1			
Melon			1			
Milk (fat basis)	0.5					
Milk products (fat basis)	0.5					
Mung beans	1					
Mushrooms		0.05				
Navy beans	1					
Nuts	0.2					
Oats					0.1	
Oil seeds	1					
Olive						1
Onion	0.2					
Other vegetables	2					
Parsley			0.1			
Peanuts	1					
Pear			2			
Peas					1	
Pepper			1			1
Pome fruit				2	1	
Potato			0.1		0.1	0.1

Commodity	MRL, mg/kg					
	Austr[1]	Belg[2]	Can[3]	Den[4]	Ger[5]	Spain
Poultry meat	0.2					
Pumpkin			1			
Rape		0.05			0.5	
Raspberry					1	
Rice (in husk)	0.1					
Rutabaga			0.1			
Rye					0.1	
Soya bean	1					
Spinach			2			
Squash			1			
Stone fruit			2	2	1	1
Strawberry			1			
Sugar beet			0.1			
Sunflower			0.1			
Sweet corn			0.1			
Sweet potato	0.2					
Tea (dry manufactured)	30					
Tomato			1		1	1
Turnip			0.1			
Water	0.04					
Watercress			0.1			
Wheat					0.1	

[1] Australia [2] Belgium [3] Canada [4] Denmark [5] Germany

APPRAISAL

Endosulfan has been reviewed by the JMPR eight times since 1967, including a major re-evaluation in 1989. At the 24th (1992) Session of the CCPR it was pointed out (ALINORM 93/24, paras 81-86) that the MRLs for head cabbages, Savoy cabbage and cauliflower did not reflect the residues expected from GAP in the USA. MRLs for broccoli, Brussels sprouts, head cabbage and Savoy cabbage were therefore held at Step 7B pending review by the present Meeting. The proposed deletion of the general MRLs for "Fruit" and "Vegetables, except as otherwise listed", as recommended by the 1989 JMPR, was also delayed until after this review.

Information on the current GAP of 21 countries was made available to the Meeting by the manufacturer, including full details from the USA. A large quantity of residue data that had not previously been submitted for review was also provided.

Residue data from supervised trials on many fruits, vegetables, cereals, oilseed and beverage seeds which had not been reviewed previously were also provided and are recorded in the monograph on this compound. The Meeting confirmed that the data emphasised the desirability of withdrawing the current general MRLs for fruit and vegetables and replacing them with MRLS for individual commodities, usually at a lower level. It was also possible to make recommendations for MRLs on some additional crops.

The residue data on oranges were adequate to allow an MRL of 0.5 mg/kg to be recommended but the data on clementines and lemons were only in summary form and thus not sufficient to extend the MRL to the citrus fruit group.

Residues from trials on apples, cherries and plums were within the current MRLs of 1 mg/kg. Data for peach residues supported a similar MRL of 1 mg/kg. Residue data on grapes also allowed an MRL of 1 mg/kg to be recommended but the strawberry data were only summaries and were thus inadequate.

A dip treatment of pineapples with endosulfan is required in Australia for export quarantine purposes. The resultant residues are up to 2 mg/kg, within the CXL for "Fruits", and so an MRL of 2 mg/kg was recommended to cover this post-harvest use.

Unfortunately, the only residue data on brassica crops treated in the USA concerned two trials on Brussels sprouts that were carried out in 1964 and had been reported previously; at a 14-day PHI a maximum residue of 1.2 mg/kg was observed. In one trial on Brussels sprouts in the UK in 1976, 0.1 mg/kg was found after 14 days and 0.06 mg/kg after 21 days but the data were not adequate to support an MRL recommendation. Data from other countries under their GAP conditions were available for broccoli, head cabbage, Savoy cabbage and cauliflower; these results were consistent with the existing draft MRLs of 0.5, 1, 2 and 0.5 mg/kg, respectively.

For some other vegetables, currently covered by the CXL for "Vegetables, except as otherwise listed", the data presented

were sufficient to allow recommendations to be made for broad bean, cucumber, melons except watermelon, summer squash and tomato, all at 0.5 mg/kg, and for soya bean at 1 mg/kg. Data for sweet peppers were inadequate.

Residues on celery (2 mg/kg), common bean (0.5 mg/kg), head lettuce (1 mg/kg) and potato (0.2 mg/kg) were within the respective CXLs.

For cereals, residue data were presented for maize and wheat, allowing recommendations of 0.1 mg/kg and 0.2 mg/kg, respectively, to be made.

Trials on some oilseeds gave sufficient residue data for MRLs to be recommended for cotton seed (1 mg/kg), rape seed (0.5 mg/kg) and sunflower seed (1 mg/kg).

New residue data were also available which allowed recommendations to be made for MRLs on cacao beans and coffee beans, both at 0.1 mg/kg.

Processing data were available for apples (juice and pomace), grapes (wine and must) and common beans (washing and cooking).

RECOMMENDATIONS

On the basis of the data on residues resulting from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits.

Definition of the residue: sum of alpha- and beta-endosulfan and endosulfan sulphate (fat-soluble).

Commodity		Recommended MRL (mg/kg)		PHI on which based, days
CCN	Name	New	Previous	
VP 0522	Broad bean	0.5	2[1]	21
VB 0400	Broccoli	0.5	0.5	28
VB 0403	Cabbage, Savoy	2	2	14
VB 0041	Cabbages, Head	1	1	27
SB 0715	Cacao beans	0.1	---	28
VB 0404	Cauliflower	0.5	0.5	7
VS 0624	Celery	2	2	14
FS 0013	Cherries	1	1	21
SB 0716	Coffee beans	0.1	---	30

Commodity		Recommended MRL (mg/kg)		PHI on which based, days
CCN	Name	New	Previous	
VP 0526	Common bean	0.5	0.5	14
SO 0691	Cotton seed	1	1	15
VC 0424	Cucumber	0.5	2[1]	7
AO2 0001	Fruits	W	2	--
FB 0269	Grapes	1	2[2]	60
VL 0482	Lettuce, Head	1	1	14
GC 0645	Maize	0.1	---	54
VC 0046	Melons, except Watermelon	0.5	2[1]	7
FC 0004	Oranges, sweet, sour	0.5	2[2]	14
FS 0247	Peach	1	2[2]	15
FI 0353	Pineapple	2 Po	2[2]	--
FS 0014	Plums (including Prunes)	1	1	21
FP 0009	Pome fruits	1	1	21
VR 0589	Potato	0.2	0.2	28
SO 0495	Rape seed	0.5	---	56
VD 0541	Soya bean	1	2[1]	30
VC 0431	Squash, Summer	0.5	2[1]	7
SO 0702	Sunflower seed	1	---	63
VO 0448	Tomato	0.5	2[1]	7
AO1 0002	Vegetables, except as otherwise listed	W	2	--
GC 0654	Wheat	0.2	---	14

[1] Previously "Vegetables, except as otherwise listed", 2 mg/kg.

[2] Previously "Fruits", 2 mg/kg.

FURTHER WORK OR INFORMATION

Required (by 1996)

Residue data from supervised trials on brassica crops carried out in the USA under their current GAP.

REFERENCES

(all references are unpublished)

Australia, 1993. Data on GAP and residues supplied by Australia for JMPR.

Canada, 1993. Data on GAP and residues supplied by Canada for JMPR.

Hoechst AG, 1993. A large number of residue reports in the A..... series, as
indicated in the Tables or text as appropriate.

Portugal, 1993. Data on GAP and residues supplied by Portugal for JMPR.

Spain, 1993. Data on GAP and residues supplied by Spain for JMPR.

ETHYLENETHIOUREA, ETU (108)

EXPLANATION

Ethylenethiourea (ETU) is a metabolite and decomposition product of the ethylenebis(dithiocarbamate) (EBDC) fungicides. It was first evaluated in 1974, and MRLs have been established to reflect maximum residue levels in raw agricultural commodities at harvest; they do not include ETU formed from ethylenebis(dithiocarbamate) residues during processing.

ETU was scheduled (ALINORM 93/24A, para 135) for toxicological and residue evaluation by the 1993 JMPR.

Extensive data on ETU residues in raw agricultural commodities and processed foods from supervised trials and in trade, and data on ETU occurrence from the plant and animal metabolism of mancozeb and maneb, were made available to the Meeting.

RESIDUES RESULTING FROM SUPERVISED TRIALS

Residues of ETU resulting from the uses of mancozeb and maneb are summarized in the monographs on those compounds. Mancozeb and maneb have a wide range of approved uses on agricultural and horticultural crops in many countries.

ETU residue data were available on citrus fruits, pome fruits, stone fruits, berry fruits, tropical and subtropical fruits, bulb vegetables, Brassica vegetables, fruiting vegetables, leafy vegetables, legume vegetables, root and tuber vegetables, stalk and stem vegetables, cereal grains, hops, oilseeds, tree nuts, cereal straws and fodders, legume animal feeds and miscellaneous fodder and forage crops.

In many situations ETU residues were low (0.1 mg/kg or less) or undetectable (LOD mostly 0.01-0.02 mg/kg). In those situations where mancozeb or maneb residues were 10 mg/kg or higher, such as in animal feeds, ETU residues were also sometimes higher. Some of these higher residues could be an artefact, because a small percentage of the ethylenebis(dithiocarbamate) residues can be converted to ETU during analysis (Onley *et al*., 1977).

Animal transfer studies on mancozeb with lactating dairy cows and laying hens are summarized in the mancozeb monograph.

In the dairy cow study ETU residues were not detected (<0.01 mg/kg) in milk from the highest feeding group (45 ppm mancozeb). ETU was detected in the thyroid of all animals (5, 15 and 45 ppm mancozeb in the feed), with the highest doses causing the highest levels. Levels in the thyroid decreased during 7 days on residue-free feed. ETU was not detected (<0.01 mg/kg) in the fat from the highest dose group; residues were present in other tissues of the highest feeding group on day 29 (28 days of dosing), but disappeared after 7 days on a residue-free diet. ETU levels in muscle tissue from the highest dose group on day 29 were <0.01-0.034 mg/kg.

In the laying hen study ETU levels in eggs from the highest feeding group (45 ppm mancozeb) were in the range <0.01-0.017 mg/kg. ETU residues were not detected (<0.02-<0.08 mg/kg) in the tissues.

FATE OF RESIDUES

In animals

Mancozeb and maneb metabolism studies on lactating goats and laying hens were made available to the Meeting. Summaries are included in the respective monographs. ETU was generally a minor metabolite, constituting few % or less of the total ^{14}C in tissues, milk and eggs.

In plants

Mancozeb metabolism studies on tomatoes, soya beans, sugar beet and wheat, and maneb metabolism studies on lettuce, potatoes and tomatoes are summarized in the respective monographs.

ETU was generally not detected in the mancozeb plant metabolism studies. In the maneb studies ETU levels expressed as a percentage of the total residue were lettuce 7%, potato peel 0.49%, tomato 7.9%. ETU was not detectable in the potato pulp. A major part of the detected ETU was present in the surface rinsings from the lettuce and tomatoes. Hoagland and Frear (1976) demonstrated that ^{14}C-labelled ETU was absorbed by the petioles and roots of maize, lettuce, peppers and tomatoes, and translocated principally in the xylem.

UV irradiation of ETU on silica gel yielded ethyleneurea as the major identified product (Cruickshank and Jarrow, 1973).

In soil treated with ^{14}C-labelled ETU the half-life of intact ETU was less than one week (Rhodes, 1977). The disappearance of residues from tomato and bean plants treated with radiolabelled ETU is shown in Table 1. The compounds identified by TLC in the extract from tomato plants one day after treatment and their percentages of the extractable ^{14}C were ETU 2%, ethyleneurea 21% and 1-(2-imidazolin-2-yl)-2-imidazolidinethione 75%.

Table 1. Disappearance of residues from tomato and bean plants treated with radiolabelled ETU (Rhodes, 1977).

Days after treatment	Tomato foliage and stems		Bean foliage and stems	
	Total ^{14}C, mg/kg as ETU	ETU, mg/kg	Total ^{14}C, mg/kg as ETU	ETU, mg/kg
0	2.2	0.08	5.5	0.66
1	2.1	0.06	6.2	0.21
3	0.95	0.01	3.9	0.05
7	0.75	0.03	3.0	0.03
14	0.25	<0.01	2.0	0.04
21	0.14	<0.01	1.9	0.01
35	0.06	<0.01	1.5	<0.01

Rhodes (1977) identified by TLC the products of the aqueous UV photodecomposition of ETU by a mercury-vapour lamp (Table 2). ETU was completely degraded (>99%) after 6 hours irradiation with no photosensitiser, and after 3 hours in the presence of acetone.

Table 2. Products identified from the photodecomposition of radiolabelled ETU (Rhodes 1977).

Compound	% of total ^{14}C	
	No photosensitiser	0.1M acetone
Hydantoin	9.3	24
Ethyleneurea	14	7.3
1-(2-imidazolin-2-yl)-2-imidazolidinethione	11	17
Glycine	63	50

Ross and Crosby (1973) showed that ETU in aqueous solution was stable to sunlight, but in the presence of dissolved oxygen and sensitisers such as acetone or riboflavin it was rapidly converted to ethyleneurea and glycine sulphate.

In storage and processing

Processing studies for mancozeb on apples, grapes, sweet corn, tomatoes, potatoes, sugar beet, barley, wheat, maize and peanuts and for maneb on apples, beans, grapes, sugar beet, sweet corn and tomatoes were made available to the Meeting and are summarized in the respective monographs. The studies included data on ETU residues.

Mancozeb and maneb residues, which are on the surface, can be substantially diminished by vigorous washing. The remaining ethylenebis(dithiocarbamate) residues will be converted in part to ETU residues if processing includes a heating step. Levels of ETU in the processed product bear no relationship to the ETU levels in the raw commodity.

Table 3 presents a selection of data from the supervised processing trials. It should be noted that the trials were conducted with exaggerated rates of application to achieve high residues in the raw commodity. The chances of measuring residues in the processed commodity are then improved. The data suggest that the degree of conversion to ETU depends very much on the process.

Table 3. ETU residues in processed commodities in processing trials. Data were selected from more detailed tables in the mancozeb and maneb monographs.

Raw commodity	EBDC residues (as CS_2), mg/kg	Processed commodity	ETU residues, mg/kg
Mancozeb			
Washed apples	2.8	Unclarified canned juice	0.04
		Clarified canned juice	<0.03
Apples	4.7	Apple juice	<0.01
Grapes, de-stemmed and heated	19	Clear juice	2.5
Grapes	9.0	Unfiltered juice	0.025
		Red wine	0.64
		White wine	0.79
Washed tomatoes	0.2	Canned tomato juice	0.09
Potatoes, washed and brushed	<0.06	Baked potato flesh	0.013
Maneb			

Apple	9.7	Fresh juice	0.018
Raw bean pods	3.5	Canned beans	0.49
Raw grapes	6.6	Thick grape juice	5.0
Sugar beet roots	0.069	White sugar	<0.01
Tomatoes, unwashed	0.087	Tomato juice, from paste	0.02

Marshall (1977) showed that conversion of ethylenebis(dithiocarbamates) to ETU during cooking depended on pH. The yields of ETU from two hours refluxing of mancozeb were: pH 2.2 11%, pH 4.0 19%, pH 5.6 70%, pH 8.0 79%.

Studies in the open literature on the fate of EBDC residues during food processing were included in a recent review of the effects of processing on pesticide residues (Holland *et al., in press*).

The effects of typical consumer practices during food preparation on residues of dithiocarbamates were reported by Johnson (1991), and are summarized in the mancozeb monograph. Very little, if any, ETU is produced during the removal of dithiocarbamate residues by washing, scrubbing and drying.

Stability of pesticide residues in stored analytical samples

Studies of the freezer storage stability of ETU in apples, tomatoes, wheat, dry beans, frozen corn, lettuce, raw potatoes, raw tomatoes, meat and milk are included in the mancozeb monograph.

More than 70% of the ETU remained in tomato and wheat matrices after 12 months storage at -20°C, but not after two years. ETU residues in the apple matrix had declined to less than 70% after 6 months storage and to less than 50% after 12 months. ETU residues were shown to be stable in 3-6 month tests at -20 ± 5°C in stored analytical samples of dry beans, corn, lettuce (marginal stability), meat, milk, raw potato (marginal stability), and tomato.

Residues in the edible portion of food commodities

Information on ETU residues is discussed in conjunction with mancozeb and maneb residues in the respective monographs.

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

A number of studies were made available to the Meeting and are summarized in the mancozeb monograph.

Under a US Food and Drug Administration monitoring programme 864 samples of baby foods were monitored for pesticide residues (Yess *et al.*, 1993). ETU residues were detected in 65 samples; the highest levels detected were 0.06 mg/kg.

In 1989-90 in the USA a large survey of food items (approximately 300 samples each of 19 different raw and processed commodities) was conducted for dithiocarbamate and ETU residues (Slesinski, 1990). No measurable residues of ETU (LOD 0.001 mg/kg) were found in 82% of the samples. All ETU residue levels were less than 0.1 mg/kg. A summary of the results is included in the mancozeb monograph.

ETU was not detected (<0.005 mg/kg) in any of 100 commercial grape juice samples in the USA taken from producers using grapes from areas where dithiocarbamate fungicides were used (Honeycutt, 1991).

METHODS OF RESIDUE ANALYSIS

The methods for ETU are reviewed in association with the dithiocarbamate methods in the mancozeb monograph.

APPRAISAL

Ethylenethiourea (ETU) is a metabolite and decomposition product of the ethylenebis(dithiocarbamate) (EBDC) fungicides. MRLs have been established to reflect maximum residue levels in raw agricultural commodities at harvest. ETU was scheduled (ALINORM 93/24A, Appendix IV, Annex I) for periodic (toxicological and residue) re-evaluation by the 1993 JMPR.

Extensive data were made available to the Meeting on ETU residues in raw agricultural commodities from supervised trials, in processed foods from supervised trials, and in raw and processed commodities in trade, and on the production of ETU in the plant and animal metabolism of mancozeb and maneb.

ETU residues in raw agricultural commodities were generally low (0.1 mg/kg or less) or undetectable (LOD mostly 0.01-0.02 mg/kg). Some reported ETU residues could be an artefact of the analysis, because a small percentage of the ethylenebis(dithiocarbamate) residues can be converted to ETU during the determination.

Animal metabolism and animal transfer studies with mancozeb and maneb on lactating dairy cows, lactating goats and laying hens showed that ETU was a minor metabolite and that ETU residues in milk, eggs and tissues arising from ethylenebis(dithiocarbamate) (EBDC) feed residues would normally be very low or undetectable.

ETU was either undetectable or a minor residue in plant metabolism studies with applied mancozeb or maneb. Where ETU was detected, it was mostly in surface rinsings.

ETU was generally short-lived when applied to plant leaves or soil. It was rapidly degraded by UV light.

Ethylenebis(dithiocarbamate) residues are readily converted in part to ETU if processing includes a heating step. Levels of ETU in processed products bear no relationship to the ETU levels in the raw commodities. ETU levels in processed commodities depend on the levels of EBDC which are present at crucial stages where heating takes place and the duration and temperature of that heating.

Under a US Food and Drug Administration monitoring programme (1990-1991) a variety of baby foods (864 samples) were monitored for pesticide residues. ETU residues were detected in 65 samples; the highest levels detected were 0.06 mg/kg. In 1989-90 in the USA a large survey of food items (approximately 300 samples each of 19 different raw and processed commodities) was conducted for dithiocarbamate and ETU residues. No measurable residues of ETU (LOD 0.001 mg/kg) were found in 82% of the samples. All ETU residue levels were less than 0.1 mg/kg. ETU was not detected (LOD 0.005 mg/kg) in any of 100 commercial grape juice samples in the USA taken from producers using grapes from areas where dithiocarbamate fungicides were used.

The Meeting agreed that MRLs for ETU did not assist in deciding whether GAP in the use of EBDCs was being followed. The Meeting agreed to recommend the withdrawal of all MRLs for ETU.

Normally the regulation of a residue in the raw agricultural commodity sets a limit

on the levels in processed food because some or all of the residue is lost during the process. The levels of ETU in the processed commodity bear no relation to the levels in the raw agricultural commodity. ETU is more likely to occur in processed food where it can be generated by the heating of EBDC residues during the process.

Processing trials demonstrate that under some conditions considerable conversion of EBDCs to ETU can occur. Processing studies available to the Meeting showed that an initial commercial washing and cleaning of the raw agricultural commodity removes much of the EBDC, which is a surface residue, and reduces the potential for ETU formation.

The extensive food surveys in the USA, which have included many processed foods, have generally found only low levels of ETU (less than 0.1 mg/kg) and only in a minority of samples (fewer than 20%). The data suggest that, if good processing practices are followed, ETU residues in processed food would rarely exceed 0.1 mg/kg.

The 1990 JMPR reported results of the monitoring of food in commerce or at consumption for ETU in Canada for 1975-1985. Residues in a number of processed products were all below the limit of detection (0.05 mg/kg). Limited 1989-1990 data from Canada on a variety of fruit juices and drinks showed residues to be below 1 µg/kg.

RECOMMENDATIONS

On the basis that ETU residues in raw agricultural commodities are not a useful indicator of good agricultural practice and bear no relationship to potential ETU residues in processed commodities the Meeting recommended the withdrawal of previous recommendations for ETU maximum residue levels.

Definition of residue: ethylenethiourea.

Commodity		Recommended MRL, mg/kg	
CCN	Name	New[1]	Previous TMRL
VR 0577	Carrot	W	0.01*
VS 0624	Celery	W	0.01*
VL 0482	Lettuce, Head	W	0.01*
VR 0589	Potato	W	0.01*

[1] W: the previous recommendation is withdrawn.

REFERENCES

1. Cruickshank, P.A. and Jarrow, H.C. 1973. Ethylene thiourea degradation. J. Agric. Fd Chem. 21, 333-335.

2. Hoagland, R.E. and Frear, D.S. 1976. Behaviour and fate of ethylenethiourea in plants. J. Agric. Fd Chem. 24, 129-133.

3. Holland, P.T., Hamilton, D., Ohlin, B. and Skidmore, M. *in press*. Effects of storage and processing on pesticide residues in plant products. Pure & Appl. Chem.

4. Honeycutt, R.C. 1991. Collection and analysis of produce grape juice for ETU and

EBDCs. Hazard Evaluation and Regulatory Affairs Company, USA. Study 90-113RA, report 91-104. Unpublished.

5. Johnson, G.E. 1991. EBDC/ETU consumer practice study. Project ETU 90-02. Report 90-0084ATO-1. EN-CAS Analytical Laboratories, USA. Unpublished.

6. Marshall, J.W. 1977, Thermal decomposition of ethylenebis(dithiocarbamate) fungicides to ethylenethiourea in aqueous media. J. Agric. Fd Chem. 25, 357-361.

7. Onley, J.H., Giuffrida, L., Ives, N.F., Watts, R.W. and Storherr, R.W. 1977. Gas-liquid chromatography and liquid chromatography of ethylenethiourea in fresh vegetable crops, fruits, milk and cooked foods. J. Assoc. Off. Analyt. Chem., 60, 1105-1110.

8. Rhodes, R.C. 1977. Studies with manganese [^{14}C]ethylenebis(dithiocarbamate) ([^{14}C]maneb) fungicide and [^{14}C]ethylenethiourea ([^{14}C]ETU) in plants, soil and water. J. Agric. Fd Chem. 25, 528-533.

9. Ross, R.D. and Crosby, D.G. 1973. Photolysis of ethylenethiourea. J. Agric. Fd Chem. 21, 335-337.

10. Slesinski, R.S. 1990. EBDC/ETU national food survey. Project ETU 89-01. Report ETU 90-09. Technical Assessment Systems, Inc., USA. Unpublished.

11. Yess, N.J., Gunderson, E.L. and Roy, R.R. 1993. US Food and Drug Administration monitoring of pesticides residues in infant foods and adult foods eaten by infants/children. J. Assoc. Off. Analyt. Chem., 76, 492-507.

FENBUTATIN OXIDE (109)

EXPLANATION

Fenbutatin oxide was first reviewed by the 1977 JMPR for both toxicology and residues. It was scheduled for a periodic re-evaluation by the 1992 JMPR at which time the toxicology re-evaluation was conducted. Owing to the work load and late receipt of data, the residue re-evaluation was postponed until 1993. The 1993 Meeting received residue data and information on GAP for most of the commodities with current CXLs (although in some cases only summary data) and for several commodities currently without CXLs. Extensive GAP information for Europe and some from many other parts of the world was also provided.

The data available to the Meeting included a joint submission to the 1992 JMPR and a 1992 submission to the European Commission (EC). Except for certain studies on apples, grapes, plums and cucumbers in the joint submission to the 1992 JMPR, the EC submission includes (reformatted) most of the residue information available for review by the Meeting. Less comprehensive information was provided for critical supporting studies except processing studies,.

IDENTITY

ISO common name: fenbutatin oxide

Chemical name:
IUPAC: bis[tris(2-methyl-2-phenylpropyl)tin] oxide

Chemical Abstracts: hexakis(2-methyl-2-phenylpropyl)distannoxane

Synonyms: SD 14114, SD 14114-U, "Torque", "Vendex", "Osadan"

Structural formula:

Molecular formula: $C_{60}H_{78}OSn_2$

Molecular weight: 1053

Physical and chemical properties

Pure active ingredient

Vapour pressure: Not volatile. 8.5×10^{-8} Nm^{-2} at 20°C (Shell)

Melting point:	140-145 °C (Fisk, 1991)	
Octanol/water partition coefficient:	1.4 x 10^5 (log K_{ow}	5.15) (Melander, 1988)
Solubility (g/l):	water	5 x 10^{-6} at 23°C (1977 JMPR)
at 20°C:	hexane	3.49 (MacDonald *et al.*, 1992a)
	ethyl acetate	11.4 (MacDonald *et al.*, 1992a)
	propan-2-ol	25.3 (MacDonald *et al.*, 1992a)
	toluene	70.1 (MacDonald *et al.*, 1992a)
	benzene	140 (1977 JMPR)
	methanol	182 (MacDonald *et al.*, 1992a)
	dichloromethane	310 (MacDonald *et al.*, 1992a)
Absolute density (powder):	1.31 g cm^{-3} (Fisk, 1991)	
Hydrolysis:		None up to 30 days in 25°C dark sterile aqueous solutions buffered at pH 5, 7 or 9 (Horne, 1988)

Other properties: (MacDonald *et al.*, 1992b):

EEC Method	Test	Result
A7	Fat solubility	1.1 g/100g at 37 °C
A10	Flammability	none (melts)
A16	Auto-flammability	none

Technical material

Purity: ≥ 97% w/w (1977 JMPR). February-May 1990 5-batch composite yielded 98.8% fenbutatin oxide, other components ≤0.014% except one up to 0.8%. The Meeting was provided with the identity of the components (Broadbent and Smith, undated).

Formulations

Wettable powder: 250 and 500 g ai/kg. Suspension concentrate: 500 and 550 g ai/l
Dusting powder: 20 g ai/kg. 4L Liquid: 42% ai by weight (4 lb. ai/gallon = 479 g ai/l)

USE PATTERN

Several formulations are registered for a variety of crops around the world, including suspension concentrate (SC) formulations at 550 g ai/l, dustable powders (DP) at 2%, wettable powders (WP) at 25 and 50%, and liquid formulations (4L) at 42% by weight. These may act as contact and stomach acaricides for the control of various mites in a variety of fruit, vegetable and field crops. Registered or approved uses were provided in the form of labels, label translations or GAP provided by national authorities. Table 1 summarizes registered and approved uses in countries for which supervised trials data were provided or countries whose GAP is most relevant to the conditions of the reported trials.

Table 1. Summary of fenbutatin oxide approved and registered uses.

Crop Country	Application			PHI, days	Comments
	Formu-lation	Rate per applicn. kg ai/ha (g ai/hl)	No.		
Apples					
Australia	SC	(22)	as reqd.	2	
Austria	SC	(50)	1	21	≥1000 l/ha
Belgium	WP SC	(25) (27.5)	multi multi	28 28	
Canada	SU	0.3-0.9	≤4*	14	*incl. pre-bloom
Cyprus	WP	(30-50)	repeat*	14	*after 10-15 d.
Germany	WP	0.45 (30)	multi*	14	*Reregistration expected 1993
Greece	SC	(22-27.5)		14	
New Zealand	WP	0.5-0.6[1] (20)	repeat* 2-3	14	*after 7-10 d.
Spain (top fruit) pome fruit	WP FL	0.4-1 (25-50) 0.4-0.8 (28-55)	 1-2	21	
USA (same for pears)	4L (liq.)	0.6-1.7 (conc. spray) (15-30) (dil. spray)	≤4*	14	* ≤3 petal fall to harvest
	WP	0.8-1.7 (conc. spray) (15-30) (dil. spray)	same	14	same
Bananas					
Australia	SC	0.2 (≤50)	as reqd.	1	≥400l/ha
Guatemala	WP	(21)		14-21	
Spain	SC	0.4-1.1(27.5)		21	
	WP	0.4-1.1(25)		21	
	FL	0.4-0.8 (28-55)	1	21	
Beans					
France	SC	(50)		7* 3*	*"vines" *veg. crops
Germany (legume veg.)	WP	0.2-0.3 (30)		21	
Netherlands (glass)	WP	(25)	repeat*	7	*after 10-12 d
Spain (green beans)	WP	0.25-0.5 (25-50)	1-3	10	Field or greenhouse

Table 1 (contd.)

Crop/ Country	Application			PHI (days)	Comments
	Formu-lation	Rate per applicn. k gai/ha (g ai/hl)	No.		
Citrus					
Australia	SC	(11-25)	repeat*	7	*as required
Brazil	SC	(30)	repeat*	14	*as required
Cyprus	WP	(30-50)		7	
Greece	SC	(22-27.5)		**1**	
Guatemala	WP	(28-50)		14	
Italy	SC WP	(50) (30-40)		60 60	
Spain	SC DP WP FL	(28-55) 0.4-0.6 (25-50) 1.1-3.3 (28-55)	 1	21 15 21 21	1500-2000l/ha 1500-2000l/ha 4000-6000l/ha
Uruguay	SC	(30-40)		14	
USA	WP	1.1-2.2 [conc. spray] (15-30) [dil. spray]	2 (max./ 12 mo.[2]	7	≥60 days between sprays
	4L (liq.)	same as WP	same as WP	7	same as WP
Orange or tangerine					
Brazil	SC	(40)		14	
Cucumber					
Belgium (under glass)	WP	(25)	repeat*	3	*after 10-12 days
Canada (glass)	WP	(25)	repeat*	3	* as required
Denmark (under glass)	WP	(25)		3	
France	SC	(50)	*	3	*flowering and during honey dew
Germany (fruiting veg.)	WP	0.2-0.3 (30)		4	
Hungary (field or glass)	WP	0.25-0.3 (20-50)*		3	*600-1500l/ha (more dilute in greenhouse)

Crop/	Application			PHI,	

Country	Formu-lation	Rate per applicn. kg ai/ha (g ai/hl)	No.	days	Comments
Cucumber					
Italy	SC WP	(50) (30-40)		30 30	
Netherlands (glass)	WP	(25)		3	
Poland (under cover)	WP	0.25-0.5 (25)	repeat*	3	*if necessary
Spain (cucurbits)	SC	0.4-1.1 (28-55)		8	
	DP WP	0.4-0.6 0.4-1 (25-50)		21 10	
Switzerland (glass)	FL WP	0.28-0.55* (25)	1-3 repeat*	10 3	* 28-55 g ai/hl *after 3-7 days
UK		(25)	*	3	greenhouse *No information
Egg plant					
Belgium (glass)	WP	(25)	repeat*	3	*after 10-12 days
Netherlands (glass)	WP	(25)		3	
USA	WP or 4L liq.	1.1-2.2	≤6	3	≤6.7 kg ai/ha per annum
Grapes					
Austria	SC	0.2-0.3 (28)		21	
Cyprus	WP	(30-50)		7	
France	550g/l	0.35		28	Dual pack applied in mix (no label)
	SC	(50)	*	7	* flowering and honeydew
	WP	0.5 (50)		7	(no label)
Germany	WP	(25)	≤2	28	Reregistration expected 1993
Hungary	SC	0.6-0.7		10	1200-1500 l/ha

Table 1 (contd.)

Crop/	Application	PHI,	

Country	Formu-lation	Rate per applicn. kg ai/ha (g ai/hl)	No.	days	Comments
Grapes cont'd					
Italy	SC	(50)		45	
	WP	(30-40)		45	
Netherlands	SC	(25)	multi*	42	* with egg hatch blossoming after harvest
Spain	SC	0.4-1.1 (28-56)		21	
	WP	0.4-1 (25-50)		21	Field
	FL	0.17-0.33 (28-55)	1	21	*up to 56 g ai/hl
Switzerland	SC	0.6-1.1 (28-33)*		21	* same
	WP	0.5-1 (25-30)		21	
	WS*	0.5 (23)			* mix, 4.5% f. oxide
USA	WP	0.7-1.4 (30-60)	≤2*	28	* 28 d. between applications
	4L liq.	same	same	same	same
Hops					
Australia	SC	(22)	repeat*	2	*as required
Germany	WP	0.2-0.9 (30)		14	
Greece	SC	(23-28)		1	
Japan	WP	(16-25)		45	

Table 1 (contd.)

Crop/ Country	Application			PHI, days	Comments
	Formu-lation	Rate per applicn. kg ai/ha (g ai/hl)	No.		
Melons					
Belgium	WP	(25)		3	
France	SC	(50)	*	3	*during flowering and honeydew
Germany (fruiting veg.)	WP	0.2-0.3 (30)		4	
Netherlands (incl. glass)	WP	(25)		3	
Spain (cucurbits)	SC WP	see cucumber see cucumber		8 10	
Switzerland (glass)	WP	(25)		3	
Nuts					
Almonds, walnuts					
Cyprus	WP	(30-50)		14	
USA	WP or 4L liq.	0.7-1.4 [conc. sprays] (15-30) [dil. sprays]	≤2*	14	* per season; 4700 l/ha max.
Pecans					
USA	WP or 4L liq./	see almonds/ walnuts	≤2*	14	* same
Peppers					
Belgium (glass)	WP	(25)	repeat*	3	* as required after 10-12 d.
Denmark (glass)	WP	(25)		3	
Germany (fruiting veg.)	WP	0.2-0.3 (30)		4	
Hungary (paprika, incl. glass)	WP	0.3*			* 600 l/ha open, 1000- 1500 glass
Netherlands (incl. glass)	WP	(25)		3	

Table 1 (contd.)

Crop/ Country	Application			PHI, days	Comments
	Formu-lation	Rate per applicn. kg ai/ha (g ai/hl)	No.		
Pears					
Australia	SC	(11-22)	repeat*	2	*as required
Belgium	WP	(25)	repeat*	28	*after 15 d.
	SC	(28)	repeat*	28	*after 15 d.
Germany (pome fruit)	WP	(25)	repeat*	14	*after 1-2wk. Reregistration expected 1993
Netherlands	WP or SC	(25)		28	
New Zealand (pome fruit)	WP	0.5-0.9 (20)	repeat*	14	*after 7-10 d.
Spain (top fruit)	WP	0.4-1 (25-50)		21	
USA see apples					
Raspberries					
Netherlands	SC	(25)		*	*pre-bloom spray
Poland	WP	0.5*			*750-1000 l/ha; pre-bloom and post-harvest
Soya beans					
France (beans)	SC	(50)	*	7	* flowering & during honeydew
Germany (legumes)	WP	0.2-0.3 (30)		21	
Spain (beans)	SC	0.4-1.1 (28-55)		8	
	WP	same		10	
Stone fruits					
Germany	WP	0.5 (25)	repeat*	14	*after 1-2 wk
New Zealand	WP	0.5-0.6[1] (20)	2-3*	21	*as reqd. Oct.-Dec.
Poland	WP	0.5		14	500-1000 l/ha

Table 1 (contd.)

Crop/ Country	Application			PHI, days	Comments
	Formu-lation	Rate per applicn. kg ai/ha (g ai/hl)	No.		
Stone fruits cont'd					
Cherries					
Germany	WP	(25)	repeat*	14	* after 1-2 wk Reregistration expected 1993
Italy	SC	(50)		30	
	WP	(30-40)		30	
Netherlands	SC	(25)		42	incl. Morrello
Spain	FL	0.42-0.83 (28-55)	12	21	Field
USA (sweet and sour)	WP or 4L liq.	0.8-1.7 [conc. spray] (15-30) [dilute spray]	≤2*	14	* with fruit on tree; ≤ 5640 l/ha for dilute spray
Peaches					
Australia	SC	(11-22)	repeat*	14	* as required
Denmark (glass)	WP	(25)		28	
Germany	WP	(25)	repeat*	21	*after 1-2 wk; Reregistration expected 1993
	WP	0.5 (30)		21	
Hungary	SC	0.6-0.7		14	1200-1500 l/ha
Italy	SC & WP	see cherries		30	
USA	WP or 4L liq.	0.6-1.1 [conc. spray] (15-30) [dilute spray]	≤2	14	≤4760 l/ha for dilute spray
Plums					
Germany	WP	(25)	repeat*	14	*after 1-2 wk Reregistration expected 1993
Hungary	SC	0.6-0.7		21	1200-1500 l/ha
Italy	WP	see cherries		30	
Netherlands	SC	(25)		42	
USA	WP or 4L Liq	see peaches	see peaches	14	see peaches

Table 1 (contd.)

Crop/ Country	Application			PHI, days	Comments
	Formu-lation	Rate per applicn. kg ai/ha (g ai/hl)	No.		
Stone fruits contd.					
Nectarines					
Australia	SC	(11-22)		14	
USA	WP & 4L liq.	see peaches	see peaches	14	see peaches
Strawberries					
Australia	SC	0.17(<8.5)* 0.22(8.5-55)* 0.39(96-193)* (40)[3]	repeat as req. (up to 3)	1	* >2000 l/ha * 400-2000 l/ha * 200-400 l/ha
France	SC	(50)	*	5	* flowering & during honeydew
Germany	WP	(25)		*	* pre-bloom & post-harvest registration pending
	WP	0.5 (30)		*	* pre-bloom & post-harvest; 1500 l/ha
Netherlands	SC	(25)		*	* pre-bloom & post-harvest
UK (glass)	WP	(25)		7*	* 3 for "tunnel" grown
USA	WP or 4L Liq.	0.6-1.1 [conc. spray] (45-60) [dilute spray]	≤4*	1	* per season
Tomatoes					
Belgium (glass)	WP	(25)	repeat*	3	*after 10-12 d.
Canada (glass)	WP	(25)	repeat*	5	* as required
Denmark (glass)	WP	(25)		3	
Germany (fruiting veg.)	WP	0.2-0.3 (30)		4	600-1000 l/ha

Table 1 (contd.).

Crop/ Country	Application			PHI, days	Comments
	Formu-lation	Rate per applicn. kg ai/ha (g ai/hl)	No.		
Tomatoes cont'd					
Hungary (incl. glass)	WP	0.25-0.3		4	600 l/ha in open; 1000-1500 l/ha glass
Italy	WP SC	(30-40) (50)		30 30	
Netherlands (glass)	WP	(25)	repeat*	3	* after 10-12 d.
UK (glass)	WP	(25)	as required	3	≥10 d. between applicns.
Tea					
Japan	WP	(16-25)	1	21	excludes tea grown under cover

[1] Label rate cites 20 ai/hl and 2500-4500 l/ha (0.5-0.9 kg ai/ha). New Zealand government submission cites maximum GAP of 20 g ai/hl and 0.6 kg ai/ha.
[2] Previous US labels permitted up to 4 applications per season.
[3] Label rates given in ml product/ha (converted to kg ai/ha in Table 1), with repeat applications. The g ai/hl rates are calculated from the range of label volumes cited for each g ai/ha rate. A 1992 JMPR submission cites 0.039% ai and maximum of 3 applications.

RESIDUES RESULTING FROM SUPERVISED TRIALS

In plants

The Meeting received 251 individual studies or reports representing 456 supervised trials on 29 different commodities (and processed products from some of them) in many countries. Supervised trials data are summarized in 7 tables:

Table
2. Selected crops - avocado, bananas, beans, cucumbers, egg plant, hops, melons, peppers, soya beans and tomatoes.
3. Citrus
4. Grapes
5. Tree Nuts
6. Pome fruit
7. Raspberries and strawberries.
8. Stone fruit

Underlined residues in the Tables are from treatments approximating GAP.

The analytical methods for fenbutatin oxide and the metabolites dihydroxybis(2-methyl-2-phenylpropyl)stannane (SD 31723) and 2-methyl-2-phenylpropylstannoic acid (SD 33608) are described in the section "Methods of residue analysis". Methods used in individual trials are referred to by their code numbers in the paragraphs on the commodities concerned.

Avocado. Supervised trials data (Table 2) were available from Australia and the United States. Trials on single trees with 3 replicates/tree and application by boom sprayer were carried out in Australia and trials with

multi-tree replicates and ground hydraulic sprayers in the USA. Results were generally uncorrected for controls or recoveries (mostly >80%) and samples were shipped and stored in an acceptable manner.

The analytical method SAMS 215-1 was used in the Australian trials and MMS-R-494-2 in the US trials.

Maximum residues ranged from 0.23 mg/kg at day 0 to 0.08 mg/kg after 28 days from maximum rates and 0.18 and 0.11 mg/kg at days 0 and 14 respectively from rates comparable to GAP for "stone fruit" in other countries. In two of the three studies samples were also analyzed for the metabolites dihydroxybis(2-methyl-2-phenylpropyl)stannane (SD 31723) and 2-methyl-2-phenylpropylstannoic acid (SD 33608). In the representative data provided neither was detected at a 0.01 mg/kg limit of detection, except in one sample with a residue of 0.02 mg/kg SD 33608.

Bananas. Results of supervised trials which appeared to be according to GAP were available from three locations and two years in Australia. Although application rates were in terms of spray concentration whereas GAP information was in g ai/ha, information on minimum spray volumes indicated that GAP rates were used. Applications were by backpack mister to run-off and transit and storage conditions were acceptable. The analytical method was SAMS 215-1. Recoveries were generally $\geq$90%. The interval from the last sampling to receipt at the laboratory was less than 2 months. The period of storage before analysis was not stated, but a period of less than 6 months can be deduced. No sample chromatograms were provided.

Results (Table 2) were not corrected for recovery or controls and reflect 1 to 4 applications at 20-40 g ai/hl. In one study peel weights averaged 39% of the total fruit weight. Most of the residues were in the peel (pulp residues were approximately 1-2% of the whole banana level). Whole banana residues are proportional to the spray concentrations used and increase with the number of applications. Residue levels in whole bananas on the day of application ranged from 1.6 mg/kg after 4 applications at 10 g ai/hl to 5.7 mg/kg from four applications at 40 g ai/hl. Residues from single applications at the same two rates ranged from 0.6 to 3.4 mg/kg after 2 days. Maximum pulp residues under all conditions were 0.14 mg/kg.

Beans. Supervised trials data were available from outdoor use of a mixed EC formulation on green beans in France and from two glasshouse trials with a WP formulation on French beans in The Netherlands (Table 2).

The French trial was on a 5 x 2.4 m plot with no replicates. Sample storage conditions were acceptable. One month elapsed between the last sampling and receipt of frozen samples, and extraction was three weeks later. Although the period from extraction to analysis was not stated, the date of the report indicated that it would have been less than three months. The method was MMS-R-494-2 for which the limit of detection in beans was reported as 0.01 mg/kg. The mean recovery was 91% a 0.1 mg/kg fortification level. Results were not corrected for recovery. Sample chromatograms were not provided.

The maximum residue from the single application to green beans was 0.5 mg/kg at the French PHI of 3 days for vegetables, although the field trial application rate of 0.4 kg ai/ha (75 g ai/hl) was higher than the 50 g ai/hl French GAP rate for SC formulations (no French GAP provided for mixed EC formulations). The ai/ha rate is comparable to the German 0.3 kg ai/ha GAP rate for a WP on legume vegetables, although the German rate is only 30 g ai/hl and the German PHI is 21 days. The g ai/hl field rate in the French trials is 3 times that for The Netherlands glasshouse WP use.

Four replicates were taken in glasshouse trials on French beans in The Netherlands. Samples were received in cool condition 1-2 months after the last sampling and were stored under acceptable conditions. The interval from laboratory receipt to analysis was not reported, but a period of less than one month can be deduced. The analytical method for fenbutatin oxide was SAMS 215-1 and for SD 31723 SAMS 121-1. Recoveries were 95% of fenbutatin oxide and 65% of SD 31723, both at 0.5 mg/kg fortification.

Maximum residues in whole French beans after 6 days in The Netherlands glasshouse trials were 0.4 mg/kg from applications at 20 g ai/hl compared to The Netherlands GAP of 25 g ai/hl with a 7-day PHI. No sample chromatograms were provided. No residues of metabolite SD 31723 were detected by TLC at a 0.1 mg/kg limit of detection.

Cucumber. Supervised trials data were available from four European countries and the United States, although available information indicates that the use is not GAP in the USA where the outdoor application rates in terms of both kg ai/ha and concentration were greater and PHI intervals shorter than European GAP. Generally the interval from harvest to laboratory receipt or extraction in the European trials was less than 4 months and sample handling and storage appear to have been adequate. However, the interval from receipt or extraction to analysis was not reported. This information was provided for the US studies, for which the sampling-to-analysis intervals were generally $\leq$ 2 months. While the US results may not reflect current GAP, they are useful to illustrate the effect of varying application rates or numbers of applications under the US trial conditions. While Table 2 summarizes only the results of US applications at 2.2 kg ai/ha, data from trials at 4.4 kg ai/ha were also provided and showed an increase in residue levels roughly proportional to the application rate.

Table 2 summarizes the data for single European applications (with one exception), with 9 trials representing GAP. Residues reflecting GAP ranged from 0.03 to 0.3 mg/kg fenbutatin oxide from the single applications. Residues of fenbutatin oxide in peeled cucumbers at PHIs near GAP were $\geq$33% of the whole- fruit residues. The US trials showed no residues of metabolites SD 31723 or SD 33608 after 2 days.

Egg plant. Residues were 0.15, 0.11 and 0.07 mg/kg 0, 7 and 14 days after a single application to egg plants in a French glasshouse trial (Table 2, reference 15) from 50 g ai/hl applications of an SC formulation. Although no GAP information was provided for France, one or more applications at 25 g ai/hl with a PHI of 3 days is GAP in other European countries.

Gherkins. Summary data were provided from The Netherlands on a trial in which a single application of a 50% WP formulation to glasshouse gherkins at 0.75 kg ai/ha gave rise to residues of 1.8, 0.8, 0.7 and 0.9 mg/kg fenbutatin oxide after 0, 3, 5, and 10 days respectively (Shell Chemie, 1974).

Hops. Supervised trials data were available from Australia and Germany (Table 2). No results from applications at GAP rate were available at the Australian 2-day PHI, although they were available for dry cones at 1 day, 3 days and later intervals. Maximum residues were 5.2 mg/kg from GAP application rates and up to 16 mg/kg from double rates. Maximum residues in green cones in the German trials were 0.3 mg/kg at the German 14-day GAP PHI, but from exaggerated application rates.

The analytical method used in the Australian trial was SAMS 215-1 with recoveries of 110% at 0.2 mg/kg fortification and recoveries were <0.2 mg/kg. The analytical method in the German trial was SAMS 345-1 with recoveries of 102% at 1.5 mg/kg. Controls were 0.01 mg/kg.

Melons. In a supervised French trial on Vedrantais melons in 1976 no residues of fenbutatin oxide or its metabolite SD 31723 (<0.01 mg/kg fenbutatin oxide, <0.1 mg/kg SD 31723) were detected 7 or 14 days after a single application of a WP formulation at 0.09 g ai/ha (30 g ai/hl). The French GAP PHI is 3 days. The analytical method was SAMS 215-1 for which fenbutatin oxide recoveries were 80% at 0.2 mg/kg with control values <0.01 mg/kg (Table 2, reference 18).

Peppers. Two supervised trials were available, one outdoor trial on red peppers in Belgium and one glasshouse trial on paprika peppers in The Netherlands. In the Belgian trial residues were 1 mg/kg at the Belgian glasshouse 3-day PHI from a single application (repeat permitted) at

glasshouse GAP rates. Residues in The Netherlands trial were 0.6 mg/kg at the 3-day GAP PHI from a single application at 0.5 kg ai/ha compared to GAP of 25 g ai/hl (Table 2 references 19 and 20).

Soya beans. In three supervised trials in France, 1 or 2 applications at 0.5 kg ai/ha (100 g ai/hl), twice the GAP spray concentration, gave no detectable residues (<0.01 mg/kg) 67-80 days after application. The French GAP PHI is 7 days however (Table 2, reference 21).

Tomatoes. Seven studies in 6 countries, involving 12 supervised trials, were available to the Meeting. Eight of the 12 trials were in glasshouses. The maximum residues reflecting GAP were 0.4 mg/kg in Denmark (glasshouse), 0.3 mg/kg in Italy (field), and 0.3 mg/kg in the UK (glasshouse). In the French glasshouse trial residues were up to 0.08 mg/kg 14 days after applications in accordance with the Italian application rate (no French GAP was provided). Glasshouse trials in The Netherlands were at 0.5 kg ai/ha whereas GAP in The Netherlands is 25 g ai/hl. Information was not sufficient to determine whether the trials reflected GAP. Data from South African field trials could also not readily be related to the GAP information available. No tomato processing data were provided.

In the South African trials no residues (<0.1 mg/kg) were found of the metabolite SD 31723.

The interval from laboratory receipt of the samples to analysis was ≤8 months except for 11 months in the French trials and 15 months in the South African trials. Samples were generally received and/or stored in an acceptable manner. The analytical methods were SAMS-215-1, MMS-R-345-1 and SAMS-332-1. The last included methylation with methyllithium/lithium bromide and GLC determination with an FPD. This appears to be similar to MMS-R-494-2. Analytical recoveries ranged from 80 to 110% at fortification levels varying from 0.1 to 1 mg/kg and apparent residues in untreated samples reported as ≤0.1 mg/kg.

Table 2. Residues of fenbutatin oxide and metabolites SD 31723[1] and SD 33608[2] in selected crops resulting from supervised trials.

Crop Country Year	Application			Residues, mg/kg, at intervals (days) after last application	Ref
	Form	No	Rate kg ai/ha (g ai/hl)		
Avocado			Day	0 7 14 21 28	
				One tree (3 replicates) (mature, flesh):	
Australia 1982	55 SC	2	(20)	0.11 0.09 0.11	1
(avocado pears)			(40)	0.18 0.11 0.10	
				controls 0.02	
USA 1983				Single trees replicated 5 times (mature, flesh):[3]	2
	WP	3	2.8	0.09 0.18 0.09 0.05 0.03	
			(30)	0.08 0.07 0.09 0.08 0.03	
				0.07 0.08 0.08 0.04 0.02	
				0.07 0.10 0.07 0.03 0.02	
				0.06 0.06 0.12 0.04 0.04	
				5 trees/replicate; 3 replicates (mature, flesh):[3]	
1983	SC	3	2.8 (83)	0.07 0.07 0.05 0.08 0.07	3
			5.6 (165)	0.23 0.12 0.13 0.12 0.08	

Crop Country Year	Application			Residues, mg/kg, at intervals (days) after last application						Ref
	Form	No	Rate kg ai/ha (g ai/hl)							
				controls <0.01						
Banana			Day	0	7/8	14	16/17	21	Sample	
Australia	SC	4	(20)	0.05	0.03		0.04		pulp	4
Tully 1982			(40)	0.07	0.14		0.13		pulp	
				controls 0.02						
Euramo 1984	SC	4	(10)	1.6	1.7	1.6	1.0		whole	5
			(20)	3.2	3.5	3.1		2.2	whole	
				0.02	0.02	0.01		0.02	pulp	
			(40)	5.7	6.3	4.8		4.6	whole	
			GAP (≤50)	controls 0.01 pulp, 0.12 whole						
			Day	0	1	2	7/8	14	21	
Australia	SC	1	(10)			0.6	0.4	0.2	whole	6
Yanding 1981						<0.01	<0.01	<0.01	pulp	
			(20)			1.2	0.8	1.0	whole	
						0.02	<0.01	<0.01	pulp	
			(40)			3.4	2.7	2.3	whole	
						0.08	0.04	0.02	pulp	
				controls <0.01 whole and pulp						
Beans, green (whole)			Day	3		6		7	10	
France 1982 (outdoor)	EC	1	0.4 (75)	0.5				0.4	0.4	7

French beans (whole)				3 days	6 days					
Netherlands 1978	WP	2	1 (20)	0.8	0.6					8
(glasshouse)				0.7	0.4	Replicate sub-samples				
				0.7	0.3	Replicate				
				0.7	0.4	Replicate				
1978	WP	2	0.5 (45)	0.6	0.2	Controls <0.02				
Cucumbers			Day		0	2	3/4	7/10		
Denmark 1977	WP	1	0.63 (25)				0.03			9
		2					0.07			
				controls		<0.01				
				SD 31723			<0.1			
France 1980	WP	1	0.75 (50)	whole	0.1	0.1		0.03		10
glasshouse				pulp	0.03	0.03		<0.01		
				control <0.01						
			Day		0	1	2	3/4	5/6	7/10
France 1980	WP	1	0.5 (50)	whole	0.1			0.08		0.04	10
glasshouse				pulp	0.03			<0.01		<0.01	
			0.95 (50)	whole	0.4			0.1		0.07	
			0.95 (50)	pulp	0.08			0.03		<0.01	
				untreated <0.01							
1982	EC	1	0.64 (38)	whole	0.08	0.09				0.07	
glasshouse				pulp	<0.01	<0.01				<0.01	
				untreated <0.01							
Netherlands	WP	1	(25)	whole	0.2			0.1	0.1	<0.1	12
1974		1	(25)	whole	0.2			0.2	0.2	<0.1	
				untreated <0.1							
UK 1980	WP	1	(50)	whole	0.3	0.3	0.2	0.2	0.2		13
		1	(50)	whole	0.3	0.3	0.4	0.3	0.1		
		1	(100)	whole	1			0.5	0.3		
			Day		1		2				
USA 1988 FL	SC (4L)	2	2.2 (1800)		1		0.87				14
		3			1.1		0.6				
PA		2	2.2 (790)		0.4		0.2				
		3			0.3		0.3				
IN		2	2.2 (600)		0.2		0.4				
		3			0.5		0.2				
USA 1988 WI	SC (4L)	2	2.2(980)		0.3		<0.05				
		3			0.5		0.2				
CA	WP	2	2.2 (620)		0.5		0.2				
		3			0.4		0.2				
		2	2.2 (600)		0.7		0.2				

		3		0.9		0.5				
	SC (4L)	2	2.2 (480)	0.5		0.4				
		3		0.7		0.3				
		2	2.2(650)	0.3		0.09				
		3		0.3		0.2				
TX	SC (4L)	3	2.2 (480)	0.9		1				
		2	2.2 (610)	0.8		0.8				
			3	1		1.3				
				Controls ≤0.07 mg/kg SD 31723 & SD 33608 not detected (<0.05 mg/kg)						
Egg plant			Day	0	7	14				
France 1983 glasshouse	SC	1	1 (50)	0.15	0.11	0.07				15
Hops			Day	0	1*	3*	7	22		
Australia	SC	2	0.4 (22)	7.5	5.8	3.5	5.2	5.1		
1988 dry cones		1	0.8 (44)	11.2	16	16	14		* GAP = 2 days	16
			Day	0	7	10	14	23		
Germany 1989 green cones[4]	SC	3	4.5 (150)[5]	2.8	2.6	1.4	0.3	0.01		17

Melons			Day	0 3 7 14	
France 1976 Vedrantais	WP	1	0.09 (30)	<0.01 <0.01	18
Peppers					
Belgium 1975	WP	1	(25)	1.2 1 0.9 0.6	19
red pepper				controls <0.1	
Netherlands 1975 paprika (glasshouse)	WP	1	0.5	0.4 0.6 0.4 0.1 controls <0.1	20
Soya beans					
France 1988 Kador	SC	1-2	0.5 (100) [500l/ha]	65-70 days <0.01 (3) [3 trials, three locations]	21
Tomato			Day	0 1 3 7 14 30	
Denmark 1981	WP	1	0.75 (25)	0.4 0.4 0.5 (5 d.)	22
Ida glasshouse				0.3 (7 d.) controls <0.01	
France 1983 Foxy glasshouse	C	1	0.5 (50)	0.15 0.04 0.08	23
1983 Pyros HFI	SC	1	0.75 (50)	0.1 0.06 0.06 controls <0.02	
Italy 1974 Tonda Liscia	WP	1	(30)	0.4 0.2	24
1974 S. Marzano				0.5 0.3	
				controls <0.1	
Netherlands 1975 Sonate glasshouse	WP	1	0.5 [GAP= (25)]	0.2 0.4[2] 0.4 0.4	25
				controls <0.1	
S. Africa 1976	WP	1	1.5 (20)	0.5 0.4,4d. 0.4 0.2	
Heinz			3 (40)	1 0.7 0.8 0.6 control <0.1; SD 31723 <0.1	
UK glasshouse			Day	0 1 2 3 4 6 7	
Solatine 1980	WP	1	(50)	0.7 0.6 0.9 0.7 0.7	27
				0.3 0.2 0.3 0.4 0.3	
Eurocross 1974	WP	1	(100)	0.8 1 0.8 0.9 1.1 controls <0.01	28
			(25) [GAP]	0.2 0.3 0.4 controls <0.1	

Unless otherwise indicated, residues are parent compound only.

Notes to Table 2
[1] SD 31723 = dihydroxybis(2-methyl-2-phenylpropyl)stannane
[2] SD 33608 = 2-methyl-2-phenylpropylstannoic acid
[3] Representative data
[4] The Volume 9 detailed report gave no information on stage of crop, while the Volume 1 summary stated green cones.
[5] Rate is from Volume 1 of 1992 EC summary submission. The volume 9 detailed report gives application rates of 0.075% ai or 0.225 and 0.375 g ai/plant. Insufficient information was available to relate g ai/plant to g ai/ha. No explanation was provided for the discrepancy between the ai concentrations.

Citrus (Table 3). There were 27 studies in 5 countries representing 53 trials (38 from the USA) on oranges (29), lemons (9), limes (1), grapefruit (12) and mandarins (2). Of these trials 6 on oranges, 2 on grapefruit, 3 on lemons and 1 on mandarins reflect current GAP. Most US trials include 4 applications within 12 months compared with current GAP of 1 or 2/year with 60-day intervals. The excessive number of applications provide information for processing considerations, but not necessarily for estimates of maximum residue levels. Most but not all trials were on only single trees. Some low-volume applications are included. Generally sample transit and laboratory storage conditions were acceptable and sampling-to-analysis intervals were generally <1 year. Several analytical methods were

described, but most were validated at near tolerance levels (e.g. 5 mg/kg). Few limits of determination were provided, although the limits of detection were generally reported as 0.02 mg/kg for the parent compound. In several cases residues of two metabolites were also determined and in some trials processing fractions were analysed. Trials were with both SC and WP formulations, although generally similar residues resulted where comparisons could be made.

Maximum residue level reflecting GAP were 0.14, 0.2, 0.3, 0.7, 0.8, 1.3, and 3.2 mg/kg in oranges, 2.4 mg/kg in mandarins, 0.5, 2.4 and 4 mg/kg in lemons (the last at 21 days compared to GAP of 7 days) and 0.7, 0.9 and 1.5 mg/kg in grapefruit. Other trials at GAP rates but with twice the number of applications resulted in residues ranging up to 14 mg/kg, although most were $\leq$10 mg/kg. Although there were few trials on lemons reflecting GAP, one of them resulted in the highest residue from GAP rates, and at a 21-day PHI compared with the 7-day GAP PHI.

Table 3 shows residues of fenbutatin oxide in citrus pulp typically to be $\leq$5% of the whole fruit residue. It also shows that residues of the metabolites SD 31723 and SD 33608 were about 2-10% and 1-5% or less of the parent compound. However, the trials in which the metabolites were determined were not in accordance with current GAP.

Table 3. Residues of fenbutatin oxide and metabolites SD 31723[1] and SD 33608[2] in citrus and citrus by-products resulting from supervised trials.

Crop, Country, state, year, variety	Application			Residues, mg/kg, at intervals(days) after last application	Ref
	Form	No.	Rate kg ai/ha (g ai/hl)		
Oranges			Day	071428-30	
USA, FLA 1972	WP	4	1.1 (16)	whole0.60.50.80.8	1
Valencia		GAP=2		Replicate0.60.80.70.6	
	WP	4	2.1 (30)	whole1.51.521.5	
				Replicate1.61.51.51.7	
	WP	4	4.2 (60)	whole3.83.23.13.6	
			2xGAP rate	Replicate44.13.23.1	
				controls <0.05 mg/kg	
Oranges				Washing and processing (30 day samples):	
USA, FLA 1972				WholeWholeFinisherDry	
Valencia				unwashedwashedJuicepulppulp	
(contd.)	WP	4	1.1 (15)	0.24<0.05<0.05<0.05 0.3	
			2.1 (30)	1.30.05<0.05<0.05 1.2	
			4.2 (60)	1.40.4<0.05<0.05 2.2	
				SD 31723 (TLC method): <0.2 mg/kg all samples.	
				Method: MMS-R-345-1 GLC and TLC - not provided.	
CA 1978	SC	1	1.7 (30)	whole full size1.314 days	2
Navel			3.4 (60)	whole full size3.9 14 days	
				controls <0.02; SD 31723 and SD 33608 <0.02 mg/kg each	
	WP	1	1.7 (30)	whole full size0.8 7 days	3
			3.4 (60)	whole full size2.5 7 days	
				controls<0.02	
				SD 33608 0.02 both applicn. rates	
				SD 31723<0.02 low rate; 0.08 high rate	
				Distribution of residues (7-day PHI, replicates)	
				Peeled Whole orangePeel	
AZ 1978	SC	4	2.2 (130)	Parent5.5, 4 0.23, 0.275.6, 4.8	4
Valencia		(GAP		SD 317230.08, 0.07 <0.02(2)0.07,0.06	
		= 2)		SD 33608<0.02,<0.02 <0.02(2)≤0.02(2)	
				Total[3]5.6, 4.1 0.29, 0.235.7,4.9	
			4.5 (270)	Parent17, 9.6 0.82, 0.2623, 9.6	
				SD 31723[1]0.31, 0.26 <0.02(2)0.51,0.31	
				SD 33608[2]0.03, <0.02 <0.02(2)0.07,0.05	
				Total[3]17, 10 0.88, 0.4224, 10.1	
				Controls: Parent ≤0.04 in all fractions	
				SD 3172 and SD 33608<0.02 in all fractions	
				7 days	
FLA 1980	SC or	4	2.2 (48)	SC WP	
Hamlin	WP			Trial 1Trial 2Trial 1Trial 2	
				Parent 8.6, 8.79.33.7, 4 3	5
				SD 31723 0.7, 0.60.8 0.3, 0.33 0.4	
				SD 33608 0.4, 0.40.40.13, 0.15 0.13	
				Total[3] 10, 10114.4 , 4.7 4	
				controls <0.02 mg/kg in all compounds	
1980	SC or	4	2.2 (120)	SC WP	
Navel	WP	(GAP=2)		Rep 1Rep 2Rep 1Rep 2	
				Parent 1010711	6
				SD 31723 0.50.60.30.6	
USA, CA 1980				SD 33608 0.20.30.20.2	

Crop, Country, state, year, variety	Application			Residues, mg/kg, at intervals(days) after last application	Ref
	Form	No.	Rate kg ai/ha (g ai/hl)		
Navel contd.				Total[3] 1111812	
				controls <0.02 in all compounds	
				7 days	
				ParentSD 31723SD 33608Controls	
FLA 1979	SC	4	2.2 (70)	7, 5.40.7, 0.50.2, 0.2<0.02 all cpds.	
Hamlin			4.4 (140)	12, 141.1, 1.20.3, 0.4<0.02 all cpds.	7
				Parent SD 31723 SD 33608 (control)(control)(control)	
FLA 1987	SC	4	2.2 (80)	unwashed fruit3.3 0.19 <0.05	8
Hamlin		GAP		(0.21)(<0.05)(<0.05)	
		= 2		washed fruit2.10.1<0.05	
				(0.12)(<0.05)(<0.05)	
				oil emulsion0.05<0.05<0.05	
				(<0.05)(<0.05)(<0.05)	
				juice0.06<0.05<0.05	
				(<0.05)(<0.05)(<0.05)	
				chopped peel/4.30.190.1	
				pulp(0.1)(<0.05)(<0.05)	
				dried peel/160.70.2	
				pulp(0.4)(<0.05)(<0.05)	
				press liquor1.60.06<0.05	
				(0.07)(<0.05)(<0.05)	
				molasses0.6<0.05<0.05	
				(0.11)(<0.05)(<0.05)	
				peel frits8.80.60.2	
				(0.14)(<0.05)(<0.05)	
				orange oil231.2<0.05	
				(0.9)(<0.5)(<0.5)	
				finisher pulp<0.05<0.05<0.05	
				(<0.05)(<0.05)(<0.05)	
			Day	0714	
Australia 1982	SC	3	(20)	whole4.43.03.2	9
Navel				peel13 99	
				pulp<0.05<0.05<0.05	
	SC	3	(40)	whole7.44.84	
				peel211412	
				pulp0.10.050.1	
				controls<0.05 whole	
			Day	0 2 7 14	
Australia 1981	SC	1	(10)	whole0.47 0.210.230.14	10
Valencia				pulp<0.02<0.02<0.02<0.02	
				juice<0.02<0.02 <0.02<0.02	
	SC		(20)	whole1 0.80.60.7	
			[GAP]	pulp<0.02<0.02 <0.02 <0.02	
				juice<0.02<0.02 <0.02 <0.02	
				controls<0.02<0.02 <0.02<0.02	
				14 days	
				whole pulp	
Brazil 1985				Adjusted	
Natal				to GAP rate	
	SC	2	(45) [1.5XGAP]	0.40.27<0.03	11
			(60)	0.30.15<0.03	
			(90)	0.4<0.03	

Crop, Country, state, year, variety	Application			Residues, mg/kg, at intervals(days) after last application	Ref
	Form	No.	Rate kg ai/ha (g ai/hl)		
			(120)	0.9<0.03	
				controls<0.3 whole	
				115 days (GAP=21 days)	
				WholePulp (peeled orange)	
Spain 1973	WP	1	0.6	<0.05	12
Clementino			1.2	<0.05	
				controls<0.05 whole	
				156 days	13
Navel	WP	1	(15)	0.16<0.05	
			(30)	0.1<0.05	
			[GAP=25-50]	controls <0.1 whole	
Mandarins					
Australia 1982	SC	3	(20)	Day0 714	
Hickson			GAP=25	whole3.42.42.1	9
				peel149.58	
				pulp0.10.10.05	
	SC	3	(40)	whole6.23.82.6	
				peel251611	
				pulp0.250.10.15	
				controls0.05	
Lemons				Days314461	
Italy 1974	WP	1	(30)	whole0.90.60.5	14, 15
Nostrana				pulp0.1<0.02<0.02	
				juice0.08<0.02<0.02	
				controls <0.02<0.02<0.02 all fractions	
				Metabolite SD 31723 <0.1 mg/kg in all fractions	
				Days071421	
USA, CA 1981	WP	4	2.2 (20)	Parent3.4	16
Lisbon		GAP		SD 336080.05	
				SD 317230.2	
		=2		Total[3]3.7	
Eureka		1	1.7 (33)	<0.02	17
Eureka			1.7 (33)	whole4	18
				peeled fruit0.14	
				peel5.8	
				controls0.05 whole	
Eureka		1	1.7 (33)	whole2.12.41.51.3	19
				peeled fruit0.140.230.030.08	
				peel3.66.31.53	
				controls0.05 parent	
				7 days (the 2 results are replicates)	
				whole pulp peel	
USA, CA 1981	SC	4	2.2 (130)	Parent 11, 9.30.42, 0.3411, 10	20
Lisbon		GAP		SD 317230.3, 0.3<0.020.4, 0.3	
		=2		SD 336080.06, 0.07<0.020.08,0.06	
				controls<0.02 all compounds whole	
Lisbon	SC	4	4.5 (270)	Parent16, 321.2, 1.426, 28	
				SD 317230.6, 10.03, 0.041.3, 1	
				SD 336080.14, 0.17<0.020.3, 0.3	
				Controls	
AZ --	SC	4	4.5 (480)	Parent5.4, 3.80.03, 0.07	21
				SD 317230.3, 0.2<0.02	
				SD 336080.1, 0.08<0.02	

Crop, Country, state, year, variety	Application			Residues, mg/kg, at intervals(days) after last application	Ref
	Form	No.	Rate kg ai/ha (g ai/hl)		
CA Lisbon	SC	4	2.2 (20)	Parent4.9	16
				SD 317230.3	
				SD 336080.08	
Limes				Controls	
USA, FLA 1981	SC	4	4.5 (190)	whole25, 260.04	22
				SD 317230.9, 0.8<0.02	
				SD 336080.3, 0.3<0.02	
				7 days	
Grapefruit				ParentSD 31723SD 33608	
USA, TX 1979	WP	4	2.2 (96)	whole1.30.10.03	23
Red Blush		GAP=2		controls0.30.03<0.02	
FLA 1980	WP	4	2.2 (48)	whole2.5, 2.30.2, 0.20.1, 0.08	24
Marsh seedless				controls0.02<0.02 <o.o2	
TX 1977	WP	4	8.4 (60)	whole20.130.08	25
Ruby Red		4	16.8(120)	3.20.30.13	
				controls<0.02 <0.02<0.02	
				Days 0 7 142845	
CA 1972	SC	2	0.21 (15)	whole0.8,0.5, 0.9,0.4,0.4, 0.70.5 0.70.40.4	26
Red Blush			[GAP=(15)-(30)]		
				pulp0.14,0.02, 0.07,0.04,<0.02 0.10.05 0.04<0.02--	
				peel1.5,0.7, 1.6,0.8,0.5, 1.31.1 1.30.50.5	
	SC	2	0.4(30)	whole1.6,1, 1.3,0.9,0.9, 1.70.8 1.50.70.7	
				pulp0.23,0.09, 0.12,0.08,0.1, 0.20.1 0.20.060.02	
				peel2.5,1.7, 3,2,2.1, 3.3,2.3 2.51.61.4	
	SC		2	whole3.2,3.1, 2.5,1.9,1.7, 2.92.7 2.61.52.5	
			0.84 (60)	pulp0.5,0.2, 0.3,0.3,0.1, 0.40.09 0.30.20.1	
				peel5,6, 4,4,4, 44 534	
				SD 31723 <0.2 mg/kg each matrix and interval	
				7 days	
				(2 results are replicates)	
				wholepulppeel	
AZ 1979	SC	4	2.2 (130)	Parent10, 2.61.6, 0.19 21, 4.8	27
Ruby Marsh				SD 137230.2, 0.08≤0.020.2, 0.5	
				SD 33608≤0.02≤0.020.05,0.03	
		4	4.5 (260)	Parent6.6, 141.4, 1.721, 27	
				317230.2, 0.30.02, 0.050.5, 0.6	
				33608<0.02,0.03<0.020.04,0.07	
				controls<0.02 all compounds whole	
TX 1979	SC	4	2.2 (96)	Parent1.4	23
Red Blush				SD 137230.1	
				SD 336080.03	
		4	4.5 (192)	Parent3.7	
				SD 137230.3	

Crop, Country, state, year, variety	Application			Residues, mg/kg, at intervals(days) after last application	Ref
	Form	No.	Rate kg ai/ha (g ai/hl)		
				SD 336080.1	
				controls0.3 Parent, 0.03 SD 13723, <0.02 SD 33608	
FLA 1980				Parent5.2, 4.5	
Marsh seedless	SC	4	2.2 (48)	SD 137230.4, 0.4	24
				SD 336080.3, 0.2	
				controls<0.02 all compounds	

Unless otherwise indicated, residues are parent compound only.
[1] SD 31723 = dihydroxybis(2-methyl-2-phenylpropyl)stannane
[2] SD 33608 = 2-methyl-2-phenylpropylstannoic acid
[3] Total organotin residues of fenbutatin oxide and its two metabolites calculated as fenbutatin oxide.

Grapes. Studies were available from 5 countries representing over 60 supervised trials, two thirds of which were from the United States (Table 4). Several varieties of grape and WP and SC formulations were covered and many of the US studies also included analyses for the two major metabolites SD 31723 and SD 33608. Several studies included analyses of various processed fractions and one was a simulated processing study. Generally plot sizes and transit/storage conditions gave credence to the data. Analysis was typically <7 months after sampling. Various analytical procedures were used and described, but not provided (e.g. MMS-R-494-2, MMS-R-345-1, SAMS-345-1, MMS-391-1), with recoveries generally >70%. Limits of detection for fenbutatin oxide were <0.02 mg/kg in grapes, when reported. Control values were generally $\leq$0.05 mg/kg in grapes and often <0.02 mg/kg, depending on the study, but sometimes only recorded as 0.1 mg/kg. Residues have not been corrected for recoveries or control values.

Maximum residues in grapes from the French trials (Table 4 references 1-4) were approximately 1 mg/kg after 28 days or 55 days; the French PHI is 7 days for the WP formulation used. Twenty one to 28 days are common PHIs in other countries which generally have comparable GAP rates, at least on an ai/ha basis. In one trial (Table 4 reference 2) no residues (<0.02 mg/kg) were found in wine from grapes treated in accordance with GAP. Maximum residues reflecting German GAP from the German results were 0.5 mg/kg after 35 days compared to a 28-day PHI. Most of the German results were from application rates higher than GAP or from more than the recommended 2 applications. Residues were up to 1.9 mg/kg after 21 days at rates similar to the GAP rates of other European countries. Maximum residues from the Italian trials were 1.1 mg/kg under the GAP conditions of other European countries, although the PHI of 21 days is shorter than the Italian GAP PHI.

Maximum residues reflecting US GAP were 4.1 mg/kg, a level found in two different locations on two varieties in different years (Table 4 references 18 and 24). Maximum residues of SD 31723 and SD 33608 from uses according to GAP were 0.2 and 0.04 mg/kg respectively. Generally SD 31723 residues were $\leq$6% of fenbutatin oxide residues and in individual samples SD 33608 residues were about half those of SD 31723, as observed for citrus.

Table 4. Residues of fenbutatin oxide and metabolites SD 31723[1] and SD 33608[2] in grapes and grape processed products from supervised trials.

Country, Year Variety	Application			Residues, mg/kg, at intervals after last application	Ref
	Form	No	Rate kg ai/ha (g ai/hl)		
			Days	28*4855 136	1
France 1974	WP	1	0.5 (50)	0.3 * French WP GAP is 7 days	
Lavallet				0.5	
Grenache			0.5 (125)	1.1Controls <0.02	
			[GAP=(50)]		
1974 Grenache	WP	1	0.5 (50) or	wine<0.02 (whole grape values not given)	2
			(125)		
1972 Semillon	WP	1	(30)	0.7	3
			(50)	0.9	
				Controls<0.1	
1974 Grenache	WP	1	0.5	0.5	4
Merlot Rouge				0.13	
Merlot Rouge				<0.05	
				Controls <0.05	
Germany			Days	0 4 7 10 14 21 28 [GAP PHI]	
1979 Riesling	WP	2	0.4 (25)[GAP]	0.10.10.080.070.08	5
4 Trials				0.50.30.30.30.3	
				0.40.50.20.20.2	
				0.30.30.30.20.1	
				Controls <0.01	
1980 Riesling	WP	4	0.3 (20)	2.62.22.51.91.2	6
		4	0.3 (75)	2.21.42.41.41.8	
1980 Müller-Thurgau		5	0.3 (20)	1.60.90.40.40.4	
		5	0.3 (75)	0.50.50.50.50.4	
1980 Riesling		4	0.3 (20)	0.40.30.140.30.1	
		4	0.3 (75)	0.20.10.30.20.3	
				Controls <0.01	
			Days	014212835	
1987 Müller-Thurgau	SC	2	0.45 (56)	0.60.60.70.50.14	7
Portugieser			0.45 (50)	0.50.40.40.20.2	
Müller-Thurgau				0.80.20.70.2	
Spätburgunder			0.45 (25)	0.70.40.60.40.5	
Riesling			0.45 (50)	0.90.70.60.60.4	
				2.521.91.51.3	
				In each study filtered and unfiltered juice and	

Country, Year Variety	Application			Residues, mg/kg, at intervals after last application	Ref
	Form	No	Rate kg ai/ha (g ai/hl)		
				bottled and unbottled wine contained <0.01 mg/kg from 35-day grapes	
Italy 1974			Days	142131	
Barbera	WP	1	(30)	1.31.10.5	8
Nebbiolo	WP	1	(30)	1.81.10.7	
				Controls <0.02	
Switzerland 1974			Days	59127	
Pinot Noir	WP	1	(100)	<0.02	9
Riesling				<0.02	
Chasselas				2.1	
				Controls <0.1 mg/kg	
				Day 12 (GAP=28)	
USA				GrapeMaturing wine	
1974 Mission	WP	2	1.1	2.8<0.02	10/11
		2	2.2	2.90.02Controls <0.02 mg/kg both	
			Days	07142852	
1973 Rabier	WP	3	0.5 (30)	0.90.70.20.2	12
		3	1 (60)	20.632.3	
				07142852	
1973 Mission	WP	3	0.5 (30)	10.510.7	12
			1 (60)	2.20.50.71.2	
			0.5 (30)	1.51.51.81 raisin 4.2	
Thompson			1 (60)	3.91.81.40.7 raisin 3.2	
				Day 14	
				ParentSD 31723SD 33608	
Valdepeñas	SC	3	1.4 (75)	2.1, 3.1, 2.8, 0.06,0.07, <0.02 (5)	13
				4.5, 4.5 reps.0.09, 0.15 reps.	
	WP	3	1.4 (75)	7, 6, 5, 6, 50.2, 0.1, 0.2,≤0.04 (5)	
				replicates0.2 replicates	
				Controls 0.05	
	SC	3	1.4 (75)	0.5, 1.3,<0.02<0.02	14
				2 (re-analysis)0.12, 0.06<0.02	
	WP	3	1.4 (75)	0.4, 0.7≤0.03 (2)<0.02 (2)	
				Controls 0.13, 0.31 (contamination suspected, not	
				subtracted from treated sample values).	
				Day0142128	
Concord	WP	3	1.1 (60)	3.122.21.8	15/16
				Pomace5.2	

Country, Year Variety	Application			Residues, mg/kg, at intervals after last application	Ref
	Form	No	Rate kg ai/ha (g ai/hl)		
				dry pomace11 (55% moisture)	
				Wine<0.02	
		3	1.7 (90)	5.62.83.42.7	
				Pomace8.9	
				dry pomace15 (55% moisture)	
				Wine<0.02	
				Controls ≤0.04 mg/kg grape, 0.07 mg/kg pomace	
				Day14	
1975 Niagara	WP	1	0.56 (60)	1.2	17
			1.12(120)	1.6	
			2.2 (240)	1.3	
				Controls 0.02 mg/kg	
				Day30	
				wholeraisinwine dry pomace	
1976 Thompson	WP	2	1.1 (60)	4 15<0.02 45, 65 (10% water)	18
			2.2 (120)	13 51<0.02 240, 190	
				Controls: grape 0.04, raisin 0.12, wine <0.02,	
				dry pomace 0.6 mg/kg.	
				Day 28	
				ParentSD 31723 SD 33608	
1980 Thompson	SC	2	1.4 (50)	1, 1.1,0.08, 0.08, 0.02, 0.03	
				1.4, 2.10.08, 0.1 <0.02(2)reps	
	WP	2	1.4 (50)	1.4, 1.6,0.06, 0.06, <0.02 (2)	19
				1.9, 2.20.15, 0.16 0.04(2)	
				Controls: ≤0.06, <0.02<0.02	
1980 Thompson	WP	2	1.4 (50)	grapes2.3, 1.70.1, 0.09, 0.04, <0.02	20
				2.2, 1.80.03, 0.02	
				raisins2.70.1 <0.02	
	SC	2	1.4 (50)	grapes0.8, 0.6,0.03, 0.02, <0.02(2)	
				1, 0.90.02, <0.02	
				raisins1.70.04 <0.02	
				Controls: <0.02 ≤0.03<0.02	
				Day 28	
				ParentSD 31723SD 33608	
1980 Thompson	WP	2	1.4 (50)	grapes 1.5, 1.60.09, 0.060.04, 0.03	21
				raisins 3.2, 3.30.14, 0.150.03	
	SC	2	1.4 (50)	grapes 1.6, 0.70.05, 0.04<0.02(2)	

Country, Year Variety	Application			Residues, mg/kg, at intervals after last application	Ref
	Form	No	Rate kg ai/ha (g ai/hl)		
				raisins 2.8 2.90.1, 0.10.02	
1980 Carignan	WP	2	1.4 (50)	grapes 0.6, 0.5, 0.3,<0.02(4)<0.02(3)	22[3]
				0.2, 0.3	
				Controls <0.02 mg/kg, all compounds in all substrates	
	SC	2	1.4 (50)	grape 0.4, 0.3, 0.4≤0.02 (2)<0.02 (2)	
				Controls <0.02 mg/kg all compounds in all substrates	
				Day 14	
1978 Thompson	WP	3	1.1 (60)	grapes 0.7<0.02<0.02	23
			2.2 (120)	60.20.06	
			1.1 (60)	dry pomace*50.10.05	
			2.2 (120)	391.40.4	
				* lab dried to 10% moisture	
			1.1 (60)	juice<0.02<0.02<0.02	
			2.2 (120)	0.05<0.02<0.02	
			1.1 (60)	wine<0.02<0.02<0.02	
			2.2 (120)	<0.02,0.02<0.02	
			1.1 (60)	raisins40.120.08	
			2.2 (120	70.160.06	
			1.1 (60)	raisin waste80.30.2	
			2.2 (120)	140.50.3	
				Controls <0.02 all compounds in all substrates	
				Day 33	
				parentSD 31723SD 33608	
1981 Concord	SC	2	1.4 (75)	4.1, 4.10.09, 0.080.04, 0.03	24
			(1.3X)	DayUnrinsedRinsedRemoved from grapes[4]grapesgrapes by rinse	
1977 Emperor	WP	1	0.7 (30)	01.9,1.2,0.74,	25
				2,1.3,0.7,	
				2.41.70.66	
				13 1.6,1.3,0.33,	
				1.2,0.98,0.18,	
				0.90.720.16	
				Controls <0.02 mg/kg	
			Days	03	
1974 Concord	WP	2	2.2 (60)	9.14.6 Control <0.02	26
				Day 14	
				ParentSD 31723SD 33608	

Country, Year Variety	Application			Residues, mg/kg, at intervals after last application	Ref
	Form	No	Rate kg ai/ha (g ai/hl)		
1980 Thompson	WP	3	1.4 (150)	4.9, 50.2 (2)0.06, 0.04	27
				3.8, 4.20.2, 0.10.04, 0.05	
				2.5, 2.20.08, 0.05<0.02 (2)	
				15, 14*0.6, 0.4*0.2, 0.08*	
				Controls≤0.13<0.02<0.02	
				*Apparent outliers	
1980 Thompson	WP	3	1.4 (50)	4, 3.7<0.1, 0.08<0.02 (2)	28
	SC			5.3, 4<0.2, 0.10.04 (2)	
				Controls<0.02 <0.02<0.02	
				Day 14	
				ParentSD 31723SD 33608	
1979 Aurora	WP	3	1.4 (740)	duplicate analyses19, 181.2, 1.40.57, 0.46	29
				replicate sample130.60.2	
	SC	3	1.4 (740)	duplicate analysis7.2, 6.80.27, 0.390.09 (2)	
				replicate sample4.70.20.08	
				Controls <0.02 all compounds	
				Parent SD 31723 SD 33608	
1979 Thompson	WP	3	1.4 (75)	grapes21, 12,0.3, 0.2, 0.06, <0.02,	30
			(replicates)	(23,12.2)*(0.4, 0.2)*,0.04 (2)	
				130.2	
				controls≤0.09<0.02<0.02	
f				raisins(27, 13)*(0.9, 0.5)*(0.18,0.08)*	
				controls<0.02 <0.02<0.02	
1979 Thompson	SC	3	1.4 (75)	grapes14, 7.9,0.2, 0.2,<0.02 (2),	
			(replicates)	(14, 17)*0.1, (0.2,0.03, (0.03,	
				0.3)* 0.04)*	
				raisins(39, 46)*(1.4, 2.6)*(0.2, 0.6)*	
				controls≤0.04<0.02<0.02	
				* duplicate analyses	
1976 Concord	SC	3	1.1(60)	3.8, 5.2	31
			2.2(120)	14, 17	
				Day 7 Grape processing	
				Concentration	
				Residue factor	
1989 Thompson	WP	2	1.4 (180)	grapes, unwashed0.54	32

Country, Year Variety	Application			Residues, mg/kg, at intervals after last application	Ref
	Form	No	Rate kg ai/ha (g ai/hl)		
				grapes, de-stemmed,	
				washed0.330.6	
				stems11.9	
				wet pomace2.34.3	
				dry pomace9.818	
				juice, unclarified0.070.13	
				juice, clarified, canned<0.05<0.09	
				sediment0.10.19	
				raisins2.44.4	
				stem waste1018.5	
				SD 31723 0.13 mg/kg in dry pomace, 0.2 mg/kg in stem waste, <0.05 mg/kg in other processed fractions. SD 33608 ≤0.2 mg/kg in stem waste, <0.05 mg/kg in other processed fractions.	

Unless otherwise indicated residues are parent compound only in grapes.
[1] SD 31723 = dihydroxybis(2-methyl-2-phenylpropyl)stannane.
[2] SD 33608 = 2-methyl-2-phenylpropylstannoic acid.
[3] Reference 22 also included analyses of juice and pomace and one additional set of data for the SC formulation, none of which were legible in the submission.
[4] Calculated by adding two right-hand columns. "Removed from grapes by rinse" assumed to be on a grape basis.

Tree Nuts. There are no current Codex MRLs for fenbutatin oxide in nuts. Twenty four studies representing 43 supervised trials in various years on several varieties of almonds, pecans, filberts and walnuts were available from the major nut-growing areas of the United States. Both WP and SC formulations and both dilute and low-volume sprays were included (Table 5). Residues of fenbutatin oxide in the nut-meats reflecting GAP application rates, PHIs and number of applications included residues of ≤0.02, 0.04, 0.05, 0.13, 0.16, and 0.3 mg/kg (the last at a 1.5-fold application rate). More results were provided at the GAP application rates and PHI, but with 3 applications instead of the approved 2 per season. These included residues of ≤0.02 (10 results), 0.03 (4), 0.04 (2), 0.05, 0.07, 0.08, 0.1, 0.2 (3) and 0.3 mg/kg. Other results were provided from exaggerated rates or at PHIs which do not closely reflect GAP. Apparent residues in untreated samples were <0.02 mg/kg except in one trial with apparent residues up to 0.04 mg/kg.

The shells and hulls of nuts were also analyzed, showing maximum fenbutatin oxide residues reflecting GAP of 8.3 mg/kg in the shells and 56 mg/kg in the hulls of almonds, 1 and 12 mg/kg respectively in walnuts and 1.9 mg/kg in pecan shells, although 3 applications were made to pecans instead of 2. Residues in pecan and walnut shells were generally some 20-30 times the levels in the meat, although in one study only about 3 to 4 times. In almonds, hull residues were generally between 60 and several hundred times the level in the meat.

In many cases samples of nut-meats, shells and hulls were also analyzed for metabolites SD 31723 and SD 33608. Results are summarized in Table 5. In all cases the metabolite residues were <0.02 mg/kg in nut-meats. Maximum residues reflecting GAP of SD 31723 and SD 33608 in almonds were 1.3 and 0.3 mg/kg respectively in the shells and 7.1 and 1.3 mg/kg in the hulls. Generally residues in the hulls were ≥3 times those in the shells.

Table 5. Residues of Fenbutatin oxide and its metabolites SD 31723[1] and SD

33608[2] in tree nuts resulting from supervised trials[3,4] in the USA.

Crop Year, Variety	Application			Residues, mg/kg, at intervals after last application	Ref
	Form	No.	Rate kg ai/ha (g ai/hl)		
				Day 14	
Pecans				ParentSD 31723SD 33608Controls (parent)	1
1981 Success	SC	3	1.4 (50)	meat<0.02<0.02<0.02<0.02	
	WP	3	1.4 (50)	<0.02<0.02<0.02<0.02	
1981 Stuarts	SC	3	1.4 (150)	meat<0.02(2)<0.02<0.02<0.02	2
				shell0.5,0.4,<0.02<0.02<0.02	
				0.3(2)	
	WP	3	1.4 (150)	meat0.03,0.04<0.02<0.02<0.02	
				shell0.6(2),<0.02<0.02<0.02	
				0.5,0.4	
1979 various	SC	3	0.7 (150)	meat0.02,0.04<0.02<0.02≤0.02	3
				shell0.08,0.09<0.02<0.02≤0.02	
			1.4 (300)	meat0.05,0.08<0.02<0.02≤0.02	
				shell0.2,0.3≤0.02<0.02≤0.02	
	WP	3	0.7 (150)	meat0.2, 0.3<0.02<0.02<0.02	
				shell1.9, 1.5 0.06(2)0.03,0.02 <0.02	
				14 35 42 70 76	
1974 Schley	WP	1	0.4 (15)	meat <0.02<0.02	4
		1	0.7 (30)	meat <0.02	
1974 Native	WP	1	1.4 (30)	meat + shell 0.03 0.02 <0.02	5
				meat<0.02	
1974 Money maker	WP	3	1.4 (30) or	meat≤0.02(4)<0.02	6
			2.8 (30)		
				Day 14	

Crop Year, Variety	Application			Residues, mg/kg, at intervals after last application	Ref
	Form	No.	Rate kg ai/ha (g ai/hl)		
				ParentSD 31723SD 33608Control	
Walnuts				(parent)	
1980 Payne	WP	3	1.4 (60)	meat<0.02(2)<0.02<0.02<0.02	7
				shells 0.3,0.5≤0.02<0.020.05,0.13	
	SC	3	1.4 (60)	meat<0.02,0.03<0.02<0.02same	
				shells 0.5,0.4<0.02,0.05<0.02,0.03same	
1979 Payne	SC	3	1.4 (75)	meat<0.02,0.04<0.02,0.03<0.02<0.02	9
		3	2.8 (150)	0.06(2)<0.02<0.02<0.02	
1981 Hartley	SC	3	1.4 (75)	meat<0.02 <0.02<0.02<0.02	10
1981 Franquette	SC	3	1.4 (424)	meat0.02,0.03<0.02<0.02<0.02	11
				(green)	
1977 Franquette	WP	2	1.4 (30)	meat 0.04<0.02<0.02<0.02	12
				shells 10.05<0.02<0.02	
				hulls 120.30.130.03	
				(nuts 1/2 size)	
				meat <0.02 <0.02<0.02<0.02	
				shells 0.5<0.02<0.02<0.02	
		2	2.8 (60)	hulls 5.10.20.070.03	
1977 Franquette				meat 0.05<0.0l2 <0.012<0.012	13
				shell 1.50.06<0.02<0.02	
	WP	2	1.4 (30)	hulls 5.80.20.09<0.02	
				(nuts 1/2 size)	
		2	2.8 (60)	meat 0.05<0.02 <0.02<0.02	
				shell 0.90.03<0.02<0.02	
				hulls 60.120.05 0.02	
Filberts				ParentSD 31723SD 33608Controls	
1979 Barcelona	WP	3	1.4 (70)	meat<0.02<0.02<0.02<0.02	8
	SC	3	1.4 (70) or	<0.02 <0.02<0.02<0.02	
			2.8 (140)		
1974 Barcelona	WP	2	0.4 (15) 0.8 (30) 1.7 (60)	meat0.02 0.02 0.02 0.02 0.02 0.02	14
Almonds					
1980 Nonpareil	WP	3	1.4 (150)	meat0.2(2)<0.02 <0.02<0.02	15
				hulls 12(2)0.4(2)0.08(2)<0.02	
	SC	3	1.4 (150)	meat0.1,0.07<0.02 <0.02<0.02	
				hulls 5,60.1,0.1<0.02<0.02	
1981 Nonpareil	SC	3	1.4 (250)	meat<0.02 <0.02 <0.02<0.02	16
				hulls 60.80.3<0.02	
1981 Carmel	SC	3	1.4 (300)	meat≤0.02 ≤0.02≤0.02≤0.02	17
				hulls 9,80.7,0.80.3,0.30.2,0.3	
1981 Mission	SC	3	1.4 (300)	meat0.03(2)<0.02<0.02 <0.02	18
				hulls 20,360.7,1.60.2,0.54,6	
			Day	0714 28	
				(Parent compound)	

Crop Year, Variety	Application			Residues, mg/kg, at intervals after last application	Ref
	Form	No.	Rate kg ai/ha (g ai/hl)		
1973 Mission	WP	2	1.7 (45)	meat 0.90.70.3 0.5	19
1973 Mission	WP	4	0.6 (30)	nut in shell 3.32.11.6 1.3	20
			1.1 (60)	nut in shell 1.91.92.4 1.6	
				Controls <0.02	
				Day 14	
				ParentSD 31723SD 33608Controls (parent)	
1977 Nonpareil	WP	2	1.3 (30)	meat 0.13<0.02 <0.02<0.02	21
				shell 130.30.10.06	
				hulls 562.70.80.3	
		2	2.5 (60)	meat 0.6<0.02 <0.02<0.02	
				shell 361.30.30.06	
				hulls 1707.11.30.3	
1977 Nonpareil	WP	2	1.3 (30)	meat 0.16<0.02 <0.02<0.02	22
				shell 8.30.30.20.06	
				hulls 241.10.30.4	
	WP	2	2.5 (60)	meat 0.12<0.02 <0.02<0.02	
				shell 3.50.10.070.06	
				hulls 391.70.60.4	
			Day	0 514	
				(Parent compound)Control	
1976 Merced	WP	1	1.4 (60)	meat 0.050.050.07<0.03,04	23
				shell1.50.70.10.03,0.1	
				hulls1412210.3,0.4	
		1	2.8 (120)	meat0.20.070.05as above	
				shell6.62.84.4	
				hulls 283357	
				Day 14	
				ParentSD 31723SD 33608Controls (parent)	
1978 Nonpareil	WP	2	1.4 (60)	meat 0.2<0.02<0.02<0.02	24
				shells 40 1.90.3<0.02	
				hulls 29 1.50.3<0.02	
	WP	2	2.8 (120)	meat 0.2<0.02<0.02<0.02	
				shells 45 2.20.4<0.02	
				hulls 36 1.70.3<0.02	

[1] SD 31723 = dihydroxybis(2-methyl-2-phenylpropyl)stannane
[2] 33608 = 2-methyl-2-phenylpropylstannoic acid
[3] Unless otherwise indicated, residues are parent compound only in nut-meats without shell.
[4] For evaluation purposes, spray volumes of ≥935l/ha (≥100 gal/acre) were treated as dilute sprays for comparing with GAP information.

Pome fruit (Table 6)

Apples. Fifty one studies were conducted in 10 countries, representing 103 supervised trials, about half of them in the United States. Both WP and SC formulations and about 20 varieties of apple were included. Plot sizes ranged from 1 to as many as 10 trees, although in many trials the number was not stated. The interval from sampling to laboratory receipt, extraction or analysis was generally ≤9 months and samples were generally

shipped and stored satisfactorily. The analytical methods were SAMS 215-1, MMS-R-494-2 and MMS-R-345-1, although the methods were not provided to the Meeting. Limits of detection ranged from 0.01 to 0.05 mg/kg and recoveries were typically 80-110% at 0.1 to 5 mg/kg fortification levels. Controls were generally <0.05 mg/kg but occasionally as high as about 0.1 mg/kg, and one was recorded as 0.5 mg/kg.

Maximum residues approximately reflecting GAP were 2.9 mg/kg in Australian trials (after 6 days compared to a 2-day GAP PHI) and ≤1.6 mg/kg in other non-US trials. All but four of the US apple trials were with dilute sprays, with maximum residues of 4.3 mg/kg from GAP applications. Three of the four trials with concentrated sprays reflected GAP, a WP application resulting in maximum residues of 9.6 mg/kg and an SC 12 mg/kg.

Pears. Twelve studies including 17 supervised trials were conducted in 5 countries, again about half of them in the United States. Maximum residues approximately reflecting GAP were 2.3 mg/kg in Australian trials and 2.7 mg/kg in South African trials (referred to Australian GAP). Conditions in other non-US trials were not according to available GAP. Maximum residues in the US trials from dilute sprays were 2.3 mg/kg and from concentrated sprays 5.6 mg/kg for WP and 3 mg/kg for SC formulations. Pulp residues were generally about 10 to 30% of those in the whole fruit.

Some pome fruit samples (mostly USA trials) were also analyzed for the metabolites SD 31723 and SD 33608. Maximum residues resulting from GAP were 0.3 and 0.05 mg/kg respectively (in pears from concentrated spray applications). Generally residues were ≤0.1 mg/kg SD13723 and <0.02 mg/kg SD 33608, both being <10% of the fenbutatin oxide residue in whole fruit and SD 31723 the higher.

Table 6. Residues of fenbutatin oxide and its metabolites sd 31723[1] and sd 33608[2] in pome fruit and processed products of pome fruit resulting from supervised trials[3]

Crop/ Country (State), Year, Variety	Application	Residues, mg/kg, at intervals after last application	Ref

	Form	No.	Rate, kg ai/ha (g ai/hl)		
			Days	1(GAP=2)61321 28	
Apples				Whole fruit (Pulp)	
Australia 1979	SC	5	(10)	1.6 1.11.10.8 0.05	1
			(GAP=20)	(0.05) (0.06)(0.07)(0.07)(0.06)	
		3	(20)	2.7 2.92.61.91.1	
				(0.2) (0.2)(0.2)(0.1)(0.1)	
				Controls <0.02 (<0.01)	
			Day	127	
1974 Josephine	WP	1	(19)	0.90.60.8	2
				(0.1)(0.1)(<0.1)	
				Controls <0.1 (whole)	
			Day	3 9 19 29	
1975 Granny Smith	WP	2	(20)	1.3 1.52.21.6	3
	SC	2	(20)	1.4 1.41.10.9	
		2	(30)	1.8 22.12	
				Controls <0.1	
			Day	56(GAP=28) 70	
Belgium 1973	WP	1	(25)	0.6	4
G. Delicious		2	(25)	0.8	
		1	(30)	1.3	
		2	(30)	1.2	
				Controls 0.12	
				Day14	
Brazil 1986	SC	2	(60)	2.1 (<0.01)	5
Fuji			(120)	2.5 (<0.01)	
			[1.1 or 2.1 g ai/tree]	Controls <0.01	
			Day 0 3 7 1435 70		
				Whole fruit (pulp)	
Canada 1975	WP	1	(13)	0.2,	6
Cortland				0.5	
				(0.02,	
G. Delicious				0.1)	
		1	(25)	1 0.9 0.7 0.5	
				(0.3) (0.2)(0.2)(0.1)	
		1	(13)	0.2	
				(0.01)	
				Controls <0.01	
			Day	7 14 3241 5972	
				Whole fruit (pulp)	

Crop/ Country (State), Year, Variety	Application			Residues, mg/kg, at intervals after last application	Ref
	Form	No.	Rate, kg ai/ha (g ai/hl)		
France 1974	WP	1	0.3 (30)	0.3 0.04	7
Cardinal				(0.02) (<0.01)	
			0.5 (50)	0.4 0.09	
				(0.03) (0.01)	
G. Delicious	WP	1	0.6 (75)	0.08	8
				(<0.02)	
Melrose		1	0.9 (50)	0.07	
				(<0.02)	
G. Delicious			0.6 (75)	0.2	
				(<0.02)	
				Controls <0.02	
				Day 41	
1977	WP	1	0.3 (30)	0.02 (<0.02), 0.6 (<0.02)*, 0.12 (<0.02)*	9
G. Delicious				* separate trials using different anti-dry-off oils	
				Controls <0.02	
				SD 31723 <0.2 mg/kg in Whole fruit and pulp.	
				Day 7 14 21 117	
				Whole fruit (pulp)	
France 1982	EC	1	0.45 (45)	0.2(<0.01)0.3(<0.01)0.1(<0.01)	10
G. Delicious					
			0.45 (90)	1.5(0.02)0.6(0.02)	
				Controls <0.01	
1972	WP	1	(30)	<0.1	11
			(50)	0.1	
				Controls <0.1	
1973			Day	55/56 65102	
G. Delicious	WP	2	0.5	0.4	12
Cardinal		1		0.08	
Cardinal		2		0.4	
Richard		1		0.3	
			Day	0 7 101421	
Germany 1977	WP	3	0.25 (**25**)	1.7 1.2 1.1 1	13
G. Delicious				1.3 1.1 1 0.5	
	SC	3	0.25 (25)	2.6 1.5 0.7 0.5	
				2.3 1.5 1.3 1	
				Controls <0.01	
				(pulp <0.01 in all samples)	
				SD 31723 <0.1 in all whole fruit and pulp samples	
1989	SC	3	0.75 (50)	0.4 0.40 0.30.25	14
Melrose				puree0.22	
				juice0.27	
				0.5 0.20 0.140.3	15
1989 Jonathan	SC	3	0.75(100)	0.7 0.50 0.5	16

Crop/ Country (State), Year, Variety	Application			Residues, mg/kg, at intervals after last application	Ref
	Form	No.	Rate, kg ai/ha (g ai/hl)		
				0 7 101421	
1989 G.Delicious	SC	3	0.5 (50)	1.5 1.10.90.60.4	17
1989 Melrose	SC	3	0.75 (100)	0.3 0.30.30.30.23	18
				puree0.23	
	SC		3	juice0.28	
			Day	0 7 10 1421 28	
Germany				Whole fruit (pulp)	
1989 Jonagold	SC	3	0.75 (100)	0.4 0.4 0.3 0.30.2	19
1989 Melrose	SC	3	0.38 (25)	0.4 0.4 0.4 0.30.25	20
				mashed apple0.22	
				juice 0.27	
Jonagold				0.5 0.2 0.2 0.140.3	
Jonathan				0.7 0.5 -- 0.50.5	
G. Delicious			0.5 (50)	1.4 1.1 0.9 0.60.4	
Melrose			0.38 (50)	0.3 0.3 0.3 0.30.23	
				mashed apple 0.23	
				juice 0.28	
Jonagold			0.38 (50)	0.4 0.4 0.30.30.2	
				Controls 0.01	
1978 G. Delicious	SC	3	0.025[4]	1.8 1.3 1.81.2 1	21
				(<0.01)(0.02)(0.03)(0.01) (0.01)	
				Control 0.15 (parent in Whole fruit apple)	
				SD 31723 ≤0.1 in all Whole fruit and pulp samples	
1980 Jamba	WP	5	0.3 (20)	0.2 0.1 0.090.01	22
				(pulp <0.01 all intervals)	
Melrose	WP	5	0.3 (20)	1.4 10.90.7	
				(0.3) (0.1) (0.1) (0.07)	
G. Delicious	WP	5	0.3 (20)	1.6 1.7 1.61.1	
				(0.2) (0.07) (0.05) (<0.01)	
				Controls <0.01	
1973	WP	4	1	1.9 1.1 1.10.7 1.1	23
Gold Parmene		3	2X	0.8 0.8 0.40.3 0.3	
				Controls <0.05	
			Day	0 237 14	
South Africa				Whole fruit (Pulp)	
1975 Granny Smith	WP	1	(30)	1.8 1.311	24
				(0.14) (0.1) (0.1) (0.07)	
			(50)	2.5 2.41.51.2	
				(0.4) (0.2) (0.2) (0.1)	
				Controls <0.01	
1974	WP	1	(100)	2 2.42.9 (4 days)	25
Granny Smith				Controls <0.05	

Switzerland			Day	10 19 26/27 35 61	
1973 Jonathan	WP	3	0.5	0.6 0.4 0.50.4 0.3	26
UK 1979				Controls 0.1	
Worcester	SC	2	(12.5)	0.15	
			(25)	0.33	
Bramley			(12.5)	0.2	27
			(25)	0.16	
				SD 33723 <0.02. Controls <0.02	
				Days 626598	
1973 James Grieve	WP	2	(25)	Controls 0.081.30.25	28
1975 G. Delicious		1	0.36(30)	<0.1	29
				Controls <0.1	
				SD 31723 <0.2	
			Days	0 63	
USA 1973	WP	4	3.4 (45)[5]	1.6 Controls <0.08	30
G. Delicious			1.5X	0.7 washed[6]	
1972 York Imperial	WP	1	0.6 (15)	0.6, 0.7, 1	31
			1.1 (30)	1.5, 1.3, 1.7Controls <0.05	
				SD 31723 <0.1	
			Day	0 71428/30 45	
1972 G. Delicious	WP	4	0.84 (15)	1.6 1.51.5 1.3	32
			1.7 (30)	3.5 2.53.1 3.2	
			3.4 (60)	7.9 4.75 3.3 4.1	
				Controls <0.05	
				SD 31723 <0.1	
1972	WP	4	0.84 (15)	2.3,2.6 2.2,2.22.1,1.51.3, 0.9,	33
Ben Davis				1.6 0.9	
			1.7 (30)	5.6,5.4 3.5,4.22.8,3.63, 2.6,	
				2.5 2.6	
			3.4 (60)	8.5,8 5.6,65.6,6.23.8, 5.4,	
				3.8 5.6	
				Controls <0.05	
				SD 31723 <0.1	
1972	WP	4	0.84 (15)	sauce*<0.05	34
G. Delicious				peel+core 4.3	
			1.7 (30)	sauce<0.05	
				peel+core 6.9	
			3.4(60)	sauce<0.05	
				peel+core16	
				Controls <0.05	
				SD 31723 <0.1 sauce, <0.2 peel+core	
				* washed, peeled apples pre-cooked, passed through finisher. Whole fruit apple residue not stated.	
			Day	0 56	
Apples cont'd				juice wet pom. dry pom. juice wet pom. dry pom.	
USA (MD) 1972	WP	1	0.56 (15)	<0.02 2 11	
Winesap			1.1 (30)	<0.02 4 18	

(VA) 1972	WP	2	0.56 (15)	<0.02 3.7 14	
Winesap			1.1 (30)	<0.02 6.7 23	
				All samples washed before processing, whole apple residues not stated.	
				Wet pomaces ca. 74% moisture, dry pomace ca. 1.5%	
				Controls ≤0.08	
				SD 31723 ≤0.2 all samples	
(NY) 1972	WP	4	0.84 (15)	<0.02 3 10.4	36
Ben Davis			1.7 (30)	<0.02 5.7 22	
			3.4 (60)	<0.02 10 40	
				Controls <0.02 juice, <0.05 wet pomace, 0.14 dry pomace	
				Whole apple residues not stated.	
				Wet pomace ca. 71% moisture, dry pomace ca. 1.7%	
				SD 31723 <0.1 juice or wet pomace, <0.2 dry pomace	
			Day	0 7 14 21 45	
(WA) 1971	WP	4	0.84 (15)	0.9 0.8 0.8 0.7 0.7	37
G. Delicious			1.7 (30)	1.7 1.4 1.5 1	
			3.4 (60)	3.7 3.5 3.6 2.8	
				Controls <0.05	
				SD 31723 <0.1 all samples	
				14 days	
				juice* wet pomace dry pomace	38
(NY) 1972	WP	4	0.84 (15)	0.4 3.7 11, 13	
Ben Davis			1.7 (30)	0.1 7.5 21, 24	
			3.4 (60)	0.09 12 45, 39	
				* Sample mislabelling suspected.	
				Controls <0.05 all matrices	
				Wet pomace ca. 69% moisture, dry pomace ca. 3.2%	
				Whole apple residues not stated.	
				SD 31723 <0.1 juice, <0.2 pomaces	
				30 days	
				juice* wet pomace dry pomace	
USA (NY) 1972	WP	4	0.84 (15)	0.17 3, 3.5 8.2, 7.8, 6.3	39
Ben Davis			1.7 (30)	0.08 4.2, 5.7 13, 11, 12	
			3.4 (60)	0.05 9.2, 10 25, 25, 23, 23	
				* mislabelling suspected	
				Controls 0.2 juice, <0.05 pomaces	
				Whole apple residues not stated.	
				Wet pomace ca. 69% moisture, dry pomace 2.3%	
				SD 31723 <0.1 juice, <0.2 pomaces	
			Day	54/56 65 98	
(PA) 1972	WP	1	0.56 (15)	0.2, 0.2	40
G. Delicious			1.1 (30)	0.5, 0.3	
				Controls <0.05, SD 31 723 <0.1	
(MD) 1972			0.56 (15)	0.5, 0.3, 0.2	41
R. Del.			1.1 (30)	0.7, 0.6, 0.8	
				Controls <0.05SD 31723 <0.1	
(VT) 1978	WP	1	0.45 (16)	0.05	42

R. Del.				Control<0.02	
				SD 31723<0.02	
(VA) 1972	WP	2	0.56 (15)	0.7, 0.9, 0.9,	43
Winesap				1, 0.8 Controls <0.05	
			1.1 (30)	1.5, 1.8, 1.7, SD 31723 <0.1	
				1.7, 1.7	
			Day	0 714	
(CA) 1973	WP	3	1.4 (30)	2.7 3.94.3	44
Gravenstein			2.8 (60)	4 3.92.2	
				Controls <0.05	
				14 days	
Apples cont'd				Whole fruit JuiceW.pomaceD.pomace	
USA 1973	WP	5	1.4 (300)	0.9, 1.3 <0.1(2)2.3, 1.59, 9.2,	45
York Imperial		(GAP		3.1, 3.39.5, 8.3	
		=4)	2.8 (600)	2.6, 2.2 <0.1(2)5.2, 5.613, 16	
				6.4, 617, 16	
				Controls <0.2 dry pomace, <0.1 other matrices	
				moisture: wet pomace ca. 74%, dry pomace ca. 1.8%	
				Day 14 48 98	
(Me) 1978	WP	2	0.8 (15)	0.3 Controls:	46
Red Del.	SC	2	0.8 (15)	0.3 all compounds <0.02	
(CA) 1979	WP	1	1.7 (45)	0.5	47
Red/G. Delicious	SC	1	1.7 (45)	0.5 Controls parent <0.05 SD 317243 & SD 33608 <0.02	
(CA) 1980 Red delicious	WP	3	1.7 (180}	9.6, 8.3	48
Newton Pippins	SC	3	1.7 (180)	10, 12	
		(GAP	935l/ha	Controls: all compounds <0.02	
1978		=4)			
Red Delicious	SC	1	0.45 (16)	0.03	49
				Controls, SD 31723 and SD 33608 <0.02	
			Days	0 7 14 28 43 52	
USA (NY) 1973	WP	4	(15)	1.8 1 1.1 1.4 juice <0.1 0.6	50
Red Rome				wet pomace 2.6,3.5	
				dry pomace 7.8,6.6,	
				11	
			(30)	2.2 1.4 1.6 1.2 juice <0.1 1	
				wet pomace 3.6,3.3	
				dry pomace 12,13	
			(60)	2.3 2.8 3.2 2.7 juice <0.1 2.2	
			[76 l/tree]	wet pomace 7.3, 6.2	
				dry pomace 20,15	
				Controls, <0.1 apple and juice, 0.25 dry pomace, 0.12 wet pomace.	
				Day 7 (GAP=14)	
				parent* SD 31723* SD 33608*	
				residue (concn. factor)**	
(NY) 1988	SC	4	(85)	apples, unwashed 3 (1)0.080.05	51
Red Rome			2.8X	apples, washed 2.5 (0.83)0.060.05	
				wet pomace 5 (1.7) 0.1 0.1	

				dry pomace 18 (6) 0.31 0.36	
				juice, unclarified 1.9 (0.6) 0.05<0.05	
				slices 0.07 (0.02) <0.05<0.05	
				* Each value the average of duplicate samples	
				** Factors relative to unwashed apples	
				Controls <0.05 all compounds in all samles	
Pears				Day 1 2 6/7 14 27/28	
Australia 1979	SC	5	(10)	Whole fruit 1 0.6 0.5	52
Beurre Bosc				pulp 0.06 0.04 0.02	
				Controls <0.01	
1974	WP	2	(19)	Whole 0.5 <0.1 0.4 0.30.5	53
Josephine				pulp <0.1<0.1	
				Controls <0.1	
				Day 3 9 19 29	
1975	SC	2	(20)	1.5 1.61.51.3	54
Packman			(20)	1.4 1.21.31.2	
			(30)	2.3 1.41.61.2	
				Controls <0.5 (confirmed by GC, different column)	
				113 or 125 days	
Belgium 1974 Durondeau	WP	1	0.75 (50)	Whole fruit and pulp, two trials<0.02	55
				0 371421 35	
France 1975	WP	1	0.3 (60)	Whole 0.20.10.03	56
Williams				pulp <0.02 <0.02<0.02	
			0.5 (100)	Whole 1.70.2 0.1	
				pulp 0.05 <0.02<0.05	
				Controls <0.02	
S. Africa 1975	WP	1	(30)	Whole 1.7 1.61.40.9	57
Packman's Triumph			15l/tree	pulp 0.2 0.10.10.05	
			(50)	Whole 3.6 3.12.71.6	
			15l/tree	pulp 0.3 0.30.20.2	
				Controls ≤0.07 Whole fruit	
			Days	0 2 5	
S. Africa 1974	WP	1	(10)	3.8 2.7 2.5	58
Wintermelis				Controls 0.11	
				14 days	
				Parent SD 31723SD 33608	
USA (WA) 1980	SC	4	1.7	2.1,2.10.1 (2)0.04 (2)	59
Bartlett	WP		(30-45)	2.3,2.20.1 (2)0.03,0.04	
(CA) 1980	WP	3	1.7 (180)	5.1, 2.90.2,0.10.05,0.03	60
Bartlett	SC	3		5.6,5.50.4,0.30.08,0.06	
				Controls: parent 0.04, metabolites <0.02	
(MI) 1980	WP	4	1.7 (700)	4.5,4.50.3,0.30.08,0.1	61
Bartlett	SC			2.5,3 0.2 (2)0.06,0.05	
				Controls: parent ≤0.04, metabolites <0.02	
				7 days	
(CA) 1979	SC	1	1.7 (45)	ParentSD 31723SD 33608	62
Bartlett				10 0.20.05	

				Controls: parent 0.05, metabolites 0.02	
			Day	0 7 1430	
(MI) 1971	WP	1	0.42 (15)	3 1.71.61	63
Kieffer			0.84 (30)	6 511.1	
			1.7 (60)	4.743	
				Controls: parent <0.05, SD 31723 <0.2	

[1] SD 31723 = dihydroxybis(2-methyl-2-phenylpropyl)stannane
[2] SD 33608 = 2-methyl-2-phenylpropylstannoic acid
[3] Unless otherwise indicated, residues are parent compound only in whole fruit.
[4] Residue levels suggest a rate error, but reported as 0.025 kg "am"/ha in several places in the report.
[5] For evaluation purposes, spray volumes of ≥935l/ha (≥100 gal/acre) were treated as dilute sprays for comparing with GAP information.
[6] 5 min. water soak, soap solution rinse, 30 sec. brush, fresh water rinse

Raspberries. Supervised trials information was available from only one country (Germany, Table 7, reference 28). Residues ranged from 19 mg/kg on the day of application to 0.8 mg/kg after 21 days at the spray concentration approved in The Netherlands, but no German GAP was provided for raspberries and the sampling intervals could not be related to the pre-bloom applications in the GAP of The Netherlands or Poland.

Strawberries. Twenty seven reports were available, representing 48 supervised trials in 7 countries: Australia (5), France (5), Mexico (2), The Netherlands (1), South Africa (2), the UK (2) and the USA (31) (Table 7). Plots ranged typically from 9 to 60 metre rows. Both SC and WP formulations were used on several varieties of strawberry. Most sprays were dilute, with a few concentrated sprays at GAP rates. Sampling-to-analysis intervals were as high as a year, but generally less than 9 months. Samples were generally shipped and stored satisfactorily. Several analytical methods were used (SAMS-215-1, MMS-R-391-1, MMS-R-345-1, MMS-R-494-2), although none of them were provided. Limits of detection were generally reported as 0.02 mg/kg, a few as 0.01 mg/kg. Control values were mostly ≤0.02 mg/kg, but one (Australia, 1982) was as high as 0.25 mg/kg.

The PHI in the USA and Australia is 1 day compared with 5 days in France and 7 days in the UK. In The Netherlands and Germany fenbutatin oxide is used pre-bloom and post-harvest. The results of the trials in The Netherlands and South Africa could not readily be related to available GAP information.

The highest residues from uses approximating GAP were 1.3 mg/kg in the Australian trials, 0.4 mg/kg in the French trials, 0.5 mg/kg in the UK trials and 7 mg/kg in the Mexican trials (referred to US GAP). The more numerous US trials resulted in a fairly evenly spaced range of residues, except two values, up to 9.9 mg/kg (this from a 1.2-fold application rate). The two exceptions were residues of 12 and 18 mg/kg from two trials at one site (Table 7, reference 27). The report mentioned difficulties in getting a reliable project history from the co-operator in these trials. For this reason and because the results were inconsistent with those of many other similar trials the author of the report doubted their validity.

Some samples were also analyzed for the metabolites SD 31723 and SD 33608 (expressed as parent), mostly in the US trials. Maximum residues from treatments according to GAP were 0.1 and 0.05 mg/kg respectively. Residues of SD 31723 were generally ≤5% of the fenbutatin oxide residues and SD 33608 was usually half of SD 31723 or less. Most analyses were after one day.

Table 7. Residues of Fenbutatin oxide and its metabolites SD 31723[1] and SD 33608[2] in strawberries and raspberries resulting from supervised trials.[3]

Crop, Country (State), Year Variety	Application	Residues, mg/kg, at intervals after last application	Ref.

	Form	No.	Rate kg ai/ha (g ai/hl)		
Strawberries			Day	0123579	
Australia 1982	SC	3	(20)	0.40.3	1
Early sweet		3	(40)	0.60.6	
				Control 0.25	
Australia 1981	SC	1	(10)	0.40.20.1	4
Redlands,			(20)	0.90.40.3	
Early sweet			(40)	1.30.50.4	
				Controls <0.01	
France 1976	WP	1	0.3 (30)	0.40.2 at 14 days	5
Gorela				SD 31723 <0.1	
				Control <0.01	
				Day 035712/1314	
1986	SC	1	0.55 (73)	0.040.02	6
Belle rubis				0.50.1	
				0.70.06	
Bogotta	SC	1	0.55(110)	0.060.03	
Gorella			0.5 (50)	0.80.140.12	
Gariguette			0.5 (50)	0.40.080.05	
				Controls <0.01	
			Days	0 1 23/4 5714	
Mexico 1976	SC	1	0.55	2.3[4] 220.9[4]	7
Fresa			1.1	7 4.1[4]1.9[4]5.9	

Crop, Country (State), Year Variety	Application			Residues, mg/kg, at intervals after last application	Ref.
	Form	No.	Rate kg ai/ha (g ai/hl)		
			(US GAP)	Controls <0.1[4]	
Netherlands 1980	WP	2	0.25 (25)	0.7,1.5,1.1,	8
				2.1,1.6,1.5,	
				2.2,1.0,1.1,	
				1 (reps.)1.2 (reps)1 (reps)	
				Controls <0.05	
S. Africa 1976	WP	1	0.13 (25)	0.50.20.2	9
Parfaite			0.19 (25)	0.80.50.5 Control <0.1	
UK 1974			Day	23/471481/84	10
Favourite	WP	1	(25)	<0.1	
Red Gauntlet				0.70.70.5<0.1	
Favourite			(30)	0.40.20.50.2	
				Day 1 3 7	
USA (WI) 1975	WP	2	1.1 (120)	1.60.5	2
Catskil			2.2 (240)	2.21.5	
			[930l/ha]	Controls <0.02	
(WA) 1975	WP	1	0.56 (60)	0.71.11	3
North West			1.1 (120)	1.11.20.8	
				2.44.33.1	
				Controls <0.02	
				Day 1 3 7	
				parent=A SD 31723=B SD 33608=C	
				ABCAA	
USA (CA) 1983	SC	4	1.7 (72)	9.90.10.05	11
			(GAP=60)	50.080.02 (reps)	
				6.50.10.03	
				5.20.080.02	
				Controls <0.02 (parent)	
(CA) 1984	SC	4	1.7 (72)	3.10.030.02	12
Heidi				2.4<0.010.04(reps)	
				Controls parent <0.01	
USA (NJ) 1975	WP	2	0.56 (60)	8.60.90.6	13
Raritan			1.1 (120)	1.41.30.6	
			2.2 (240)	51.9	
			[930l/ha]	Controls (parent) 0.07	
(FL) 1981	WP	6	1.1 (60)	1.70.04<0.02	14
Tuffs		GAP		1.60.04<0.02 (replicates)	
		= 4		30.10.05	
	SC	6	1.1 (60)	2.30.090.03	
				2.70.070.03 (replicates)	
				2.70.10.06	
USA cont.				1.90.10.07 Controls ≤0.02 (parent)	
(CA) 1976	WP	1	0.56 (30)	2.3	15
Heidi			1.1 (60)	3.6Control <0.02 (parent)	

Crop, Country (State), Year Variety	Application			Residues, mg/kg, at intervals after last application	Ref.
	Form	No.	Rate kg ai/ha (g ai/hl)		
(FL) 1979	WP	10	1.1 (60)	8.90.20.1	16
Tioga				7.50.20.09 (replicates)	
		10	2.2 (120)	190.50.3	
				180.40.2 (replicates)	
(KY) 1976 Tenn Beauty	WP	2	0.56 (60)	2.9 Control <0.02 (parent)	17
(CA) 1979 Shasta	SC	10	1.1 (67)	4.60.30.1Control <0.02 (parent)	18
(FL) 1979	SC	10	1.1 (60)	6.80.20.1	19
Tioga				7.70.30.1Control <0.02 (parent)	
(CA) 1981	SC	6	1.1 (200)	0.90.03<0.02	20
Shasta			[560l/ha]	1.20.04<0.02 (replicates)Control <0.02 (parent)	
				Note: 1 yr. between sampling and analysis, 57% recovery of parent. (Not stated whether results corrected for recovery).	
USA (CA) 1984	SC	4	1.7 (72)	4.40.040.02	21
G-4		=GAP	[GAP=(60)]	4.70.050.04 (replicates)	
				Controls 0.01 all compounds	
(CA) 1981	SC	6	1.1 (48)	1.70.04<0.02Note: 11.5 months between sampling and analysis	22
Heidi				2.80.060.02 (replicates)	
				Controls <0.02 all compounds	
			Day	0137 (parent) (parent)	
(CA) 1973	WP	4	4.5 (190)	4.243.33	23
G-4				Control 0.02	
				Note: 1.3 yr. between sampling and analysis	
				1 day	
				ParentSD 31723SD 33608	
(CA) 1979	WP	10	1.1 (67)	1.60.10.04	24
Shasta			2.2 (130)	2.70.20.07	
				Controls 0.03 parent, 0.02 metabolites	
(KY) 1974	WP	3	0.56 (60)	2.4	25
Tennessee Beauty			1.1 (120)	5Control <0.02	
			[930l/ha]		
				Day0 1	
(CA) 1974	WP	1	1.1 (60)	2.31.4	26
Toiga			2.2 (120)	3.82.4	
				Control 0.02	
				Note: 11 months between sampling and analysis	
(MI) 1974	WP	3	0.56 (60)	12^5	27
Everberring			1.1 (120)	18^5 Control 0.31	
			[930l/ha]	Note: 9 months between sampling and analysis	
Raspberries				Day 0714 21	
Germany1980	WP	2	0.38 (25)	198.310.8	28
Schoenemann				Control <0.01	

[1] SD 31723 = dihydroxybis(2-methyl-2-phenylpropyl)stannane
[2] SD 33608 = 2-methyl-2-phenylpropylstannoic acid
[3] Unless otherwise indicated, residues are parent compound only in the whole fruit.
[4] Samples arrived at laboratory cold but unfrozen, and consisted of fruit and some separated juice.
[5] Doubtful result. See text.

Stone fruit (Table 8)

Forty seven studies on stone fruit were available from 8 countries representing 76 supervised trials on cherries, plums, peaches and nectarines. In some samples of peaches the whole fruit and pulp were analysed separately, and plums and peaches were analysed before and after drying. In some cases the metabolites SD 31723 and SD 33608 were determined. Most of the results were for de-stoned fruit, but in a few cases it was not clear whether the stones had been removed. No attempt was made to calculate the result on a whole-fruit basis since the stone weights were only about 6% of the whole fruit weights.

The analytical methods were SAMS-215-1, MMS-R-391-1, and MMS-R-494-1 or -2. Limits of detection were generally reported as 0.02 mg/kg. Analytical recoveries were generally 80%, although in some studies as low as 55-65% with no indication of whether the results were corrected for analytical recoveries. In general plot sizes ranged from 1 tree (2-3 replicates) to 5 trees and samples were stored satisfactorily.

Cherries. The highest individual residue from trials approximately reflecting GAP was 0.6 mg/kg in Germany (6 trials) and 5.1 mg/kg in the United States (14 trials) (it was not stated whether results were adjusted for 69% recoveries). The 2 trials in The Netherlands were not according to GAP. Analytical recoveries ranged from 58 to 110% and controls were $\leq$0.3 mg/kg, except in the trials in The Netherlands where they were up to 0.6 mg/kg. Maximum residues of SD 31723 and 33608 were 0.9 mg/kg and 0.04 mg/kg respectively from treatments according to GAP. The ratios of the residues of fenbutatin oxide to those of the metabolites were similar to those found in other commodities.

Peaches. The higheset residues from treatments close to GAP in Australia (4 trials) was 2.5 mg/kg. The highest in the 9 United States trials was 8 mg/kg from a 1.5-fold rate (corresponding to 5.3 mg/kg at the GAP rate) and 5.8 mg/kg from 3 instead of the permitted 2 applications at approximately the GAP rate, and after a PHI of 21 instead of 14 days. The intervals from sampling to analysis were 34 to 44 months in most of the US trials (9 months in one and 18 months in another). Maximum residues from 3 Canadian trials according to GAP were 0.8 mg/kg, from 5 French trials according to German GAP 1.3 mg/kg, and from 3 trials in Germany 3.3 mg/kg from the approved spray concentration but after 28 days rather than the GAP interval of 21 days. Three trials in South Africa resulted in residues up to 7.8 mg/kg after 10 days and 6 mg/kg after 13 days (a 14-day PHI is common in other countries), although information on GAP in South Africa was not provided. Apparent residues were up to 0.2 mg/kg in untreated samples, depending on the method or trial, and in at least one case the residue was confirmed as fenbutatin oxide.

Fenbutatin oxide residues in the pulp were 5 to 10% or less of the whole fruit residues. Maximum residues of SD 31723 and SD 33608 from GAP treatments were <0.1 mg/kg. The ratios of the residues of the metabolites to those of fenbutatin oxide in whole fresh fruit were again similar to those in other commodities.

Plums. Maximum residues of fenbutatin oxide from uses approximating GAP were 0.7 mg/kg in 9 German, and 2.1 mg/kg in 12 United States trials, while residues of SD 31723 were $\leq$0.07 (or <0.1) mg/kg and of SD 33608 $\leq$0.03 mg/kg except for 1 value of 0.1 mg/kg. SD 31723 residues were usually <5% of the fenbutatin oxide residues and SD 33608 similar to or lower than SD 31723. Control values for fenbutatin oxide ranged from <0.01 to 0.1 mg/kg, depending on the analytical method used. Fenbutatin oxide residues were concentrated in prunes (dried plums).

Table 8. Residues of Fenbutatin oxide and its metabolites SD 31723[1] and SD 33608[2] in stone fruit resulting from supervised trials[3,4].

Crop, Country (State), Year Variety	Application			Residues, mg/kg, at intervals after last application	Ref
	Form	No.	Rate kg ai/ha (g ai/hl)		
Cherries (de-stoned)[4]			Day	04710142128	
Germany 1976					
Biggarau	WP	3	(25)	0.70.20.09	1
Rote Leber			[GAP]	1.50.20.040.02	
Schattenmorelle (Morello)				1.10.40.20.10.09	
				Controls <0.01-0.3 SD 31723 <0.1 all samples	
1980		3	0.3 (20)	3.41.71.5 0.9 0.6	2
Schattenmorelle				1.61.30.9 0.8 0.2	
Rubin				3.51.4 1 0.7 0.1	
				Controls <0.01	
USA (OR) 1974	WP	2	0.4 (15)	0.3	3
Sweet			0.8 (30)	0.5	
			1.7 (60)	Control 0.04 1.2	
(NY) 1974	WP	1	0.7 (18)	0.4	4
Montmorency			1.3 (36)	1.3	
			2.7 (71)	1.9	
				Control <0.02	
				14 days	
				ParentSD 31723**SD 33608**	
(OR) 1981	SC	2	1.7[6] (45)[7]	1.7,0.02,<0.02	5
Royal Ann				1.8 (repl.)*0.02 (repl.)<0.02	
				* Not stated whether corrected for recoveries 58% parent, 61% metabolite. ** Calculated as parent	
				Controls <0.02 all compounds	
(CA) 1981	SC	2	1.7[6] (71)[7]	3.5, 0.05,	6
				3 (repl.)*0.04*	
				* Not stated whether corrected for recoveries 75% parent, 73% metabolite	
				Controls <0.02 all compounds	
USA (MI) 1980					
Montmorency	SC	2	1.7 (180)	4.7, 5.1*0.2, 0.10.04, 0.03*	7,9
	WP	2	1.7 (180)	2.7, 2.2*0.07, 0.050.02*, <0.02	
			[930l/ha]	* Not stated whether corrected for recoveries 69% parent, 53% metabolite	
				Controls: parent 0.02-0.17, metabolites <0.02	
(CA) 1979	SC	2	1.7 (90)	3.80.90.04	8
Bings			3.4 (180)	100.30.05	
				Controls parent 0.08, metabolites <0.02	
(OR) 1977 Royal Ann/Bings	WP	1-2	1.1-2.2 (30-60)	<0.02 parent at 292-305 days	10, 11,
			post-harvest		12
			Days	2128 (NL GAP=42 days, Italian 30)	
Netherlands 1976	WP	1	0.3 (25)	0.9*0.5 * German GAP applicn.	13

Crop, Country (State),Year Variety	Application			Residues, mg/kg, at intervals after last application	Ref
	Form	No.	Rate kg ai/ha (g ai/hl)		
Morello			0.6 (50)	1.1*1.2 * Italian GAP applicn.	
				Controls 0.4, 0.6 (confirmed)	
Plums[4]			Days	07142128	
Germany 1977 Auerbacher	WP	3	0.25 (25) [GAP]	0.080.060.04	14
Zwetschge (Victoria)				0.40.30.30.2	
Hauspflaume				0.50.50.70.50.1	
				Controls: parent <0.01, SD 31723 <0.1	
1978 Hauspflaume	WP	3	0.25 (25)	0.50.50.70.50.1	15
				Controls: parent <0.05, S31723 D <0.1	
1980 Auerbacher	WP	5	0.3 (20)	0.20.40.30.1	16
Ortenberg				0.10.070.05<0.01	
				0.50.70.40.3	
				Controls <0.01	
1976 Auerbacher	WP	3	0.25 (25)	0.20.20.20.1	17
Buehler				0.30.10.20.1	
				Control <0.1	
Netherlands 1976	SC	1	0.18 (25)	0.30.20.1<0.1<0.1	18
Czar			0.35 (50)	0.50.40.20.1 0.1	
				Control <0.1	
			Day	04 714	
S. Africa 1975	WP	1	(30)	1.2 1.30.80.9	19
Kelsey			(50)	2.22.21.81	
				Control <0.01	
				14 days	
				ParentSD 31723SD 33608	
USA (MI) 1981	SC	2	1.1[6] (60)[7]	0.2, 0.04<0.02<0.02	20
Lumbard				Controls <0.02 all compounds	
(NY) 1980	SC	2	1.1 (80)	1, 1.5*0.05, 0.07<0.02, 0.03	21
Stanley			2.2 (160)	6.7, 3.1*0.3, 0.10.1, 0.05	
				* Not stated whether corrected for 68% recoveries. Controls <0.02 all compounds	
(MI) 1979	SC	2	1.1 (340)	1.3, 2.10.03, 0.060.02, 0.1	22
Stanley			2.2 (680)	5.9, 6.10.1, 0.090.1, 0.06	
			[330 l/ha]	Controls parent 0.06, metabolites <0.02	
				Prunes, dried	
(CA) 1981	SC	2	1.1 (60)	2.5*0.070.07	23
Prunes, dried (from treated plum trees)				*Not stated whether corrected for 67% recoveries Controls: parent 0.15, metabolites 0.02	
USA (CA) 1981					
Prunes (from treated sugar plum trees)	SC [GAP=2]	3	1.1 (120)	3.1*0.070.04	24
			[930l/ha]	* Not stated whether corrected for 55% recovery. Controls: parent 0.18, metabolites <0.02	
				24 days	
USA (CA) 1981	SC	2	1.1 (120)	0.7, 0.8<0.02<0.02	25
Santa Rosa			[930l/ha]	Controls: parent 0.06, metabolites <0.02	

Crop, Country (State),Year Variety	Application			Residues, mg/kg, at intervals after last application	Ref
	Form	No.	Rate kg ai/ha (g ai/hl)		
(NY) 1974	WP	2	0.56 (15)	0.3	26
Stanley/Italian			1.1 (30)	0.5	
			2.2 (60)	0.5 Controls <0.02	
				7 days (GAP=14)	
				unwashed plums driedrehydrated prunesprunes	
(CA) 1988 domestica	WP	2	1.1 (60)[5]	(80% moisture) (18.5% moisture)(33% moisture)	27
			[426l/ha]	0.18, 0.08* 0.37, 0.280.08, 0.22	
				(0.13)* (0.32)*(0.15)*	
				Concentration factor, fresh to dry = 2.5	
				Concentration factor, fresh to rehydrated = 1.2	
				* duplicate assays, average in parentheses	
				SD 31723, SD 33608 and Controls <0.05 all matrices	
Peaches[4]			Day	1 6 13(GAP=14) 2128	

Crop, Country (State), Year Variety	Application			Residues, mg/kg, at intervals after last application	Ref
	Form	No.	Rate kg ai/ha (g ai/hl)		
				whole (pulp)	
Australia 1979	SC	5	(10)	2.8 2.4 1.7 0.90.9	28
Golden Queen				(0.1)(0.1)(0.08)(0.08)(0.07)	
		3	(20)	5.6 2.9 2.5 1.81.2	
				(0.2)(0.1)(0.1)(0.09)(0.07)	
				Controls <0.01	
				15 daysParent SD 331723 SD 33608	
USA (CA) 1978	WP	2	1.1[6] (60)[7]	fresh*1.1** 0.08**<0.02	29
Hale Haven				controls<0.02 <0.02 <0.02	
				dried*** 8.2 0.08 0.03	
				Controls 0.09 <0.02 <0.02	
				14 days	
				fresh*1.9** 0.06**<0.02	30
Riosa Gems				Controls≤0.03 <0.02<0.02	
				dried***17 0.07 0.08	
				Controls 0.08 <0.02<0.02	
				*90% moisture **Not stated whether corrected for 62-65% recoveries ***10% moisture, lab air-dried	
			Day	0 712/14 21 28	
(OH) 1973	WP	1	1.7[8] (45)[7]	2.7 (12 days, corresponds to 1.1 at GAP rate)	31
Cumberland				Control<0.02	
(CA) 1973	WP	3	0.31 (20)	3.9 2.81.9 1.5	32
Key Stone		(GAP =2)	0.62[8] (40)[7]	7.7 8.24.6 5.8	
				Controls 0.04, 0.18	
(CA) 1973	WP	3	0.56 (30)	4.31	33
Sunblest			1.1 (60)	7.32.6	
				Controls ≤0.02	
(OR) 1973	WP	2	1.7[8] (45)[7]	5.7, 8 (corresponds to 3.1, 5.3 at GAP rate)	34
Red Haven				Controls 0.02	
(CA) 1973	WP	3	1.1[6] (60)[7]	4.33	35
Sunblest				Controls <0.02	
Peaches cont'd			Day	0 3 71421 28	
Australia 1975	SC	2	(20)	2.2	36
Tatura Aurora			(30)	3.6	
	WP	2	(20)	2.2 Controls 0.2 (confirmed)	
Canada 1975	WP	2	(25)	2.6 0.6	37
Alberta			[U.S GAP]	pulp 0.05 0.03	
				Controls <0.01	
1981	SC	2	0.63 (19)	0.3, 0.4,	38
Baby Golden				0.3, 0.2	
				pulp 0.02, 0.04 (3)	
				Controls <0.01	
1982	SC	1	0.89 (26)	0.6 (2),	39
Madison				0.5 (2),	

Crop, Country (State), Year Variety	Application			Residues, mg/kg, at intervals after last application	Ref
	Form	No.	Rate kg ai/ha (g ai/hl)		
				0.7 (3),	
				0.8 (2),	
				0.4, 1, 0.3	
				pulp<0.01 (10)	
				Controls <0.01	
France 1976	WP	1	0.75 (50)	0.9 (8 days)	40
Royal Gold				pulp<0.01	
				SD 31723<0.01	
				Controls<0.01	
				0 3 71421 28	
1973 Audenot	WP	1	0.5	3.72.6 1.9 (2 days) Control 0.2	41
1982	SC	1	0.45 (90)	1.4	42
Merril s.d.				pulp0.04Control 0.01	
1973	WP	1	0.5	1.61.6 1.51.30.7	43
Early Alberta			[German GAP]	Controls<0.05	
				Day 2 815	
France 1973	WP	1	0.5	21.70.4	44
Red Aven				Controls 0.04-0.1	
			Days	01 3714212835	
New Zealand 1975	WP	4	(19)	0.80.60.10.70.5 Control <0.1	45
Golden Queen					
Germany 1976					
Rote Ingelheim	WP	3	0.25 (25)	9 3.43.421.81.3	46
Rekord aus Alf.				8.1 4.72.52.21.5	
M. Rochiat				8.13.12.52.33.3	
				Controls <0.1	
				Days 013671013	
S. Africa 1976	WP	1	(30)	76.3 5.94	47
Kakamas				pulp0.20.10.060.02	
			(50)	128.276	
				pulp0.20.20.10.1	
				Controls 0.01	
1976 Kakamas	WP	1	(30-50) [2 trials]	canned <0.01<0.01 <0.01	48
1974	WP	1	(30)	4.23.1	49
			(50)	6.47.8	
Nectarines				14 days	
				ParentSD 31723 SD 33608	
USA (CA) 1981	SC	2	1.1(230) [480l/ha]	0.2 (2) <0.02 <0.02	50
Sunglo	SC	2	1.1[6] (60)[7]	2, 3.50.05, 0.08 <0.02	51
				Controls: parent 0.1, metabolites <0.02	

[1] SD 31723 = dihydroxybis(2-methyl-2-phenylpropyl)stannane
[2] SD 33608 = 2-methyl-2-phenylpropylstannoic acid
[3] Unless otherwise indicated, residues are parent compound only in the whole fruit.

[4] All residues refer to de-stoned fruit, except references 14, 32, and 36 in which it was not indicated whether stones were removed. The Meeting did not recalculate residues on a whole-fruit basis since the stones constituted on average only about 6% of the whole fruit weight (the range was 3.8-11%). Reference 3 was an exception where the stone weighed 30-40% of the fruit in early samples and 10-15% in later samples.
[5] In reference 27 the summary (Volume 1) cites the application rate as 0.06% (60 g ai/hl). This is consistent with the text of volume 3, page 13 (based on about 200 G/A (1892 l/ha) but not with Table V, page 24, which lists the volume as 45 G/A (170 l/A = 426 l/ha). Airblast sprayer was used.
[6] GAP rate for concentrated sprays.
[7] GAP rate for dilute sprays = (15-30).
[8] GAP rate for concentrated sprays = 0.6-1.1.

Animals

The Meeting received feeding studies on cows and chickens which had also been provided to the 1979 JMPR.

Cows. Six lactating Guernsey cows were fed daily, three at 11 and three at 96 ppm unlabelled fenbutatin oxide in the diet (equivalent to 0.37 and 3.7 mg/kg body weight) for 21 or 22 days, with handling procedures similar to those described below under "Fate of residues" (Shell, 1973). In this study (Potter and Nugent, 1978), samples of milk were taken periodically and animals slaughtered within 24 hours of the last feeding for analysis of tissues.

Analyses were for fenbutatin oxide, SD 31723 and SD 33608 by method MMS-R-494-1, described later under "Methods of residue analysis", because "a previous trial, 11-112-74" was reported to have shown residues to be mainly fenbutatin oxide and SD 31723. That report was not provided to the Meeting and appears not to have been reviewed by the 1977 or 1979 Meetings.

No residues (<0.02 mg/kg) of fenbutatin oxide or its metabolites were detected in any tissue or in cream from the 11 ppm feeding level, or in skim milk or brain from either level. Residues of fenbutatin oxide were found in tissues and cream from feeding at 96 ppm, but SD 33608 was not found in any samples and SD 31723 only in liver and kidney (Table 9). Analytical recoveries of all three compounds averaged ≥84% in skim milk and cream at 0.1 and 0.2 mg/kg fortification levels, ranged between 79 and 118% in liver and kidney at 0.2 mg/kg and were ≥81% in fat and muscle at 0.1 mg/kg.

The greatest potential for residues in cattle products would be from feeding dry apple pomace, dry citrus pulp, dry grape pomace and almond hulls. These might be fed to beef cattle at 50, 33, 30 and 25% of the dry feed matter respectively, and to dairy cattle at 25, 33, 20 and 25%. The implications are discussed in the Appraisal.

Table 9. Residues of fenbutatin oxide, SD 31723 and SD 33608 in cream and in tissues of dairy cattle from feeding fenbutatin oxide for 21 or 22 days at 96 ppm in the diet (Potter and Nugent, 1978).

Sample	Residues, mg/kg		
	Fenbutatin oxide	SD 31723	SD 33608
Cream	day 1 <0.02 day 3 0.03-0.04 day 13 0.06-0.11 day 21 0.04-0.06	<0.02	<0.02
Cream fat[1]	0.08-0.14	--	--
Liver	0.04-0.07	0.09-0.12	<0.02
Kidney	0.13-0.18	0.02-0.04	<0.02
Subcutaneous fat	0.04-0.06	<0.02	<0.02
Mesenteric fat	0.05-0.06	<0.02	<0.02
Quadriceps muscle	0.03-0.04	<0.02	<0.02

[1] Calculated from levels in cream and the proportion of milk fat in cream

Chickens. In the chicken feeding study two groups of 27 White Leghorn hens were fed for periods up to 28 days with fenbutatin oxide in the total feed at either 5 or 25 ppm. Eggs were collected every 24 hours and after intervals of 7, 14, 21 or 28 days tissue samples were taken for determination of fenbutatin oxide, SD 31723 and SD 33608 by method MMS-R-494-1. Some birds were also fed untreated feed for various periods up to 28 days after withdrawal of the treated feed before slaughter.

In the 5 ppm feeding group, no residues (<0.02 mg/kg) of fenbutatin oxide were detected in any samples except egg yolks, one sample of whole egg with 0.09 or 0.15 mg/kg (suspected of being contamination) and two samples of whole egg with 0.02 mg/kg. Neither metabolite was found in any sample. Residues of both fenbuatin oxide and the metabolites were found in liver and kidney from the 25 ppm feeding level and of the parent compound only in whole egg and egg yolk but not egg white (Table 10). No residues were found in light or dark meat or fat. Analytical recoveries were ≥85% in all matrices from 0.1 to 0.5 mg/kg fortifications.

The greatest potential for residues in chicken eggs or tissues would come from dry grape pomace, which may constitute up to 5% of poultry diets.

Table 10. Residues of fenbutatin oxide and metabolites SD 31723 and SD 33608 in eggs and tissues of chickens from feeding fenbutatin oxide at 25 ppm dietary levels for intervals up to 28 days and for periods thereafter with untreated feed (Potter and Nugent, 1979)

Sample	Residues, mg/kg		
	Fenbutatin oxide	SD 31723	SD 33608
Liver	Day 7 0.02 Day 14 0.04 Day 21 0.02 Day 28 0.02 Day 31[1] <0.02 Day 36 <0.02 Day 42 <0.02	0.05 0.12 0.1 0.07 0.08[1] 0.03 0.02	<0.02 0.02 0.04 <0.02 0.03[1] 0.02 0.02
Kidney	Day 7 0.03 Day 14 0.02 Day 21 <0.02 Day 28 <0.02 Days 31[1] <0.02 Days 36 <0.02	0.03 0.03 0.03 0.02 0.04[1] <0.02	<0.02 all intervals
Whole Egg	Days 1-3 <0.02 Days 4-7 0.05 Days 8-22 0.1 Days 23-28 0.12 Days 29-30[1] 0.1 Days 33-35 0.04 Days 36-42 <0.02	<0.02 all intervals	<0.02 all intervals
Egg yolk	Days 8-15[2] 0.17 Days 23-28[2] 0.25	<0.02 <0.02	<0.02 <0.02

[1] Untreated feed after 28 days for tissues or 29 days for whole eggs
[2] 8-15 days value from 5 ppm feeding level, 23-28 days from 25 ppm

FATE OF RESIDUES

Two cow feeding studies with the radiolabelled compound (at least one of which was previously submitted to the 1977 JMPR) were available, together with processing information for apples, citrus, grapes, plums and to some extent tree nuts, cucumbers and peaches. The processing information is summarized below, but the results are included in the supervised trials

Tables 2 to 8.

In animals

In one study three lactating Guernsey cows were fed [^{119}Sn]fenbutatin oxide at 170 ppm in grain concentrates, giving a dietary equivalent of 34 ppm, for 21 days (Shell, 1973). The 34 ppm was linked to a mean feed consumption of 16 kg/day/cow so that the cows received on average 540 mg/day of labelled fenbutatin oxide. The main weight of the cows was 425 kg, so that the dosage was approximately 1.3 mg/kg body weight/day.

Milk, urine, and faeces were taken for analysis daily. The animals were slaughtered 12 hours after the last dose and tissue samples analysed by liquid scintillation counting. Faeces accounted typically for about 65-85% of the daily dose, and urine for 0.2 to 1%, but some samples were outside these ranges. The R_f of the major TLC spot from faeces extracts corresponded to fenbutatin oxide.

No radioactivity was found in milk at a 0.02 mg/kg limit of detection, nor in brain, bone, bone marrow, mesenteric fat, subcutaneous fat or quadriceps muscle at a 0.04 mg/kg limit. Residues equivalent to 0.04 mg/kg of fenbutatin oxide were reported in gastrocnemius muscle from one of the three cows but <0.04 mg/kg in the other two. Fenbutatin oxide equivalents in the kidneys of the three cows were 0.27, 0.38 and 0.15 mg/kg and in the livers 0.4, 0.41 and 0.22 mg/kg. The residues were not identified or characterized.

In the second study (Koos, 1973) three Guernsey cows were also fed twice daily with ^{119}Sn-labelled fenbutatin oxide for 21 days, at a total dietary equivalent reported to be 30 ppm. 337 mg of the compound was applied to 1 kg of grain concentrate. The daily feed was 15 kg (5 kg concentrate plus 10 kg alfalfa cubes) feed/cow/day, although the actual feed consumption was not recorded in the report. Milk and tissue samples were analysed by analytical method MMS-R-345-2. The interval from last feeding to slaughter was not recorded in the report. The analytical method was not provided, but is based on EC-GLC and TLC procedures for the determination of the parent compound and SD 31723.

No residues of fenbutatin oxide (<0.01 mg/kg) were found in milk, brain, quadriceps, gastroenemius, subcutaneous fat, mesenteric fat or liver, nor in the kidney or heart of two of the three cows. One cow had 0.03 mg/kg fenbutatin oxide in kidney and 0.05 mg/kg in heart. Limits of determination were 0.01 mg/kg in milk and 0.02 mg/kg in tissues.

No information was submitted on the fate in plants, soil, or water/sediment systems. Information on the fate in plants had been submitted to the 1977 Meeting.

In processing

Apples. The most comprehensive processing study received was a simulated commercial process with apples treated at 85 g ai/hl (an exaggerated rate) and harvested after 7 days (compared with GAP 14 days) to ensure sufficient residues in the processed fractions (Table 6, reference 51). Apples were washed, peeled and cored. Peeled and cored apples were sliced and cooked. Peels and cores were ground to produce unclarified juice and wet pomace in the finisher step. Wet pomace was dried in a forced-air dryer for 24 hours at 77°C. Concentration factors were washed apples 0.83, unclarified juice 0.6, wet pomace 1.7, dry pomace 6 and slices 0.02. Residues of SD 31723 and SD 33608 were $\leq$0.1 mg/kg in all fractions. In a separate study water washing (including soap rinse and brushing) reduced the residue level by approximately 56% (Table 6, reference 30).

Additional data were provided from processing apples treated in accordance with GAP into juice, wet pulp and dry pulp, but with fewer details of the procedure (Table 6, references 45 and 50). Concentration factors were juice <0.1, wet pomace 2.4-3.5, and dry pomace 6.9-12. In other studies apples treated in accordance with GAP were processed into juice and wet and dry

pomace, although residue levels in the whole apples were not provided to permit the estimation of concentration factors (Table 6 references 35, 36, 38, 39). Maximum residues from treatments according to GAP were 0.17 mg/kg in juice, 7.5 mg/kg in wet pomace and 24 mg/kg in dry pomace.

Many of the studies included analyses of whole apples and pulp. Pulp residues were generally ≤20% of the whole fruit residues and in most cases <10%. Similar results were observed for pears with residues in the pulp <10% of the whole fruit residue.

Oranges. In one processing study on Valencia oranges about 70% of the field-incurred residue of fenbutatin oxide was removed by "normal" washing 30 days after treatment, although details of the washing procedure were not provided. No residues (<0.05 mg/kg) were found in the juice from whole fruit with field-incurred residues up to 1.4 mg/kg, but residues in the dry pulp were 1-1.6 times the level in unwashed whole oranges (Table 3, reference 1).

A more comprehensive processing study was conducted on Hamlin oranges to simulate commercial processing in accordance with University of Florida Circular 2-266. It consisted of standard washing procedures and "F.M.C. in-line extraction" resulting in three fractions: (1) peel, seed and rag (2) oil emulsion crude and (3) unfinished juice. Fraction 1 was chopped, limed and pressed to give a peel press liquor which was concentrated into molasses and a press residue which was dried into dry citrus pulp. Fraction 2 was further divided into orange oil and peel frit, and fraction 3 into single strength juice and finisher pulp.
Washing whole Hamlin oranges harvested after only 7 days (the GAP PHI) removed 36% of the fenbutatin oxide residue of 3.3 mg/kg from applications in excess of GAP. In this case only trace levels (0.06 mg/kg) were found in the juice. Residues were concentrated in orange oil (6.6 times), dried peel/pulp (4.6 times), peel frits (2.5 times) and in chopped peel/pulp (1.2 times). Residues in the oil emulsion, press liquor, molasses, and finisher pulp were ≤50% of those in the unwashed whole fruit (Table 3, reference 8).

The levels of the total residue of the parent and the two metabolites (calculated as parent) in orange peel and peeled oranges were found to be about 100-120% and 3-7% respectively of the level in whole oranges after 7 days (Table 3, reference 4). Another study showed residues in Navel and Hickson orange pulp to be about 1-3% of those in whole oranges and the residue level in the peel to be about 2-3 times that in the whole orange. A somewhat similar distribution was observed in mandarins, but peel residue levels were a little higher relative to levels in whole oranges (Table 3, reference 9). Other results in Table 3 reveal a similar trend in the distribution of residues in lemons and grapefruit.

Cucumbers. Residues in cucumbers were also largely in the peel with pulp residues ≤1/3 of those in the whole commodity (Table 2).

Grapes (Table 4). Rinsing removed about 30-40% of the grape residue on the day of application, but only about 15-20% of the residue remaining after 13 days (Table 4 reference 25). In a simulated commercial processing study washing and destemming grapes removed approximately 40% of the fenbutatin oxide present after a PHI of 7 days (Table 4, reference 32). No fenbutatin oxide residues (<0.02 mg/kg) were detected in wine or juice from grapes treated according to GAP. The highest residues of fenbutatin oxide from treatments reflecting GAP were 15 mg/kg in raisins and 65 mg/kg in dry pomace (Table 4, reference 18).

Grapes were treated twice with a WP formulation at 1.4 kg ai/ha (180 g ai/hl) with a 14-day interval between treatments, harvested 7 days after the second treatment and subjected to simulated commercial processing. Residues of 0.54 mg/kg were reduced to 0.3 mg/kg in de-stemmed, washed fruit. Residues became concentrated in wet pomace (4.3 times), raisins (4.3 times), dry pomace (18 times), stems (1.8 times) and stem waste, (19 times). The metabolite SD 31723 was found only in dry pomace (0.13 mg/kg) and stem waste, and SD 33608 only in stem waste (Table 4, reference 32). In other studies similar concentration factors were found for raisins (Table

4, references 18 and 30), but lower factors for dry pomace (Table 4, reference 23).

Nuts. Fenbutatin oxide residues in nut shells are typically some 10-50 times those in the meats and residues in hulls ≥3 times those in shells (Table 5).

Peaches. When peaches containing 90% moisture were air-dried in the laboratory to 10% moisture, residues were concentrated from 1.1 mg/kg to 8.2 mg/kg (7.5 times) (Table 8, reference 29) and from 1.9 to 17 mg/kg (9 times) (reference 30). If the residues in the fresh samples were not corrected for 62-65% analytical recoveries the corrected concentration factors would be about 4.8 and 5.5 respectively.

Plums. Plums containing 80% moisture were treated at 1.1 kg ai/ha (60 g ai/hl), harvested after 7 days (GAP PHI 14 days), processed by a simulated commercial procedure to prunes (18.5% moisture) and rehydrated to 33% moisture (Table 8, reference 27). The plums were washed in a spray washer for 15-30 seconds with water at 165-185°F, dried at about 150°F in a forced-circulation air dryer and rehydrated in water at 165-185°F for 3-5 minutes. Pitted plums, dried prunes and rehydrated prunes were analyzed for fenbutatin oxide by analytical method AMR-720-87 (MMS-R-494-2). Samples were also analysed for metabolites SD 31723 and 33608. Mean concentration factors were 2.5 for prunes with 18.5% moisture and 1.2 for prunes with 33% moisture.

Residues in prunes from plums treated approximately in accordance with GAP have been reported up to 3.1 mg/kg (Table 8, reference 24). It is not known whether this value was corrected for the 55% recovery: if not, the "true" residue would be 5.6 mg/kg.

Stability of pesticide residues in stored analytical samples

The 1977 monograph cited a study (TIR-26-116-74) as reporting that residues were stable for 18 months when stored at -15°C, but no details were given. No reports on the stability of residues in stored analytical samples were provided for the present review. A summary discussion on "Freezer storage stability study of fenbutatin oxide (Vendex®) and metabolites in strawberries, egg plants, and almonds" is included in several reports provided to the Meeting (e.g. Table 3, reference 8). This summary reports that residues in all target samples are stable up to 8.5 months when stored at -20°C. Intervals before analysis were generally ≤1 year for the supervised trials data provided, except in some studies on stone fruit where the interval was up to 3 years or more.

Residues in the edible portion of food commodities (see also "Fate of residues in processing", above)

Washing apples was shown to remove about 15% of the residue in whole apples, although under some conditions (soap rinsing and brushing) over 50% may be removed (see processing above) (Table 6, reference 30). Generally ≤20% of the residue in whole apples will be found in the pulp and residues in juice from treatments approximating GAP were generally ≤10% of those in the whole apple (see processing above), although a residue in unclarified juice was 60% of the whole fruit residue.

Residues in banana pulp have been shown to be 2-8% of field-incurred residues in the whole banana (Table 2, reference 6). Washing has been shown to remove 40-70% of fenbutatin residues from whole oranges and it has been demonstrated that residues in peeled oranges are less than 10% of the level in whole unpeeled oranges (Table 3, e.g. reference 4). Residues may be concentrated in orange oil (6.6 times), dried peel/pulp (4.6 times), peel frits (2.5 times) and chopped peel/pulp (1.2 times).

Washing or washing and destemming grapes can remove about 20-40% of the fenbutatin oxide residues, depending on the PHI (Table 4, references 25 and 32). No residues (<0.02 mg/kg) are expected in wine or grapes from

applicatins according to GAP, although concentrations up to about 5-fold in raisins and 18-fold in dry pomace may result from processing.

Canning whole destoned peaches (no reference was made to peeling) field-treated in a manner expected to result in residues up to about 10 mg/kg in whole peaches or 0.2 mg/kg in peeled peaches (similar trials, same time, same place, same variety, same application and similar PHIs) left no detectable residues of fenbutatin oxide (<0.01 mg/kg) or SD 31723 (<0.2 mg/kg) in the canned fruit. The destoned peaches were halved, immersed in boiling sodium hydroxide for 30 seconds, washed with water, steam de-aired for 10 minutes at 85°C for 10 minutes, sealed and heated at boiling point for 20 minutes (Table 8 reference 48).

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

No information was provided.

METHODS OF RESIDUE ANALYSIS

Although various analytical methods have been used in the numerous supervised trials only two were provided to the Meeting, one as an appendix to Potter and Nugent, 1978 (method MMS-R-494-1). The principles of this and other methods, control values and analytical recoveries are included in individual field trial reports. The other was method MMS-R-345-1 (Shell, 1972) which was among thosereviewed by the 1977 JMPR (see below). It also includes a TLC procedure for the determination of metabolite SD 31723.

Two basic approaches underly the methods used in most of the trials. The first, reviewed by the 1977 JMPR, includes methods MMS-R-345-1, MMS-R-391-1 or (-2) and WAMS 215-1 (SAMS-1). The second was described in the 1979 monograph but not specifically referenced.

In the first approach extraction is by dichloromethane, chloro-derivatization by HCL digestion, clean-up on an alumina column and determination by GLC with EC detection. For method MMS-R-345-1 the limit of detection was reported as 0.05 mg/kg for the parent compound and 0.1 mg/kg for SD 31723.

In the other procedure (method MMS-R-494-1) the macerated sample is extracted by shaking for two hours with chloroform and HCl, the mixture is allowed to stand overnight, the chloroform evaporated, and the residue taken up in hexane which is partitioned with acetonitrile containing 0.05% tropolone. The acetonitrile solution is evaporated to dryness and the residue taken up in diethyl ether. The residue of the three compounds (parent, SD 31723 and SD 33608) is methylated with methyllithium. After further partitioning and clean-up on a Florisil column determination is by GLC with an FPD (6100A Sn-selective interference filter) (Shell, 1979). From summary descriptions method MMS-4-494-2 appears to be a variation of this procedure.

Although not presented to show details for individual commodities, summary validation results for MMS-494-1 in a variety of crops and animal tissues show mean recoveries of the order of ≥90% for the parent, SD 31723 and Sd 33608, but ranging from 50 to 133% from fortifications of 0.1 to 50 mg/kg of the parent and 0.1 to 0.5 mg/kg of the metabolites. Sample chromatograms suggest that limits of determination for the parent would be 0.02-0.05 mg/kg in cream and 0.1 mg/kg in liver, for SD 31723 0.1-0.2 mg/kg in grapes, 0.1 mg/kg in liver and 0.02-0.05 mg/kg in cream, and for SD 33608 0.02-0.05 mg/kg in grapes, 0.1 mg/kg in cow liver and 0.02-0.05 mg/kg in cream.

With a few exceptions discussed under individual supervised trials, analytical recoveries by the various analytical methods in the crops studied were generally ≥80%. Recoveries were quoted over a range of fortification levels, but generally near the MRLs. Limits of detection (usually expressed as a percentage of full scale deflection, e.g. 4% FS)

were reported for most methods and were generally ≤0.02 or ≤0.05 mg/kg, but few limits of determination were given. Descriptions of analytical methods for the two primary metabolites SD 31723 and SD 33608 were also provided in individual study reports, but again were not provided for review.

A proposed HPLC method for fenbutatin oxide in fruit matrices was also provided to the Meeting (Nicolas *et al.*, undated). Laboratory-fortified samples were extracted with dichloromethane, centrifuged, and filtered through a phase separator. The solvent was evaporated and the sample dissolved in the mobile phase for analysis. An octadecyl silica column was used with methanol containing triethylamine (2-5%) to minimize tailing as the mobile phase. Detection was by UV absorption at 205 nm or by fluorescence (excitation at 410 nm, emission at 490nm) after post-column derivatization with a 254 nm mercury lamp. Analytical recoveries were 56% from apples, 77% from grapes and 48% from oranges at 0.1 mg/kg. The limit of determination was reported as 0.2 mg/kg for apples and grapes with UV detection, but analysis of oranges was not possible owing to lack of specificity. The limit of determination was 0.04 mg/kg in all three matrices with fluorometric detection.

NATIONAL MAXIMUM RESIDUE LIMITS

National maximum residue limits reported to the Meeting are summarized below
(US limits include fenbutatin oxide and its organotin metabolites).

Crop/country	MRL, mg/kg	Notes
ALMONDS		
USA	0.5	
ALMOND HULLS		
USA	80	
APPLES		
Australia	3	
Austria	1.5	
Belgium	2	
Germany	2	registration in progress
Italy	0.5	
Japan	5	
Netherlands	2	
New Zealand	1	pip fruit
S. Africa	2	
Spain	2	
Switzerland	1.5	for fruits
USA	15	
APPLE POMACE, dry		
USA	75	
APRICOTS		
Germany	1	
Japan	5	
BANANAS		
Australia	5	
Spain	1	
BEANS		
Japan	0.5	
Spain	0.5	
Netherlands	0.5	French, scarlet runner, slicing beans
CHERRIES		
Australia	3	stone fruit
Belgium	1	stone fruit
Germany	1	registration in progress
Italy	0.5	stone fruit
Japan	5	
Netherlands	5	
New Zealand	1	stone fruit
Spain	2	stone fruit
Switzerland	1.5	fruits
USA	6	
CITRUS FRUITS		
Australia	5	
Brazil	0.4	peeled
China	5	
Italy	0.5	
Japan	5	
Netherlands	5	
S. Africa	1	
Spain	2	

Crop/country	MRL, mg/kg	Notes
Taiwan	2	
USA	20	
CITRUS POMACE		
USA	35	
CITRUS PULP, dry		
USA	7	
CUCUMBERS		
Belgium	0.5	fruiting vegetables
Denmark	1	greenhouse
Italy	0.5	
Japan	2	
Netherlands	1	gherkins
Spain	1	gherkins
Belgium	1	gherkins, fruiting vegetables
Switzerland	0.2	gherkins
USA	4	
EGG PLANT		
Belgium	o.5	fruiting vegetables
Japan	2	
Netherlands	1	
Spain	0.5	solanaceae
FRUIT		
Austria	1.5	
Switzerland	1.5	
FRUITING VEGETABLES		
Netherlands	1	cucumbers, egg plant, gherkins, melons, okra, peppers, pumpkins (patisson, summer squash), vegetable spaghetti, tomatoes, water melons
GRAPES		
Austria	0.01	
Belgium	2	
France	2	
Germany	4	registration in progress
Italy	0.5	
Japan	5	
Netherlands	5	
Spain	1	
Switzerland	1.5	fruits
USA	5	
GRAPE POMACE, dry		
USA	100	
HOPS		
Australia	20	
Japan	30	
NUTS		
USA	0.5	almonds, walnuts, pecans
NECTARINES		
Australia	3	
OTHER BERRIES		
Netherlands	0.2	
OTHER SMALL FRUIT		
Netherlands	0.2	
OTHER STONE FRUIT		
Netherlands	1	
PEACHES		

Crop/country	MRL, mg/kg	Notes
Australia	3	
Austria	1.5	fruit
Belgium	1	stone fruit
Denmark	5	greenhouse
Germany	4	registration in progress
Italy	0.5	stone fruit
Japan	7	
Netherlands	1	stone fruit
New Zealand	1	stone fruit
S. Africa	2	
Spain	2	
Switzerland	1.5	fruits
USA	10	
PEARS		
Austrlia	3	
Austria	0.5	
Belgium	2	
Germany	2	registration in progress
Italy	0.5	
Japan	5	
Netherlands	2	
New Zealand	1	pip fruit
S. Africa	2	
Spain	2	
Switzerland	1.5	fruits
Taiwan	2	
USA	15	
PLUMS		
Germany	1	
Netherlands	3	
USA	4	
PRUNES		
Japan	3	
USA	4	
PRUNES, dry		
USA	20	
RASPBERRIES		
USA	10	
STONE FRUIT		
Belgium	1	
New Zealand	1	
STRAWBERRIES		
Australia	1	
Japan	3	
Netherlands	3	
USA	10	
TOMATOES		
Belgium	0.5	fruiting vegetables
Denmark	1	greenhouse
Italy	0.5	
Netherlands	1	
Spain	0.5	solanaceae
RAISINS		
USA	20	

Crop/country	MRL, mg/kg	Notes
OTHER FOOD COMMODITIES		
Netherlands	0* (0.05)	
ANIMAL PRODUCTS		
USA	0.5	cattle meat, fat or meat byproducts
Netherlands	0.2	liver
Netherlands	0.2	kidney
Netherlands	0.02*	other meat
USA	0.5	hogs, meat, fat or meat byproducts
USA	0.5	horses or sheep meat, fat or meat byproducts
MILK FAT		
USA	0.1	
Netherlands	0.02*	milks

*At or about the limit of determination

APPRAISAL

Fenbutatin oxide, a miticide registered for use on many crops world-wide, was first reviewed by the 1977 JMPR for both toxicology and residues. A toxicological re-evaluation in the periodic review programme of the CCPR was conducted in 1992, but the corresponding residue review was postponed to 1993 owing to the late arrival of data. Although the present Meeting reviewed over 250 individual reports or studies containing residue data and GAP information, little or no information was provided on some critical supporting studies (e.g. plant, goat and hen metabolism studies, processing studies for tomatoes, freezer storage stability of analytical samples, analytical methods, etc.). The Meeting received processing studies for apples, grapes and citrus and, on request, cow metabolism and transfer studies as well as a chicken feeding study. The Meeting was informed that rat, hen and goat metabolism studies had been submitted to WHO and could be submitted to FAO for future review. A proposed LC analytical method for fenbutatin was also reviewed.

GAP and summary residue data received from Spain and The Netherlands for a number of commodities were received too late for full consideration. The GAP information has been added to the 1993 Monograph. Most if not all of the summary residue information appears to have been included in earlier submissions for 1993 review and has therefore been considered.

There was still a lack of critical supporting information, with the exception of the cow and chicken feeding studies and processing studies provided. Accordingly, the Meeting limited this periodic review primarily to evaluating supervised trials data and/or evaluating data in the context of the available information on current GAP.

Supervised trials data show fenbutatin oxide residues in general to be primarily on the surface or in the peel. Residues in banana pulp are 1-2% of the level in the whole banana, peeled cucumber residues are $\leq$33% of whole cucumber residues, citrus pulp residues are <5% of the whole fruit residues. In nuts residues in the shell are typically 25 times those in the nut meat, although in a few cases only 3 to 4 times. In almonds the hull residues were of the order of 60 times the level in nut meat.

Processing information was provided on a number of commodities. Washing may remove 20 to 40% of the residues on fruits and an even higher proportion in some cases on citrus fruits. Concentration occurs in some processed fractions, with concentration factors of 1.7 in wet apple pomace, 6 in dry apple pomace, 5 in dry citrus pulp, 6.7 in citrus oil, 4.3 in wet grape pomace, 18 in dry grape pomace, 4.3 in dried grapes, 2.4 in dried prunes, and up to 9 in dried peaches. Residues from GAP applications to grapes are $\leq$0.02 mg/kg in wine or grape juice.

In a number of studies samples were also analysed for residues of the metabolites dihydroxybis(2-methyl-2-phenylpropyl)stannane (SD 31723) and 2-

methyl-2-phenylpropylstannoic acid (SD 33608). The former is with few exceptions ≤10% of the fenbutatin oxide residue and the latter usually ≤1/2 the level of the SD 31723. There is some evidence that the canning process may reduce residues near the MRL to non-detectable levels, at least in stone fruit.

Avocado. There is no MRL for avocado. Because data were available only for the flesh and no GAP was available for the countries in which trials were conducted, the Meeting concluded that information was insufficient to support a limit.

Banana. There is currently no MRL for bananas. Maximum residues reflecting GAP were 6.3 mg/kg at 7 days, 3.4 mg/kg at 2 days and 5.7 mg/kg at 0 days. The GAP PHI is 1 day. About 1-8% of the whole fruit residue has been found in the pulp (maximum level 0.14 mg/kg), although ≤2% is likely to be a more reliable estimate taking into account analytical factors. Although few of the results were exactly at the GAP PHI, residues show little decline over 7 days from application, and the Meeting concluded that data over this period were relevant to estimating a maximum residue level. Because results were available from only one country, the Meeting considered additional data reflecting the GAP of other countries desirable. However, because results were available from three locations in three different years, the Meeting concluded that they were sufficient to estimate a 10 mg/kg limit.

Beans. There is no MRL for beans. Residues in green beans from a single application in a single trial in one country were 0.5 mg/kg after 3 days. Data were available from another country at a slightly exaggerated application rate (75 g ai/hl instead of 50 g ai/hl) from a formulation which is not recognised as GAP. The plot size was only 12 m^2. The Meeting could not recommend a limit for green beans.

Residues in French beans from 2 applications at GAP rates under glasshouse conditions in one country resulted in maximum residues of 0.4 mg/kg after 6 days compared with a GAP PHI of 7 days. At twice the 25 g ai/hl GAP rate residues were 0.15 mg/kg after 7 days. The Meeting concluded that the data were insufficient to support a limit for beans.

Citrus. The CXL for citrus fruits is 5 mg/kg. Although 43 trials were conducted in 5 countries, 3/4 of these were in one country and only 12 of the trials represented current GAP (6 on oranges, 2 on grapefruit, 3 on lemons and one on mandarins). Maximum residues resulting from GAP were 1.5 mg/kg in grapefruit, 2.4 mg/kg in mandarins, 3.3 mg/kg in oranges and 4 mg/kg in lemons, the last at a 21-day PHI compared with the GAP 7-day PHI. Other trials at GAP application rates, but at twice the GAP number of applications resulted in residues up to 14 mg/kg, but generally less than 10 mg/kg. Residues of the metabolite SD 31723 were typically 2-10% of the parent compound in whole oranges and in all the fruits the residue of SD 33608 tends to be about half that of SD 31723.

While additional data reflecting GAP for oranges, grapefruit and mandarins are desirable, the Meeting concluded that the available results were marginally sufficient in a mutually supportive way to confirm the existing 5 mg/kg citrus group CXL for these individual citrus fruits. This does not apply to lemons or limes, because relatively few trials representing GAP were available for lemons. As noted, the highest GAP residue (4 mg/kg) in lemons was at a 21-day PHI compared with a GAP 7-day PHI, and the next highest residue of 2.4 mg/kg was from only single applications whereas two are permitted. The Meeting concluded that additional data reflecting current GAP for lemons and/or limes with the minimum PHI and maximum application rates would be required before a limit could be recommended for lemons or limes or for citrus fruits as a group. Additional data reflecting GAP for oranges, grapefruit and mandarins are also desirable.

The available information from the simulated commercial processing of oranges with field-incurred residues indicates that residues in dry orange pulp are 2-4.6 times those in whole unwashed oranges. Assuming a fivefold concentration and a residue at the 5 mg/kg MRL level in the unprocessed fruit, the maximum dry pulp residue would be 25 mg/kg compared with the

current 7 mg/kg limit. Although there is currently no Codex MRL for citrus oil, the 6.6 concentration factor from whole unwashed oranges indicates that an MRL of 30 mg/kg would be needed.

Cucumbers. The CXL for cucumbers is 1 mg/kg. Data were available from 4 European countries (open and glasshouse) and the USA (open), although the US data do not correspond to current GAP. While application rates in the US trials were higher and PHI intervals shorter than required by GAP in Europe, the data are useful for illustrating the dependence of residues on the application rate and giving some indication of differences in residues between 2 and 3 applications. Residues from single applications representing European GAP ranged from 0.03 to 0.3 mg/kg. The Meeting concluded that a 0.5 mg/kg limit was supported.

Egg plant. The CXL for egg plant is 1 mg/kg. Because data were available from only a single glasshouse trial in one country, because no GAP information was provided for that country and because the trials did not conform to GAP application rates or PHIs of neighbouring countries the Meeting concluded that the information was insufficient to support the limit, and recommended its withdrawal.

Gherkins. The CXL for gherkins is 1 mg/kg. Because only summary information from a single glasshouse trial was available, the Meeting concluded that it was insufficient to support the limit, and recommended its withdrawal.

Grapes. The CXL for grapes is 5 mg/kg. Data were available from over 60 supervised trials in 5 countries. Maximum residues of about 2 mg/kg resulted from European GAP and 4 mg/kg from US GAP. While US GAP is comparable to that of several European countries, many more US trials were available. The Meeting confirmed the existing 5 mg/kg limit. Currently there is no Codex limit for processed grape products. Because the concentration of residues from grapes to raisins is about fourfold, the Meeting concluded that an MRL of 20 mg/kg for raisins would be appropriate, based on 5 mg/kg in the whole fruit. Similarly, concentration of the order of 18 times in dry pomace (or stem waste) supports a limit of 100 mg/kg for dry pomace.

Hops. No MRL is proposed for fenbutatin oxide in hops. Supervised trials data reflecting GAP were available from a single trial in one country, resulting in residues of the order of 5 mg/kg. The Meeting concluded that these data were inadequate for estimating a maximum residue level.

Melons. Currently there is a 1 mg/kg CXL for melons, except watermelons. Although no residues (<0.01 mg/kg) resulted 7 or 14 days after applications at GAP rates in a single trial in one country whose GAP PHI is 3 days, the Meeting concluded that the data were insufficient to support the limit, and recommended its withdrawal.

Nuts. No MRL is established for nuts. Maximum residues of fenbutatin oxide in the nut meats of almonds, pecans, walnuts and filberts from treatments at US GAP application rates, PHIs and number of applications included residues of $\leq$0.02, 0.04, 0.05, 0.13, 0.16, and 0.3 mg/kg (the last at a 1.5 times application rate). Appreciably more data were provided which were at GAP application rates and PHI, but with 3 applications instead of the maximum 2 per season which is GAP. These included residues of $\leq$0.02 (10), 0.03 (4), 0.04 (2), 0.05, 0.07, 0.08, 0.1, 0.2 (3) and 0.3 mg/kg.

Although there were no side-by-side comparisons, the overall data suggest similar residues from 2 or 3 applications at GAP rates. The Meeting therefore concluded that a 0.5 mg/kg limit could be supported for almonds, pecans, and walnuts. Residues were observed up to 56 mg/kg in almond hulls (an animal feed item).

Data were too limited to support a limit for filberts, for which no GAP information was provided.

Data were also available for metabolites SD 31723 and SD 33608 with levels of <0.02 mg/kg in nut meats.

Peppers. The CXL for peppers (sweet) is 1 mg/kg. Data were available from two supervised trials. The single application (2 are permitted) in the Belgian trial was outdoors, although the provided Belgian GAP was for glasshouse uses. The Netherlands glasshouse trial was at 0.5 kg ai/ha, which could not be related to the GAP rate of 25 g ai/hl. Although residues in two supervised trials (1 and 0.6 mg/kg after the 3-day PHIs) suggest that residues may not exceed 1 mg/kg from GAP, the Meeting did not consider two data points reflecting GAP sufficient to support the MRL, and recommended its withdrawal.

Pome fruit. The CXLs are 5 mg/kg for apples and pears and 20 mg/kg for dry apple pomace. The data base for pome fruit included 103 supervised trials for apples and 17 for pears. In many trials residues decreased little during 2-3 weeks after application. Maximum residues in apples were 2.9 mg/kg from GAP applications in non-US trials. In US trials the highest residues from GAP applications were 4.3 mg/kg from dilute sprays, 9.6 mg/kg from concentrated SC sprays and 12 mg/kg from concentrated WP sprays, although only three of the many trials on apples were with concentrated sprays at GAP application rates. The two apple trials which resulted in the higher residues were on a different variety from those in other trials, but it could not be concluded that the variety influenced the residue. While the trial plot was a single tree, this was also true of other trials. Higher residues from concentrated spray applications are also suggested by pear trials where maximum residues reflecting US GAP were 2.3 mg/kg for dilute sprays and 5.6 and 3 mg/kg for WP and SC concentrated spray applications respectively. These dilute and concentrated spray applications were all on the same variety of pear.

While the Meeting concluded that the current 5 mg/kg Codex limit is adequate for dilute spray applications, it would not accommodate the USA concentrated spray uses. The Meeting concluded that additional data reflecting GAP would be needed to accommodate these.

The Codex limit for dry apple pomace is 20 mg/kg to accommodate the current 5 mg/kg limit on apples (fourfold concentration factor). Concentration and reduction factors in apple processed products estimated from studies provided to this Meeting varied, depending on the study: juice (unclarified) 0.6 times, wet pulp 1.7-3.5 times, and dry pulp 6-12 times. Clearly the fourfold factor previously used by the JMPR is too low in view of this information. Putting greater weight on the most comprehensive processing study provided, the Meeting concluded that for estimating maximum residue levels a factor of 7 would be reasonable for whole fruit to dry pomace. With an MRL of 5 mg/kg and a concentration factor of 7 a 40 mg/kg limit can be recommended for dry apple pomace.

Raspberries. No MRL exists. Supervised trials information was available for only one country, for which no GAP information was provided and the GAP of other countries could not be used. The Meeting concluded that insufficient information was available to estimate a maximum residue level.

Soya beans. Because residue results (<0.01 mg/kg) were available from only three supervised trials in a single country 67-80 days after application compared with the GAP 7-day PHI, and because analytical recoveries by analytical method SAMS 345-1 were highly variable at a 0.2 mg/kg fortification level (50-110%), the Meeting concluded that data reflective of GAP were insufficient to support a limit.

Stone fruit

Forty-seven studies from 8 countries were available for stone fruit, representing 76 supervised trials. Most of the results referred to de-stoned fruit. No attempt was made to calculate residue in the whole fruit including stone, since average stone weights were only about 6% of the whole fruit weight.

Cherries. The CXL is 5 mg/kg. Maximum residues reflecting approximate GAP in Germany were 0.6 mg/kg and in the USA 5.1 mg/kg (whether results were adjusted for 69% recoveries was not stated). If not corrected, a maximum

residue of 7.4 mg/kg would be indicated. Other US data also did not indicate whether corrections had been made for low recoveries. If not, other residues when adjusted for recoveries would be of the order of 7 mg/kg. Results from The Netherlands data did not reflect the national 42-day PHI. However, the residues up to 1.2 mg/kg were from applications consistent with German or Italian GAP PHIs, although German GAP was reported to be due to expire in 1993. Maximum residues of SD 31723 were 0.9 mg/kg and SD 33608 0.04 mg/kg from GAP. The former was ≤25% of the fenbutatin oxide residue and SD 33608 is usually less than half of the level of SD 31723.

The Meeting was particularly concerned at the lack of information on whether results from several US studies (the major portion of the data base) were corrected for analytical recoveries less than 70% (58% in one case), and at the information that German reregistration is to expire in 1993 (the German GAP is relevant to other European trials for which no GAP was provided). The Meeting concluded that a 10 mg/kg limit could be supported for cherries.

Peaches, nectarines. The CXL is 7 mg/kg. Maximum residues in peaches reflecting approximate GAP in Australia were 2.5 mg/kg, and in the USA 8 mg/kg from a 1.5 times rate (5.3 mg/kg adjusted to the GAP rate) and up to 5.8 mg/kg from approximate GAP rates, but with 3 instead of the permitted 2 applications. Residues were up to 3.5 mg/kg in two US trials reflecting GAP on nectarines. Maximum peach residues were 0.8 mg/kg in Canada at US GAP rates, 1.3 mg/kg in France at German GAP rates, and 3.3 mg/kg in Germany. Three trials in South Africa also resulted in residues up to 3.1 mg/kg after 10 days and 4 mg/kg after 13 days (14 day-PHIs are common in other countries) at application rates which are GAP in other countries, although GAP information for South Africa was not provided. Residues were up to 6 and 7.8 mg/kg after 13 and 10 days respectively at higher application rates. The Meeting concluded that the data supported the current 7 mg/kg limit for peaches and in a mutually supportive way could support a limit at the same level for nectarines.

Plums. The CXL is 3 mg/kg. Maximum residues approximating GAP were: German trials 0.7 mg/kg, United States trials 2.1 mg/kg, Netherlands trials <0.1 mg/kg. No GAP information was available for South Africa. Residues were 0.9 and 1 mg/kg after 14 days. From GAP applications, maximum residues of SD 31723 were 0.07 mg/kg and of SD 33608 0.04 mg/kg. SD 31723 is usually <5% of the fenbutatin oxide residue and SD 33608 is similar to or lower than SD 31723. Control values for fenbutatin oxide range from <0.01 to 0.1 mg/kg, depending on the analytical method used.

Although recoveries in some US trials were below 70% and no information was provided on whether the results were corrected, recoveries were acceptable in the trial with the highest GAP residue (2.1 mg/kg). Furthermore, assuming that the results in the trials with low recoveries are uncorrected, maximum residues would be about 2.2 mg/kg. The Meeting concluded that the data were sufficient to support the CXL for plums.

There is no MRL for prunes (dried plums). Data provided indicate that fenbutatin oxide residues are concentrated in drying plums by a factor as high as 2.5. Applying this to the 3 mg/kg limit for fresh plums would imply an MRL of 7.5 or 10 mg/kg for dried prunes.

Residue levels in dried plums from trees treated in accordance with GAP were provided, although no data were included for the fresh fruit from which a concentration factor could be estimated. Maximum residues were 3.1 mg/kg. Analytical recoveries for this study were only 55% and it was not indicated whether the result had been corrected for the low recovery. If not, a residue of 5.7 mg/kg would be indicated. This would be consistent with the theoretical 7.5 mg/kg estimated above.

Strawberries. The CXL is 3 mg/kg. Twenty-seven reports were available from 7 countries representing 47 supervised trials (32 from the USA). Data from two countries could not be related to the available GAP. Maximum residues approximating GAP were 1.3 mg/kg from Australian trials, 0.4 mg/kg from French trials, 0.5 mg/kg from UK trials, and 7 mg/kg from Mexican trials

(based on US GAP). The more numerous US trials resulted in a fairly continuous distribution of residues, except for two values, up to 9.9 mg/kg (the last from a 1.2 times application rate). The exceptions were at one site with residues of 12 and 18 mg/kg. Because information on the project history for these trials was in question and because the residues (especially 18 mg/kg) were not consistent with those found in numerous other similar trials, even at exaggerated rates, the Meeting gave little weight to these two values.

Maximum residues from GAP of the metabolites SD 31723 and SD 33608 were respectively 0.1 and 0.05 mg/kg after 1 day. Generally residues of SD 31723 were $\leq$5% of fenbutatin oxide residues and SD 33608 residues were about half or less of the SD 31723 residues (after one day). The Meeting concluded that the data supported an increase in the current 3 mg/kg CXL to 10 mg/kg.

Tomato. The CXL is 1 mg/kg. Four of the 12 supervised trials were according to GAP, and the maximum residues in these: Denmark 0.4 mg/kg (glasshouse), Italy 0.3 mg/kg (field), the UK 0.3 mg/kg (glasshouse). Although results were available from 3 additional countries, they could not be related to the GAP information provided. Residues were up to 0.8 mg/kg after 3 or 4 days in two trials that could not be confirmed to reflect GAP. No tomato processing data were provided. No residues (<0.1 mg/kg) of metabolite SD 31723 were found in the two trials in which it was determined.

The Meeting concluded that the data were adequate to confirm the current limit for tomatoes, but only for glasshouse uses.

Animals. Feeding studies with labelled fenbutatin oxide at 34 ppm dietary feeding levels indicate that the greatest potential for residues is in the kidney and liver of cattle, with possible low residues in muscle. Conventional feeding studies were also conducted at 11 or 96 ppm in the cattle diet for 21 or 22 days. No residues (<0.02 mg/kg) were found in milk, cream or tissues from the lower feeding level. Residues of fenbutatin oxide were found in all cream and tissue samples from the higher feeding level, while SD 31723 was found only in the liver and kidney. SD 33608 was not detected in any sample (<0.02 mg/kg).

Depending on the assumptions used, a dietary intake of the order of 20 ppm could be estimated, about twice the level in the lower feeding level trial. Adjusting data from the highest feeding level trial to a 20 ppm dietary burden results in maximum fenbutatin oxide residues in liver of 0.02, kidney 0.05, fat of meat 0.01, muscle 0.01 and milk fat 0.05 mg/kg.

Again depending on what assumptions are made, a case could be made for a slight lowering of the previously estimated 0.2 mg/kg limits for liver and kidney, but since the levels are not much greater than the validated limits of determination for these organs, and because more than one of the feed items could be fed at one time, the Meeting concluded that the liver and kidney limits previously estimated for cattle, goats, pigs, horses and sheep could be confirmed. They have been combined under a new proposal at the same level for edible offal.

The Meeting had some reservations about the previous estimates for cattle meat and milk of 0.02 mg/kg at the limit of determination. There was no evidence that the levels would be exceeded in practice, but the analytical method had not been validated below 0.1 mg/kg for any animal matrix in studies provided to the Meeting. For this reason the Meeting recommended increasing the stated limits of determination and hence the MRLs for these commodities to 0.05 mg/kg and limits for the meat of cattle, dogs, horses and sheep have been combined at the same level as a new proposal for meat.

The Meeting also observed that residues of SD 31723 can be about twice those of fenbutatin oxide in cattle liver. Because residues of fenbutatin oxide are found in liver and because it is the only matrix in which SD 31723 exceeds fenbutatin oxide, the Meeting concluded that definition of the residue solely as fenbutatin oxide is satisfactory.

Residues in skim milk and cream indicate a propensity for fenbutatin oxide to accumulate in lipid rather than aqueous media, but levels in muscle do

not differ from those in mesenteric or subcutaneous fat sufficiently to regard fenbutatin oxide as a fat-soluble pesticide.

Feeding chickens at 5 ppm dietary levels produced no residues of fenbutatin oxide or its two metabolites in tissues or eggs, except 0.02 mg/kg fenbutatin oxide in two whole egg samples. From the 25 ppm dietary feeding level the maximum residues of fenbutatin oxide were 0.04 mg/kg in liver, 0.03 mg/kg in kidney and 0.12 mg/kg in whole eggs. These decreased to <0.02 mg/kg in liver and kidney 3 days after cessation of feeding, but the decrease was slower in whole eggs. No residues of either parent compound or metabolites were found in other tissues or organs. As in the case of cattle, residues of SD 13723 were greater than those of fenbutatin oxide in liver (3 to 5 times as high in this case) and residues of SD 33608 were generally comparable to those of the parent compound.

If it is assumed that the greatest dietary intake from feed items for which there are MRLs would be from dry grape pomace (100 mg/kg MRL) and that it is fed at a maximum of 5% of the diet, a dietary intake of about 5 ppm can be estimated. Maximum fenbutatin oxide residues of 0.02 mg/kg in whole egg from the 5 mg/kg feeding level and 0.12 mg/kg from the 25 ppm level support 0.02 mg/kg as a maximum residue level for whole eggs. While SD 317243 might occur near 0.02 mg/kg in liver (0.12 mg/kg from 25 ppm feeding), residues of fenbutatin oxide *per se* would not be expected to be above 0.02 mg/kg. Although residues would be likely not to exceed 0.02 mg/kg in whole eggs, kidney or liver, the same considerations as those mentioned above regarding the levels of method validation for cattle products led the Meeting to conclude that a limit of 0.05 mg/kg (not a limit of determination, because residues around 0.02 mg/kg may occur) would be more appropriate in whole eggs and 0.05 mg/kg (as a limit of determination) in liver and kidney. There would be no compelling need for a limit in poultry meat or fat. Because limits are proposed for eggs and chicken edible offal, 0.05 mg/kg is recommended for chicken meat as a limit of determination level.

Only one of the two analytical methods used in the supervised trials was provided, although the principles were summarized and recoveries and limits of detection were usually provided with field trials data. The two basic methods were both described in earlier monographs. The first is based on chloro-derivatization in a solvent containing HCl followed by GLC determination. The second (e.g. method MMS-R-494-1 provided to the Meeting) includes methylation of fenbutatin oxide, SD 31723 and SD 33608 with methyllithium and determination by GLC with flame-photometric detection of tin. In general determination at 0.02 to 0.05 mg/kg of each compound in cream, 0.1 mg/kg of each in cow liver, and 0.1 to 0.2 mg/kg SD 31723, 0.05 mg/kg SD 33608 and probably $\geq$0.1 mg/kg parent compound in grapes appears to be supported by sample chromatograms. Recoveries from the various substrates were generally $\geq$80%, but at near MRL levels, especially for fenbutatin oxide.

The submitted method may be adequate for regulatory analysis at proposed MRL levels, although submission of all of the analytical methods with sufficient information to permit estimation of the limits of determination and of any information on multi-residue methods suitable for enforcement is desirable.

A proposed liquid chromatographic procedure was also provided, but it was not validated sufficiently for the Meeting to recommend its use.

RECOMMENDATIONS

On the basis of the data on residues from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits. The Meeting could not confirm some current CXLs at this time. They are also indicated below.

Definition of the residue: fenbutatin oxide

Commodity		Recommended MRL (mg/kg)		PHI, days
CCN	Name	New or confirmed	Previous	
TN 0660	Almonds	0.5	-	14
FP 0226	Apple	W (Note 1)	5	14
AB 0226	Apple pomace, dry	40	20	14
FI 0237	Banana	10	-	7
FS 0013	Cherries	10	5	
PO 0840	Chicken, edible offal of	0.05*	-	
PM 0840	Chicken meat	0.05*	-	
FC 0001	Citrus fruits	W (Note 2)	5	
AB 0001	Citrus pulp, dry	25	7	7
VC 0424	Cucumber	0.5	1	
MO 0105	Edible offal (mammalian)	0.2	-	
V0 0440	Egg plant	W	1	
PE 0112	Eggs	0.05	-	
VC 0425	Gherkin	W	1	
FB 0269	Grapes	5	5	
FC 0203	Grapefruit	5	5 for citrus	7
AB 0269	Grape pomace, dry	100	-	
MO 1292	Horse, kidney	W (Note 3)	0.2	
MO 1293	Horse, liver	W (Note 3)	0.2	
MO 0098	Kidney of cattle, goats, pigs and sheep	W (Note 3)	0.2	
MO 0099	Liver of cattle, goats, pigs and sheep	W (Note 3)	0.2	
FC 0206	Mandarin	5	5 for citrus	7
MM 0095	Meat	0.05*	-	
MO 0096	Meat of cattle, goats, horses, pigs and sheep	W (Note 4)	0.02* 5 for citrus	
VC 0046	Melons, except Watermelon	W	1	
ML 0106	Milks	0.05*	0.02*	
FC 0208	Oranges, Sweet	5	5 for citrus	7
FS 0247	Peaches	7	7	14
FP 0230	Pear	W (Note 1)	5	14
TN 0672	Pecans	0.5	-	14
VO 0445	Peppers, Sweet	W	1	
FS 0014	Plums (including Prunes)	3	3	14
FP 0009	Pome fruits	5	Apple 5, Pear 5	14
DF 0014	Prunes (dried plums)	10	-	
DG 5623	Raisins	20	-	
FB 0275	Strawberry	10	3	1
VO 0448	Tomato	1	1	
TN 0678	Walnuts	0.5	-	14

* At or about the limit of determination W: The previous recommendation is withdrawn

Notes 1. Replaced by limit for Pome fruit
2. Replaced by separate limits for Grapefruit, Mandarin, and Orange, sweet
3. Replaced by Edible offal (mammalian)
4. Replaced by revised limit for Meat

FURTHER WORK OR INFORMATION

Desirable

1. Information on whether residues in US stone fruit trials in 1993 Monograph Table 8 references 5,6,7,9 (cherries), 21, 23, 24, (plums), and 29, 30 (peaches), were corrected for analytical recoveries.

2. Information on South African GAP for the use of fenbutatin oxide on peaches.

3. Submission of the analytical methods used in the supervised field trials and in the cow feeding study TIR-26-119-73, with validation information.

4. Current information on analytical methods suitable for enforcement for both plant and animal foods, including multi-residue methods.

5. Current information on the stability of residues in stored analytical samples.

6. Current information on the fate of residues in poultry, plants, soil and water/sediment systems. Metabolism studies on rats, goats and hens reportedly submitted to WHO are specifically requested .

7. Information on residues in food in commerce or at consumption.

8. Information on the interval between the last feeding and slaughter in

cow feeding study TIR-26-119-73 (Koos, 1973).

9. Submission of Report 22-112-74 (on the fate of residues), referenced in Potter and Nugent (1978), as the basis for analyses of animal products for fenbutatin oxide, SD 31723 and SD 33608.

10. Additional pome fruit data reflecting US concentrated spray GAP.

11. Tomato processing information.

REFERENCES

References cited in the text are listed first, followed by those cited in Tables 2-8. Each set of references is numbered separately.

Text references

1. Bosio, P. 1976. Residues of Torque and its Breakdown Product SD 31723 in Melons From France - 1976 Trials. Unpublished Shell Report No. BEGR.0015.77. 1992 Shell submission to the EC, Volume 9.

2. Broadbent, H. and Smith, S. Compostion of Torque Technical Material ex ACIMA Chemical Industries Ltd. Inc. Unpublished Shell International Co. Ltd. Report SBGR.90.195.

3. Fisk, P.R. 1991. Fenbutatin Oxide (TORQUE): Melting Point and Density. Unpublished Shell International Co. Ltd. Report SBGR.90.276.

4. Horne, P. 1988. Hydrolysis of ^{119m}Sn-Fenbutatin Oxide in Buffer Solutions of pH 5, 7 and 9. Unpublished E.I. du Pont Report AMR-923-87, revised July 28, 1988. FB-322-001.

5. Koos, G.A. 1973. Determination of Vendex* Miticide Residues in Milk and Tissues of Lactating Cows Fed with ^{119}Sn Labelled SD 14114. Unpublished Shell Report No. TIR-26-119-73. June 7, 1993 submission to FAO by Shell International Chemical Co. Ltd.

6. MacDonald, I., Howes, D. and Flack, I. 1992a. Fenbutatin Oxide Solubility in Various Solvents. Unpublished Huntingdon Research Report SLL211b/920659.

7. MacDonald, I., Howes, D. and Flack, I. 1992b. Fenbutatin Oxide Physico-chemical Properties. Unpublished Huntingdon Research Report SLL 211a/911576.

8. Melander, W.R. 1988. n-Octanol/warter Partition Coefficient of Fenbutatin Oxide. Unpublished E.I. du Pont Report AMR-1108-88. FB-316-001.

9. Nicolas, Pr A, Leroy, P. and Baissouni, Pr B. (No date). High Performance Liquid Chromatographic Assay of Fenbutatin Oxide Residues in Fruits. Submission to 1993 JMPR. Although undated, references cited are as recent as 1992.

10. Potter, J. and Nugent, K. 1978. Residues of SD 14114, the Active Ingredient in Vendex® Miticide, and Its Two Organotin Metabolites, SD 31723 and SD 33608, In Milk and Tissues From Dairy Cattle Fed Vendex® Miticide. Unpublished Shell Report TIR-24-223-78.

11. Potter, J. and Nugent, K. 1979. Residues of SD 14114, the Active Ingredient in Vendex® Miticide, and Its Two Organotin Metabolites, SD 31723 and SD 33608, In Eggs and Tissues From Chickens Fed Vendex® Miticide. Unpublished Shell Report TIR-24-233-79.

12. Shell (undated). Determination of the Vapour Pressures of Fenbutatin oxide. Unpublished Shell International Co. Ltd. Report SBGR.90.276. FB-306-001.

13. Shell, 1972. Residue Determination of Vendex Miticide and a Possible Metabolite (SD 31723) in Apples, Pears, Oranges, and Their Processed Products - GLC and TLC Procedures. Unpublished Shell Chemical Report MMS-345-1. FB-244-007.

15. Shell, 1973. Residues of ^{119}Sn in Milk and Tissues From Cows Fed SD 1414-^{119}Sn. Unpublished Shell Report No. TIR-22-126-73. June 7, 1993 submission to FAO by Shell International Chemical Co. Lmtd.

16. Shell, 1973. Residues of ^{119}Sn in Milk and Tissues From Cows Fed SD 1414-^{119}Sn. Unpublished Shell Report No. TIR-22-126-73. June 7, 1993 submission to FAO by Shell International Chemical Co. Lmtd.

17. Shell Chemie. 1974. Residues of Torque® in Gherkins from Holland. Unpublished Shell Report WKGR. 0164.74. Data summary from this report submitted to the 1992 JMPR by the Dutch government.

18. Shell, 1979. Residue Determination of Vendex® Miticide and its Organotin Metabolites SD 31723 and SD 33608, In Agricultural Commodities - GLC/Sn-FPD Method. Unpublished Shell Development Co. Report MMS-R-494-1.

Table references

Table 2 (miscellaneous commodities)

1. Perret, G.R. 1983a. Report on Residues of Fenbutatin oxide in Avocado Pears for Shell Chemical (Australia) Proprietary Limited. Unpublished Shell report ASTL.83.007. Vol. 9 of November 1992 Shell submission to the EC Commission.

2. Barber, G. and Wendt, M. 1984a. Residue Levels of Vendex(R) Miticide and Its Organotin Metabolites SD 31723 and SD 33608 in Avocados, Receiving Three Applications of Vendex, A California Study. Unpublished Shell Report No. RIR-24-153-83. Vol. 9 of November 1992 Shell submission to the EC Commission.

3. Barber, G. and Wendt, M. 1984b. Residue Levels of Vendex(R) Miticide and its Organotin Metabolites SD 31723 and SD 33608 in Avocados, Receiving Three Applications of Vendex, A California Study. Unpublished Shell Report No. RIR-24-171-83. Vol. 9 of November 1992 Shell submission to the EC Commission.

4. Perret, G.R. 1983b. Report on Residues of Fenbutatin oxide in Bananas for Shell Chemical (Australia) Proprietary Limited. Unpublished Shell Report No. ASTL.83.008. Vol. 9 or November 1992 Shell submission to the EC Commission.

5. Perret, G.R. 1985. Report on Residues of Fenbutatin oxide in Bananas for Shell Chemical (Australia) Proprietary Limited. Unpublished Shell Report No. ASTL.853.001. Vol. 9 or November 1992 Shell submission to the EC Commission.

6. Bosio, P.G. 1981. Residues of Torque in Bananas from Australia - 1981 Trials. Unpublished Shell Report No. BEGR.82.005. Vol. 9 or November 1992 Shell submission to the EC Commission.

7. Bosio, P.G. 1983a. Residues of Fenpropathrin and Fenbutatin Oxide in Green Beans from France Treated with Danitol* and Torque*, - 1982 trials. Unpublished Shell Report No. GEGR.83.006. Vol. 9 1992 Shell submission to the EC Commission.

8. Bosio, P.G. 1978a. Residues of Torque and Its Breakdown Product SD 31723 in Beans From Holland - 1978 Trials. Unpublished Shell Report No. BEGR.0006.79. Vol. 9 1992 Shell submission to the EC Commission.

9. Bosio, P.G. 1977a. Residues of Torque and Its Breakdown Product SD 31723 in Cucumbers From Denmark - 1977 Trials. Unpublished Shell Report No. BEGR.0022.78. Vol. 9 1992 Shell submission to the EC Commission.

10. Bosio, P.G. 1980. Residues of Torque in Cucumbers From France - 1980 Trials. Unpublished Shell Report No. BEGR.80.173. Vol. 9 1992 Shell submission to the EC Commission.

11. Bosio, P.G. 1982a. Residues of Fenpropathrin and Fenbutatin Oxide in Cucumbers From France Treated with Danitol and Torque - 1982 Glasshouse Trials. Unpublished Shell Report No. BEGR.83.007. Vol. 9 1992 Shell submission to the EC Commission.

12. Dutson, N.J. 1974. Residues of Torque® in Cucumbers From Holland. Unpublished Shell Report No. WKGR.0146.74. Vol. 9 1992 Shell submission to the EC Commission.

13. Lukar, A. and Wallace, B.G. 1981. Residues of Torque® in Cucumbers Treated in the Glasshouse. Unpublished Shell Report No. SBGR.81.223. Vol. 9 1992 Shell submission to the EC Commission.

14. Marxmiller, R.L. 1989. Magnitude of Residues of Fenbutatin Oxide and Metabolites in Cucumbers Treated With Vendex® Miticide. Unpublished E.I. du Pont de Nemours and Co. Report No. AMR-1138-88. Joint DuPont/Shell submission to the 1992 JMPR

15. Bosio, P.G. 1983b. Residues of Fenbutatin Oxide in Aubergines From France - 1983 Trials. Unpublished Shell Report No. BETR.84.026. Vol. 9 1992 Shell submission to the EC Commission.

16. Perret, G.R. 1988. Report on Residues of Fenbutatin oxide in Hops for Shell Chemical (Australia) Proprietary Limited. Unpublished Shell Report No. ASTL.88.001. Vol. 9 1992 Shell submission to the EC Commission.

17. Carlon, R. 1989. Residues of Fenbutatin oxide in Hops from Germany Treated with Torque. Unpublished Shell Report No. BETR.90.017 (listed as R303.89 in Vol. 1 1992 EC summary). Vol. 9 1992 Shell submission to the EC Commission.

18. Bosio, P.G. 1977b. Residues of Torque* and Its Breakdown Product SD 31723 in Melons From France - 1976 Trials. Unpublished Shell Report No. BEGR.0015.77. Vol. 9 1992 Shell submission to the EC Commission.

19. Wallace, B. 1976. Residues of Torque* in Red Peppers From Belgium. Unpublished Shell Report No. WKGR.0019.76. Vol. 9 1992 Shell submission to the EC Commission.

20. Sherren, A. 1976a. Residues of Torque* in Peppers From Holland. Unpublished Shell Report No. WKGR.0012.76. Vol. 9 1992 Shell submission to the EC Commission.

21. Carlon, R. 1988. Residues of Fenbutatin oxide in Soya Beans From France Treated with Torque - 1988 Trials. Unpublished Shell Report No. BETR.89.009. Vol. 9 1992 Shell submission to the EC Commission.

22. Bosio, P.G. 1982b. Residues of Torque* in Tomatoes From Denmark - 1981 Trials. Unpublished Shell Report No. BEGR.89.088. Vol. 9 1992 Shell submission to the EC Commission.

23. Bosio, P.G. 1984. Residues of Fenbutatin oxide in Tomatoes From France Treated with Torque - 1983 Trials. Unpublished Shell Report No. BETR.84.027. Vol. 9 1992 Shell submission to the EC Commission.

24. Gilham, J.A. 1975. Residues of Torque* in Tomatoes From Italy. Unpublished Shell Report No. WKGR.0179.74. Vol. 9 1992 Shell submission to the EC Commission.

25. Sherren, A.J. 1976b. Residues of Torque* in Tomatoes From Holland. Unpublished Shell Report No. WKGR.0011.76. Vol. 9 1992 Shell submission to the EC Commission.

26. Bosio, P.G. 1978b. Residues of Torque* and its Breakdown Product SD 31723 In Tomatoes From South Africa - 1976 Trials. Unpublished Shell Report

No. BEGR.89.088. Vol. 9 1992 Shell submission to the EC Commission.

27. Archer, S. and Woodbridge, A. 1981. Residues of Torque* in Tomatoes From UK. Unpublished Shell Report No. SBGR.81.138. Vol. 9 1992 Shell submission to the EC Commission.

28. Wallace, B.G. 1975. Residues of Torque* in Tomatoes From UK. Unpublished Shell Report No. WKGR.0041.75. Vol. 9 1992 Shell submission to the EC Commission.

Table 3 (citrus fruit)

1. Katague, D.B. 1972. Determination of SD 1414 and SD 31723 Residues in Oranges and Their Processed By-products (Effect of Normal Washing Procedures). Unpublished Shell Report No. TIR-26-113-72. Vol. 4, November 1992 Shell submission to the EC Commission.

2. Shell. 1978a. Residue Data for Vendex® Miticide and its Organotin Metabolites, SD-31723 and SD-33608, In Whole Oranges Resulting From one Application of Vendex to Orange Trees, a California Study. Shell Report No. TIR-24-320-78. Vol. 4, November 1992 Shell submission to the EC Commission

3. Shell. 1978b. Residue Data for Vendex® Miticide and its Organotin Metabolites, SD-31723 and SD-33608, In Whole Oranges Resulting From one Application of Vendex to Orange Trees, a California Study. Shell Report No. TIR-24-319-78. Vol. 4, November 1992 Shell submission to the EC Commission.

4. Shell. 1979a. Residue Data For Vendex® Miticide and its Organotin Metabolites, SD 31723 and SD 33608, in Oranges Following Four Applications of Vendex to Orange Trees, an Arizona Study. Unpublished Shell Report No. RIR-24-168-79. Vol. 4, November 1992 Shell submission to the EC Commission.

5. Shell. 1980a. Residue Data for Vendex® Miticide and its Organotin Metabolites, SD 33608 and SD 31723 in Oranges Following Four Applications of Vendex to Orange Trees, a Florida Study. Unpublished Shell Report No. RIR-24-379-80. Vol. 4, November 1992 Shell submission to the EC Commission.

6. Shell. 1980b. Residue Data for Vendex® Miticide and its Organotin Metabolites, SD 33608 and SD 31723 in Oranges Receiving Four Applications of Vendex, a California Study. Unpublished Shell Report No. RIR-24-372-80. Vol. 4, November 1992 Shell submission to the EC Commission.

7. Potter, J.C. and Kenney, F. H. 1980. Residue Levels of Vendex® Miticide and its Organotin Metabolites SD 31723 and SD 33608 in Whole Oranges Following Four Applications to Orange Trees a Florida Study. Unpublished Shell Report No. RIR-24-355-79. Vol. 4, November 1992 Shell submission to the EC Commission.

8. Marxmiller, R.L. 1988. Magnitude of Residues of Fenbutatin Oxide and Metabolites in Citrus and Citrus Processed Products Following Application of Vendex® at the Maximum Labeled Rate. Unpublished Shell Report No. AMR-943-87. Vol. 4, November 1992 Shell submission to the EC Commission.

9. Perret, G.R. 1983. Report on Residues of Torque in Oranges and Mandarins for Shell Chemical (Australia) Proprietary Limited. Unpublished Shell Report No. ASTL.83.001. Vol. 4, November 1992 Shell submission to the EC Commission.

10. Bosio, P.G. 1982. Residues of Torque in Oranges from Australia - 1981 trials. Unpublished Shell Report No. BEGR.82.006. Vol. 4, November 1992 Shell submission to the EC Commission.

11. Bosio, P.G. 1985. Residues of Fenbutatin oxide in Oranges from brazil Treated With Torque - 1985 Trials. Unpublished Shell Report No. BETR.86.720. Vol. 4, November 1992 Shell submission to the EC Commission.

12. Dutton, A.J. 1974a. Residues of Torque* in Citrus From Spain. Unpublished Shell Report No. WKGR.0040.74. Vol. 4, November 1992 Shell

submission to the EC Commission.

13. Dutton, A.J. 1974b. Residues of Torque* in Citrus From Spain. Unpublished Shell Report No. WKGR.0049.74. Vol. 4, November 1992 Shell submission to the EC Commission.

14. Bosio, P.G. 1976. Residues of a Breakdown Product of Torque* (SD 31723) in Citrus From Italy - 1974 Trials. Unpublished Shell Report No. BEGR.0070.76. Vol. 4, November 1992 Shell submission to the EC Commission.

15. Bosio, P.G. 1975. Residues of Torque* in Citrus From Italy - 1974 Trials. Unpublished Shell Report No. BEGR.0022.75. Vol. 4, November 1992 Shell submission to the EC Commission.

16. Shell. 1981a. Residue Data for Vendex® Miticide and its Organotin Metabolites SD 33608 and SD 31723 in Lemons Following Four Applications of Vendex to Lemon Trees, a California Study. Unpublished Shell Report No. RIR-24-129-81. Vol. 4, November 1992 Shell submission to the EC Commission.

17. Shell. 1977a. Residue Data for Vendex® Miticide in Lemons Resulting from one Application of Vendex, a California Study. Unpublished Shell Report No. TIR-24-210-77. Vol. 4, November 1992 Shell submission to the EC Commission.

18. Shell. 1977b. Residue Data for Vendex® Miticide in Lemons Resulting from one Application of Vendex, a California Study. Unpublished Shell Report No. TIR-24-209-77. Vol. 4, November 1992 Shell submission to the EC Commission.

19. Shell. 1977c. Dissipation of Vendex® Miticide in Lemons Resulting from one Application of Vendex, a California Study. Unpublished Shell Report No. TIR-24-208-77. Vol. 4, November 1992 Shell submission to the EC Commission.

20. Shell. 1979b. Residue Data for Vendex® Miticide and its Organotin Metabolites SD 31723 and SD 33608 in Lemons Following Four Applications of Vendex to Lemon Trees, an Arizona Study. Unpublished Shell Report No. RIR-24-170-79. Vol. 4, November 1992 Shell submission to the EC Commission.

21. Shell. 1981b. Residue Data for Vendex® 4L Miticide and its Organotin Metabolites SD 31723 and SD 33608 in Lemons Following Four Applications of Vendex to Lemon Trees, an Arizona Study. Unpublished Shell Report No. RIR-24-168-81. Vol. 4, November 1992 Shell submission to the EC Commission.

22. Shell. 1981c. Residue Data for Vendex$^{(R)}$ 4L Miticide and its Organotin Metabolites SD 31723 and SD 33608 in Lemons Following Four Applications of Vendex to Lime Trees, a Florida Study. Unpublished Shell Report No. RIR-24-289-81. Vol. 4, November 1992 Shell submission to the EC Commission.

23. Shell. 1979c. Residue Data for Vendex® Miticide and its Organotin Metabolites SD 31723 and SD 33608 in Grapefruit Following Four Applications of Vendex to Grape Fruit Trees, a Texas Study. Unpublished Shell Report No. RIR-24-126-80. Vol. 4, November 1992 Shell submission to the EC Commission.

24. Shell. 1980c. Residue Data for Vendex® Miticide and its Organotin Metabolites SD 33608 and SD 31723 in Grapefruit Following Four Applications of Vendex to Grapefruit Trees, a Florida Study. Unpublished Shell Report No. RIR-24-376-80. Vol. 4, November 1992 Shell submission to the EC Commission.

25. Shell. 1977d. Residue Data for Vendex® Miticide and its Organotin Metabolites SD 31723 and SD 33608 in Whole Grapefruits Resulting From Four Applications of Vendex to Grapefruit Trees, a Texas Study. Unpublished Shell Report No. RIR-24-117-78. Vol. 4, November 1992 Shell submission to the EC Commission.

26. Shell. 1972. Residue Data for SD 14114 and SD 31723 in Grapefruit From California. Unpublished Shell Report No. TIR-26-144-72. Vol. 4, November 1992 Shell submission to the EC Commission.

27. Shell. 1977e. Residue Data for Vendex® Miticide and its Organotin Metabolites SD 31723 and SD 33608 in Grapefruits Following Four Applications of Vendex to Grapefruit Trees, an Arizona Study. Unpublished Shell Report No. RIR-24-169-79. Vol. 4, November 1992 Shell submission to the EC Commission.

Table 4 (grapes)

1. Bosio, P.G. 1975a. Residues of Torque® in Vines From France - 1974 Trials. Unpublished Shell Report No. BEGR.0019.75. 1992 Vol. 6 Shell Submission to the EC.

2. Bosio, P.G. 1975b. Residues of Torque® in Vines From France - 1974 Trials Part II: Wine. Unpublished Shell Report No. BEGR.0022.75. 1992 Vol. 6 Shell Submission to the EC.

3. Dutton, A.J. 1973. Residues of SD 12114 in Grapes Grown in France. Unpublished Shell Report No. WKGR.0094.73. 1992 Vol. 6 Shell Submission to the EC.

4. Dutton, A.J. 1974. Residues of Torque ® in Grapes Grown in France. Unpublished Shell Report No. WKGR.0016.74 1992 Vol. 6 Shell Submission to the EC.

5. Bosio, P.G. 1979. Residues of Torque® in Grapes From Germany - 1979 Trials. Unpublished Shell Report No. BEGR.80.112. 1992 Vol. 6 Shell Submission to the EC.

6. Bosio, P.G. 1980. Residues of Ripcord® and Torque® in Grapes From Germany - 1980 Trials. Unpublished Shell Report No. BEGR.81.034. 1992 Vol. 6 Shell Submission to the EC.

7. Charmasson, R. 1987. Residues of Fenbutatin oxide in Grapes, Grape Juice and Wine From Germany - 1987 Trials. Unpublished Shell Report No. BEGR.88.010. 1992 Vol. 6 Shell Submission to the EC.

8. Bosio, P.G. 1974a. Residues of Torque® in Grapes From Italy - 1974 Trials. Unpublished Shell Report No. BEGR.0015.75. 1992 Vol. 6 Shell Submission to the EC.

9. Bosio, P.G. 1974b. Residues of a Breakdown Product of Torque® (SD 31723) in Grapes From Switzerland - 1974 Trials. Unpublished Shell Report No. BEGR.0069.76. 1992 Vol. 6 Shell Submission to the EC.

10. Nugent, K.D. and Krudop, W.L. 1975a. Residue Levels of Vendex® Miticide in Wine Resulting From the Application of Vendex to the Source Grapes, A California Study. Unpublished Shell Report No. TIR-24-178-74 Part II. 1992 Vol. 6 Shell Submission to the EC.

11. Nugent, K.D. and Krudop, W.L. 1975b. Residue Levels of Vendex® Miticide in Grapes Resulting From the Application of Vendex, A California Study. Unpublished Shell Report No. TIR-24-178-74. 1992 Vol. 6 Shell Submission to the EC.

12. Shell. 1973. Residue Data For Vendex® Miticide in Grapes and Raisins From Modesto, California. Unpublished Shell Report No. TIR-24-703-73. 1992 Vol. 6 Shell Submission to the EC.

13. Shell. 1979a. Residue Data For Vendex® Miticide and Its Organotin Metabolites SD 31723 and SD 33608 in Grapes Following Three Applications of Vendex® to Grape Vines, a California Study. Unpublished Shell Report No. TIR-24-244-79. 1992 Vol. 6 Shell Submission to the EC.

14. Shell. 1979b. Residue Data For Vendex® Miticide and Its Organotin Metabolites SD 31723 and SD 33608 in Grapes Following Three Applications of Vendex® to Grape Vines, a California Study. Unpublished Shell Report No. TIR-24-212-79. 1992 Vol. 6 Shell Submission to the EC.

15. Nugent, K.D. and Krudop, W.L. 1975c. Residue Levels of Vendex® Miticide in Grapes Resulting From the Application of Vendex, A Pennsylvania Study. Unpublished Shell Report No. TIR-24-238-74. 1992 Vol. 6 Shell Submission to the EC.

16. Nugent, K.D. and Krudop, W.L. 1975d. Residue Levels of Vendex® Miticide in Grape Pomace and Wine Resulting From the Application of Vendex to the Source Grapes, A Pennsylvania Study. Unpublished Shell Report No. TIR-24-238-74 Part II. 1992 Vol. 6 Shell Submission to the EC.

17. Shell. 1975. Residue Data For Vendex® Miticide in Grapes Resulting From The Application of Vendex® From Mizpah, New Jersey. Unpublished Shell Report No. TIR-24-196-75. 1992 Vol. 6 Shell Submission to the EC.

18. Shell. 1976. Residue Data For Vendex® Miticide in Grapes, Raisins, Wine and Dried Pomace From Grape Plants Receiving Two Applications of Vendex®, A California Study. Unpublished Shell Report No. TIR-24-244-76. 1992 Vol. 6 Shell Submission to the EC.

19. Shell. 1980a. Residue Data For Vendex® Miticide and Its Organotin Metabolites SD 33608 and SD 31723 in Grapes Following Two Applications of Vendex® to Grape Vines, a California Study. Unpublished Shell Report No. TIR-24-212-79. 1992 Vol. 6 Shell Submission to the EC.

20. Shell. 1980b. Residue Data For Vendex® Miticide and Its Organotin Metabolites SD 33608 and SD 31723 in Grapes and Raisins Following Two Applications of Vendex® to Grape Vines, a California Study. Unpublished Shell Report No. TIR-24-288-80. 1992 Vol. 6 Shell Submission to the EC.

21. Shell. 1980c. Residue Data For Vendex® Miticide and Its Organotin Metabolites SD 33608 and SD 31723 in Grapes Following Two Applications of Vendex® to Grape Vines, a California Study. Unpublished Shell Report No. TIR-24-287-80. 1992 Vol. 6 Shell Submission to the EC.

22. Shell. 1980d. Residue Data For Vendex® Miticide and Its Organotin Metabolites SD 33608 and SD 31723 in Grapes Following Two Applications of Vendex® to Grape Vines, a California Study. Unpublished Shell Report No. TIR-24-314-80. 1992 Vol. 6 Shell Submission to the EC.

23. Shell. 1978. Residue Data For Vendex® Miticide and Its Organotin Metabolites SD 31723 and SD 31723 in Grapes and Processed Products Resulting From Three Applications of Vendex® to Grape Vines, a California Study. Unpublished Shell Report No. TIR-24-254-78. 1992 Vol. 6 Shell Submission to the EC.

24. Shell. 1981. Residue Data For Vendex® Miticide and Its Organotin Metabolites SD 31723 and SD 31723 in Grapes Receiving Two Applications of Vendex®, a Pennsylvania Study. Unpublished Shell Report No. TIR-24-291-81. 1992 Vol. 6 Shell Submission to the EC.

25. Nugent, K.D. 1977. Reduction of Vendex® Miticide Residues on Grapes by Water Rinsing - Residues Removed by Rinsing and Residue Levels of Vendex® in Rinsed Grapes Following One Application of Vendex, A California Study. Unpublished Shell Report No. TIR-24-303-77. 1992 Vol. 6 Shell Submission to the EC.

26. Shell. 1974. Residue Data For Vendex® Miticide in Grapes Resulting From The Application of Vendex® From Lansing, Michigan. Unpublished Shell Report No. TIR-24-279-74. 1992 Vol. 6 Shell Submission to the EC.

27. Shell. 1980e. Residue Data For Vendex® Miticide and Its Organotin Metabolites SD 33608 and SD 31723 in Grapes Following Three Applications to Grape Vines, a California Study. Unpublished Shell Report No. TIR-259-80. 1992 Vol. 6 Shell Submission to the EC.

28. Shell. 1980f. Residue Data For Vendex® Miticide and Its Organotin Metabolites SD 33608 and SD 31723 in Grapes Following Three Applications of Vendex to Grape Vines, a California Study. Unpublished Shell Report No. TIR-254-80. 1992 Vol. 6 Shell Submission to the EC.

29. Shell. 1980g. Residue Data For Vendex® Miticide and Its Organotin Metabolites SD 33608 and SD 31723 in Grapes Following Three Applications of Vendex to Grape Vines, a Michigan Study. Unpublished Shell Report No. TIR-131-80. 1992 Vol. 6 Shell Submission to the EC.

30. Shell. 1979c. Residue Data For Vendex® Miticide and Its Organotin Metabolites SD 33608 and SD 31723 in Grapes and Raisins, Following Three Applications of Vendex to Grape Vines, a California Study. Unpublished Shell Report No. TIR-246-79. 1992 Vol. 6 Shell Submission to the EC.

31. Nugent, K., Doern, B. and Krudop, W. 1977. Residue Levels of Vendex® Miticide in Grapes Receiving Three Applications of Vendex, a Pennsylvania Study. Unpublished Shell Report No. TIR-332-76. Joint Shell International and E.I. Du Pont Submission to the 1992 JMPR, Vol. 3.

32. Eble, J. 1989. Magnitude of Residues of Fenbutatin Oxide and Two Metabolites in Grapes and the Processed Fractions of Grapes After Application of Vendex® Miticide. Unpublished Du Pont Report No. AMR-1126-88. Joint Shell International and E.I. Du Pont Submission to the 1992 JMPR, Vol. 3.

Table 5 (tree nuts)

1. Shell 1982. Residue Data For Vendex(R) Miticide and its Organotin Metabolites SD 31723 and SD 33608 in Pecans Following Three Applications of Vendex to Pecan Trees, a Louisiana Study. Unpublished Shell Report RIR-24-118-82. 1992 Shell Volume 8 submission to the European Commission.

2. Shell 1981a. Residue Data For Vendex(R) Miticide and its Organotin Metabolites SD 31723 and SD 33608 in Pecans Following Three Applications of Vendex to Pecan Trees, a Georgia Study. Unpublished Shell Report RIR-24-306-81. 1992 Shell Volume 8 submission to the European Commission.

3. Shell 1979a. Residue Data For Vendex(R) Miticide and its Organotin Metabolites SD 31723 and SD 33608 in Pecans Following Three Applications of Vendex to Pecan Trees, A Louisiana Study. Unpublished Shell Report RIR-24-324-79. 1992 Shell Volume 8 submission to the European Commission.

4. Nugent, K., Doern, B. and Krudop, W. 1974. Residue Levels of Vendex(R) Miticide in Pecans Resulting From the Application of Vendex, a Mississippi Study. Unpublished Shell Report TIR-24-276-74. 1992 Shell Volume 8 submission to the European Commission.

5. Nugent, K., Doern, B. and Krudop, W. 1976a. Residue Levels of Vendex(R) Miticide in Pecans Resulting From the Application of Vendex, a Texas Study. Unpublished Shell Report TIR-24-186-74. 1992 Shell Volume 8 submission to the European Commission.

6. Nugent, K., Doern, B. and Krudop, W. 1976b. Residue Levels of Vendex(R) Miticide in Pecans Resulting From the Application of Vendex, a Louisiana Study. Unpublished Shell Report TIR-24-273-74. 1992 Shell Volume 8 submission to the European Commission.

7. Shell. 1980a. Residue Data For Vendex(R) Miticide and its Organotin Metabolites SD 31723 and SD 33608 in Walnuts Receiving Three Applications of Vendex, a California Study. Unpublished Shell Report RIR-24-322-79. 1992 Shell Volume 8 submission to the European Commission.

8. Shell. 1979b. Residue Data For Vendex(R) Miticide and its Organotin Metabolites SD 31723 and SD 33608 in Filberts Following Three Applications of Vendex to Filbert Trees, an Oregon Study. Unpublished Shell Report RIR-24-273-79. 1992 Shell Volume 8 submission to the European Commission.

9. Shell. 1979c. Residue Data For Vendex(R) Miticide and its Organotin Metabolites SD 31723 and SD 33608 in Walnuts Following Three Applications of Vendex to Walnut Trees, a California Study. Unpublished Shell Report RIR-24-257-79. 1992 Shell Volume 8 submission to the European Commission.

10. Shell. 1981b. Residue Data For Vendex(R) Miticide and its Organotin Metabolites SD 31723 and SD 33608 in Walnuts Following Three Applications of Vendex to Walnut Trees, a California Study. Unpublished Shell Report RIR-24-332-81. 1992 Shell Volume 8 submission to the European Commission.

11. Shell. 1981c. Residue Data For Vendex(R) Miticide and its Organotin Metabolites SD 31723 and SD 33608 in Walnuts Following Three Applications of Vendex to Walnut Trees, an Oregon Study. Unpublished Shell Report RIR-24-323-81. 1992 Shell Volume 8 submission to the European Commission.

12. Shell. 1977a. Residue Data For Vendex(R) Miticide and its Organotin Metabolites SD 31723 and SD 33608 in Walnut Hulls, Shells and Meats Resulting From Two Applications of Vendex to Walnut Trees, a California Study. Unpublished Shell Report TIR-24-302-77. 1992 Shell Volume 8 submission to the European Commission.

13. Shell. 1977b. Residue Data For Vendex(R) Miticide and its Organotin

Metabolites SD 31723 and SD 33608 in Walnut Hulls, Shells and Meats Resulting From Two Applications of Vendex to Walnut Trees, a California Study. Unpublished Shell Report TIR-24-301-77. 1992 Shell Volume 8 submission to the European Commission.

14. Shell. 1974. Residue Data For Vendex® Miticide in Filbert Nuts Resulting From the Application of Vendex, an Oregon Study. Unpublished Shell Report TIR-24-251-74. 1992 Shell Volume 8 submission to the European Commission.

15. Shell. 1980b. Residue Data For Vendex(R) Miticide and its Organotin Metabolites SD 33608 and SD 31723 in Almonds Following Three Applications of Vendex to Almond Trees, a California Study. Unpublished Shell Report RIR-24-388-80. 1992 Shell Volume 8 submission to the European Commission.

16. Shell. 1981d. Residue Data For Vendex(R) Miticide and its Organotin Metabolites SD 31723 and SD 33608 in Almonds Following Three Applications of Vendex to Almond Trees, a California Study. Unpublished Shell Report RIR-24-271-81. 1992 Shell Volume 8 submission to the European Commission.

17. Shell. 1981e. Residue Data For Vendex(R) Miticide and its Organotin Metabolites SD 33608 and SD 31723 in Almonds Following Three Applications of Vendex to Almond Trees, a California Study. Unpublished Shell Report RIR-24-265-81. 1992 Shell Volume 8 submission to the European Commission.

18. Shell. 1981f. Residue Data For Vendex(R) Miticide and its Organotin Metabolites SD 33608 and SD 31723 in Almonds Following Three Applications of Vendex to Almond Trees, a California Study. Unpublished Shell Report RIR-24-244-81. 1992 Shell Volume 8 submission to the European Commission.

19. Shell. 1973. Residue Data For Vendex(R) Miticide in Almonds Resulting From the Application of Vendex, California Study. Unpublished Shell Report TIR-24-708-73. 1992 Shell Volume 8 submission to the European Commission.

20. Shell. 1977c. Residue Data For Vendex(R) Miticide and its Organotin Metabolites SD 31723 and SD 33608 in Almond Hulls, Shells and Meats Resulting From Two Applications of Vendex to Almond Trees, a California Study. Unpublished Shell Report TIR-24-704-77. 1992 Shell Volume 8 submission to the European Commission.

21. Shell. 1977d. Residue Data For Vendex(R) Miticide and its Organotin Metabolites SD 31723 and SD 33608 in Almond Hulls, Shells and Meats Resulting From Two Applications of Vendex to Almond Trees, a California Study. Unpublished Shell Report TIR-24-270-77. 1992 Shell Volume 8 submission to the European Commission.

22. Shell. 1977e. Residue Data For Vendex(R) Miticide and its Organotin Metabolites SD 31723 and SD 33608 in Almond Hulls, Shells and Meats Resulting From Two Applications of Vendex to Almond Trees, a California Study. Unpublished Shell Report TIR-24-269-77. 1992 Shell Volume 8 submission to the European Commission.

23. Shell. 1976. Residue Data For Vendex(R) Miticide in Almond Hulls, Shells and Nut Meats Resulting From One Application of Vendex, California Study. Unpublished Shell Report TIR-24-231-76. 1992 Shell Volume 8 submission to the European Commission.

24. Nugent, K. and Matsuyama, H. 1979. Residue Levels of Vendex(R) Miticide and its Organotin Metabolites SD 31723 and SD 33608 in Almond Hulls, Shells and Meats Resulting From Two Applications of Vendex to Almond Trees, a California Study. Unpublished Shell Report TIR-24-255-78. 1992 Shell Volume 8 submission to the European Commission.

Table 6 (pome fruit)

Unpublished Shell International Chemical Co. Ltd. Reports submitted (unless otherwise indicated) to the 1993 JMPR in the form of a 1992 Volume 3 submission (summarized in Volume 1) to the European Commission.

Apples

1. DEGR.80.114
2. WKGR.0125.74
3. WKGR.0093.75
4. WKGR.0014.74
5. BETR.86.035
6. BEGR.0043.76
7. BEGR.0105.74
8. BEGR.0016.75
9. BEGR.0004.78
10. BEGR.83.005
11. WKGR.0089.73
12. WKGR.0142.73
13. BEGR.0016.78
14. R-139-89[1]
15. R-140-89[1]
16. R141-89[1]
17. R142-89[1]
18. R143-89[1]
19. R144-89[1]
20. BETR.90.018[2]
21. BEGR.79.106
22. BEGR.81.026
23. WKGR.0027
24. BEGR.0030.76
25. WKGR.0111.74
26. WKGR.0034.74
27. BLGR.80.1116
28. WKGR.0013.74
29. WKGR.0044.76
30. TIR-26-201-73
31. TIR-26-148-72
32. RIR-26-137-72
33. TIR-26-147-72
34. TIR-26-145-72
35. TIR-26-143-72
36. TIR-26-141-72
37. TIR-26-125-71
38. TIR-26-129-72
39. TIR-26-131-72
40. TIR-26-134-72
41. TIR-26-133-72
42. RIR-24-361-78
43. TIR-26-135-72
44. TIR-26-157-73
45. TIR-26-101-74
46. RIR-24-371-78
47. RIR-25-348-79
48. RIR-24-234-80
49. RIR-24-362-78
50. TIR-26-197-73[3]
51. AMR-112588[4]

Pears

52. BEGR.80.116
53. WKGR.0180.74
54. WKGR.1202.75
55. BEGR.0011.75
56. BEGR.0029.76
57. BEGR.0031.76
58. WKGR.0107.74
59. RIR-24-283-80
60. RIR-24-233-80
61. RIR-24-274-80
62. RIR-24-351-79
63. TIR-26-139-71B

[1] Field reports, in German. Table 6 summary based primarily on Volume 1 summary data.
[2] Included in 1992 Volume 3 EC submission, but not in Volume 1 summary.
[3] Included in June 7, 1993 Shell submission, not in EC submission
[4] Included in June 4, 1992 Volume 3 Shell International/E.I Du Pont submission to the 1992 JMPR.

Table 7 (strawberries and raspberries)

Unpublished Shell International Chemical Co. Ltd. Reports submitted (unless otherwise indicated) to the 1993 JMPR in the form of a 1992 Volume 7 submission (summarized in Volume 1) to the European Commission.

Strawberries

1. ASTL.83.005
2. TIR-24-167-25
3. TIR-24-164-75
4. BEGR.82.007
5. BEGR.0014.77
6. BETR.87.004
7. BLGR.0087.77
8. BEGR.81.043
9. BLGR.0086.77
10. WKGR.0068.75
11. RIR-24-240-83
12. RIR-24-168-84
13. TIR-24-195-75
14. RIR-24-158-8L1
15. TIR-14-158-76
16. TIR-24-142-79
17. TIR-24-274-76
18. TIR-24-164-79
19. TIR-24-143-79
20. RIR-24-159-81
21. RIR-24-16L7-84
22. RIR-24-214-81
23. TIR-24-707-73
24. TIR-24-163-79
25. TIR-24-155-74
26. TIR-24-140-74
27. TIR-24-278-74

Raspberries

28. BEGR.81.122

Table 8 (stone fruit)

Unpublished Shell International Chemical Co. Ltd. Reports submitted (unless otherwise indicated) to the 1993 JMPR in the form of a 1992 Volume 5

submission (summarized in Volume 1) to the European Commission.

Cherries

1. BEGR.0066.77
2. BEGR.81.033
3. TIR-24-250-74
4. TIR-24-243-74
5. RIR-24-282-81
6. RIR-24-175-81
7,9 RIR-24-201-l80
8. RIR-24-165-79
7,9 RIR-24-201-80
10. TIR-24-205-78
11. TIR-24-204-78
12. TIR-24-177-78
13. BLGR.0066.77

Plums

14. BEGR.0015.78
15. BEGR.79.107
16. BEGR.81.037
17. BLGR.0088.77
18. BLGR.0050.77
19. BEGR.0014.76
20. RIR-24-128-82
21. RIR-24-108-81
22. RIR-24-102-80
23. RIR-24-333-81
24. RIR-24-226-81
25. RIR-24-182-81
26. TIR-24-242-74
27. AMR-1127-88*
29. TIR-24-216-78
30. TIR-24-223-77
31. TIR-24-700-74
32. TIR-24-714-73
33. TIR-24-702-73
34. TIR-24-713-73

Plums cont'd

35. TIR-24-702-73-B
36. WKGR.0101.75
37. BEGR.0044.76
38. BEGR.82.048
39. BEGR.83.028
40. BEGR.0020.77
41. WKGR.0093.73
42. BEGR.83.008
43. WKGR.0139.73
44. WKGR.0015.74
45. WKGR.0065.75
46. BLGR.0089.77
47. BEGR.0035.76
48. BLGR.79.142
49. WKGR.0108.74

Peaches

28. BEGR.80.115

Peaches cont'd

Nectarines

50. RIR-24-204-81
51. RIR-24-353-81

* Reference 27 included in June 4, 1992 Volume 3 Shell International/E.I Du Pont submission to the 1992 JMPR.

FENPROPATHRIN (186)

IDENTITY

ISO common name: fenpropathrin

Chemical names:

IUPAC: (RS)-a-cyano-3-phenoxybenzyl ,2,3,3-tetramethylcyclopropanecarboxylate

CAS: cyano(3-phenoxyphenyl)methyl 2,2,3,3-tetramethylcyclopropanecarboxylate

Synonyms: S-3206, Danitol, Meothrin, Rody, OMS 1999, WL41706, SD41706, XE-938

CAS Registry No.: 64257-84-7(racemate); 39515-41-8 (unstated steriochemistry)

Structural formula:

Molecular formula: $C_{22}H_{23}NO_3$

Molecular weight: 349.43

Physical and chemical properties

Pure active ingredient

Information was given only for the technical material.

Technical material

Purity: 90%

Physical state: Liquid or solid
Colour: Yellow to brown
Odour: Faint characteristic odour
Density: 1.105
Vapour pressure: 2.15×10^{-6} Pa
Melting range: 45-50°C
Flammability: Flash point: 205°C
Ignition point: 325°C
Solubility in organic solvents (g/l at 23°C):
Acetone > 500
Acetonitrile > 500
Cyclohexanone > 500
Ethyl acetate > 500
Methanol 216
Xylene > 500

Solubility in water 36.3 ì g/l at 25.1°C
Octanol/water partition coefficient:
log P = 6.0 $\pm$ 0.20
Stability: Unstable in alkaline media.
No significant breakdown after 20 weeks storage at 60°C.

Formulation: EC

USE PATTERN

Fenpropathrin is used to control a range of insects, especially mites, in fruits and vegetables. Registered uses are summarized in Table 1. Most countries approve a range of application rates and pre-harvest intervals. The rate of application for tree fruits is normally expressed in terms of spray concentration but a complication arises with low-volume applications, although these are not currently established on a commercial scale. The number of applications permitted for a given crop is seldom specified in official registrations although in two countries, Denmark and Germany, only 2 or 3 applications are allowed for certain crops (see Table), not to limit residues but because the competent authorities operate a policy of rotating insecticides to minimize the development of pest resistance. In practice the number of applications is determined by infection pressure and the persistence of the active ingredient. This is comparatively long because in addition to its insecticidal properties fenpropathrin also exerts a considerable repellent action. The effects of a treatment normally last for 3-4 weeks.

Table 1. Registered uses of fenpropathrin.

Crop	Country	Application			PHI, days
		No.	Rate, kg ai/ha	Spray concn., kg ai/hl	
Apple	Austria			0.005-0.008	21
	Belgium			0.005	14

Crop	Country	Application			PHI, days
		No.	Rate, kg ai/ha	Spray concn., kg ai/hl	
	Cyprus			0.005	21
	Denmark	2	0.15		14
	France			0.01 -0.02	21
	Greece			0.005-0.02	21
	Hungary	2	0.06-0.1		14
	Italy			0.005-0.025	7
	Japan	2		0.007-0.01	14
	Nthlnds			0.005	14
	Portugal			0.02	7
	Spain	2	0.09-0.225	0.006-0.015	30
	Sweden	1	0.15	0.0075	30
	Swtzlnd	2		0.01	42
	UK	2		0.003-0.005	7
	USA	8	0.45		14
Beans	Cyprus			0.005	7
	Germany	3	0.04 -0.08*	0.033-0.067	3
	Portugal			0.01*	2
	Swtzlnd	1		0.01	14
		1		0.01*	7
Currant, black	Sweden	1	0.112	0.0075	60
Cabbage, Head	Swtzlnd	2		0.01	7
Citrus fruits	Cyprus			0.005	21
	Greece	2		0.02	21
	Italy	2		0.02	30
	Japan	4		0.005	7
Cotton seed	Greece		0.13-0.15		
	Spain	2	0.13-0.15		30
	USA	10	0.22		14

Crop	Country	Application			PHI, days
		No.	Rate, kg ai/ha	Spray concn., kg ai/hl	
Cucumber	Austria			0.005	3
	Belgium			0.005	3
	Cyprus			0.007-0.02	21
	Denmark	2		0.0075	3
	Germany	3	0.04-0.08*	0.033-0.067	3
	Greece			0.02	21
	Hungary		0.03-0.1		7
	Italy			0.01 -0.02	7
	Japan	5		0.005-0.01	1
	Nthlnds			0.005	3
	Norway			0.0075	4
	Swtzlnd			0.01*	7
Egg plant	Austria	1		0.005	3
	Belgium	3		0.005	3
	Cyprus			0.005	7
	Greece	1		0.01	21
	Japan	5		0.01	1
	Nthlnds	2		0.005	3
	Spain	2	0.09-0.225	0.006-0.015	7
Gherkin	Belgium	2		0.005	3
	Nthlnds			0.01*	7
Grapes	Austria			0.005	21
	Cyprus			0.007-0.02	21
	France		0.08-0.15		21
	Greece			0.025-0.02	21
	Hungary	2	0.06-0.1		14
	Italy			0.003-0.02	7
	USA	4	0.45		21
Hops	Austria	1		0.005	21

Crop	Country	Application			PHI, days
		No.	Rate, kg ai/ha	Spray concn., kg ai/hl	
	UK	2		0.006	7
Maize	Austria	1		0.005	3
Melons, except Water-melon	Austria	1		0.005	3
	Belgium	1		0.005	3
	Greece			0.02	21
	Japan	4		0.01	1
	Nthlnds	2		0.005	3
	Portugal			0.01*	2
	Spain	1		0.01	30
Mushrooms	Austria	1		0.005	3
Peach	France	2	0.1		21
	Greece			0.02	21
	Italy	2		0.02	7
	Japan	5		0.01	1
	Portugal			0.02	7
	Swtzlnd	2		0.01	42
Pear	Austria			0.005-0.008	21
	Belgium			0.005	14
	Cyprus			0.007-0.02	21
	Denmark	2	0.15		14
	France			0.01 -0.02	21
	Greece			0.005-0.02	21
	Hungary	2	0.06-0.1		14
	Italy			0.005-0.025	7
	Japan	2		0.007-0.01	14
	Nthlnds	1		0.005	14
	Portugal			0.02	7
	Spain	2	0.09-0.225	0.006-0.015	30

Crop	Country	Application			PHI, days
		No.	Rate, kg ai/ha	Spray concn., kg ai/hl	
	Swtzlnd	2		0.01	42
	USA	8	0.45		14
Peppers	Belgium	3			0.0053
	Greece	1			0.0221
	Japan	3			0.0051
	Spain	2	0.09-0.225	0.006-0.015	7
Peppers, Sweet	Austria	1		0.005	3
	Nthlnds	2		0.005	3
Plums	Sweden	1	0.168	0.0075	60
Potato	Cyprus			0.005	7
	Greece			0.01	21
	Italy	1		0.01	21
Pumpkins	Austria	1		0.005	3
Squash, Summer	Austria	1		0.005	3
	Belgium			0.005	3
	Italy	1		0.02	7
	Nthlnds	2		0.005	3
	Spain	2	0.09-0.225	0.006-0.015	7
Tomato	Austria			0.005	3
	Belgium	3		0.005	3
	Cyprus			0.007-0.02	7
	Denmark	2		0.0075*	3
	Germany	3	0.04-0.08*	0.033-0.067	3
	Greece	1		0.01 -0.02	21
	Hungary		0.03-0.1		7
	Italy	5		0.01 -0.02	7
	Japan	3		0.005-0.01	1
	Nthlnds	2		0.005	3
	Norway	1		0.008*	4

Crop	Country	Application			PHI, days
		No.	Rate, kg ai/ha	Spray concn., kg ai/hl	
	Portugal			0.01	2
	Spain	4	0.09-0.225	0.006-0.015	7
Strawberry	Sweden	1	0.08-0.1	0.0075	BF**

* Greenhouse
** Before flowering or after harvest

RESIDUES RESULTING FROM SUPERVISED TRIALS

Residues in crops

A series of studies have been carried out in Europe, Japan and the USA to determine the level of residues likely to arise in crops when fenpropathrin is used according to the range of recommendations for use (Tables 2-9). Crops were mostly treated according to accepted or proposed use recommendations, although in the USA some crops were treated at higher rates. Crop commodities were generally sampled at maturity except in cases where the design of the study involved a range of pre-harvest intervals which were varied by changing the harvest date rather than the date of the last spray application.

The Tables are as follows.

Table
2 Residues of fenpropathrin in apples
3 Residues of fenpropathrin in pears
4 Residues of fenpropathrin in grapes
5 Residues of fenpropathrin in cotton seed
6 Residues of fenpropathrin in gherkins
7 Residues of fenpropathrin in egg plants
8 Residues of fenpropathrin in sweet peppers
9 Residues of fenpropathrin in tomatoes

Underlined residues in Tables 2-9 are from treatments according to GAP. Residues have not normally been corrected for recoveries.

Apples. The available data are summarized in Table 2. As would be expected residue levels were seen to increase with increased application rates, as measured by spray concentration, decreased PHI and, to some extent, increased numbers of applications.

Table 2. Residues of fenpropathrin in apples - whole fruit

Country, Year	Application				PHI, days	Residue, mg/kg	Ref.
	Form.	No.	kg ai/ha	kg ai/hl			
France	10% EC	1	0.2	0.029	0	0.30	10

Country, Year	Application				PHI, days	Residue, mg/kg	Ref.
	Form.	No.	kg ai/ha	kg ai/hl			
1981					7	0.36	10
					14	0.29	10
					21	0.31	10
France	10% EC	1	0.2	0.020	0	0.19	10
1981					7	0.20	10
					14	0.13	10
					21	0.13	10
France	10% EC	1	0.2	0.029	0	0.48	10
1981					7	0.49	10
					14	0.60	10
					22	0.16	10
France	10% EC	1	0.2	0.020	0	0.34	11
1983					7	0.25	11
					14	0.18	11
					21	0.06	11
France	10% EC	1	0.2	0.020	0	0.20	11
1983					7	0.16	11
					14	0.21	11
					21	0.10	11
France	10% EC	1	0.2	0.020	0	0.75	11
1983					7	0.50	11
					14	0.30	11
					21	0.50	11
Hungary	10% EC	4	0.1	0.007	0	0.08	40
1984					1	0.09	40
					5	0.08	40
					9	0.05	40
					14	0.04	40
Hungary	10% EC	1	0.1	0.010	0	0.20	5
1984					1	0.20	5
					3	0.14	5
					6	0.11	5
					10	0.07	5
					15	0.03	5

Country, Year	Application				PHI, days	Residue, mg/kg	Ref.
	Form.	No.	kg ai/ha	kg ai/hl			
					30	0.01	5
USA	2.4 EC	9	0.34	0.009	14	3.3	71
1984		9	0.45	0.012	14	3.9	71
USA	2.4 EC	6	0.34	0.009	14	0.96	66
1984		6	0.45	0.012	14	1.2	66
USA	2.4 EC	8	0.34	0.009	14	1.4	67
1984		8	0.45	0.012	14	1.0	67
USA	2.4 EC	8	0.34	0.09	14	0.38	68
1984		8	0.45	0.12	14	0.57	68
USA	2.4 EC	2	0.45	0.024	14	0.88	77
1984		4	0.45	0.024	14	2.6	77
		6	0.45	0.024	14	2.5	77
		8	0.45	0.024	14	1.7	77
USA	2.4 EC	8	0.34	0.073	14	2.1	69
1984		8	0.45	0.096	14	3.7	69
USA	2.4 EC	2	0.45	0.016	14	1.1	70
1984		4	0.45	0.016	14	2.4	70
		5	0.45	0.016	14	3.0	70
		8	0.45	0.016	14	2.6	70
USA/84	2.4 EC	8	0.34	0.009	14	2.0	78
USA	2.4 EC	8	0.45	0.012	7	2.1	78
1984					14	2.4	78
					21	2.1	78
					28	2.2	78
USA	2.4 EC	2	0.45	0.045	14	0.48	80
1985		4	0.45	0.045	14	1.9	80
		6	0.45	0.045	14	2.6	80
		8	0.45	0.045	14	3.7	80
USA	2.4 EC	8	0.112	0.010	14	0.02	38
1985		8	0.224	0.020	14	2.6	38
		8	0.45	0.040	14	4.5	38
		8	0.9	0.080	14	8.3	38
USA/85	2.4 EC	8	0.45	0.16	14	1.4	39
USA/86	2.4 EC	8	0.45	0.012	14	0.14	T-6719*

Country, Year	Application				PHI, days	Residue, mg/kg	Ref.
	Form.	No.	kg ai/ha	kg ai/hl			
USA/86	2.4 EC	8	0.45	0.016	14	3.6	T-6720
USA/86	2.4 EC	8	0.45	0.035	14	1.5	T-6721
USA	2.4 EC	8	0.45	0.012	14	3.7	T-6722
1986					42	2.8	
USA/86	2.4 EC	8	0.45	0.024	14	2.4	T-6729
USA/87	2.4 EC	8	0.45	0.012	14	2.3	T-6880
USA/87	2.4 EC	8	0.45	0.69**	12	0.40	T-6969
USA/87	2.4 EC	8	0.45	0.96**	14	0.22	T-6970
USA/87	2.4 EC	8	0.45	0.96**	14	0.88	T-6971

* All "T" references are in Fujie 1990b (No. 24).
** Aerial application, hence very low volume per ha.

Pears. The residues in pears were comparable to those in apples; the results are summarized in Table 3 and refer to essentially mature fruit. In two of the three US studies (Robinson, 1984f; 1985b) the samples were all taken on the same day, the variation of the pre-harvest interval being achieved by varying the date of the last application.

Table 3. Residues of fenpropathrin in pears: whole fruit.

Country, Year	Application				PHI, days	Residue, mg/kg	Ref.
	Form.	No.	kg ai/ha	kg ai/hl			
France	10% EC	1	0.15	0.015	0	0.25	8
1980					7	0.21	8
					14	0.20	8
					21	0.12	8
France	10% EC	1	0.15	0.015	0	0.10	8
1980					8	0.05	8
					12	0.03	8
					22	<0.01	8
France	10% EC	1	0.2	0.02	0	0.65	9
1981					7	0.35	9
					14	0.24	9
					21	0.17	9
France	10% EC	1	0.2	0.02	0	0.42	9
1981					7	0.33	9
					14	0.1	9

Country, Year	Application				PHI, days	Residue, mg/kg	Ref.
	Form.	No.	kg ai/ha	kg ai/hl			
					21	0.10	9
France	10% EC	1	0.2	0.02	0	0.19	9
1981					7	0.17	9
					14	0.19	9
					21	0.17	9
USA/84	2.4 EC	6	0.34	0.009	14	0.59	60
USA	2.4 EC	6	0.45	0.012	7	0.68	60
1984					14	0.62	60
					21	0.56	60
					28	0.49	60
USA/84	2.4 EC	6	0.34	0.009	14	0.39	61
USA	2.4 EC	2	0.45	0.012	14	0.27	61
1984		3	0.45	0.012	14	0.45	61
		5	0.45	0.012	14	0.42	61
		6	0.45	0.012	14	0.58	61
USA/85	2.4 EC	6	0.45	0.012	14	1.2	79
USA	2.4 EC	6	0.34	0.009	14	0.94	62
1984		6	0.45	0.012	14	1.3	62
USA/84	2.4 EC	6	0.34	0.024	14	1.3	63
USA	2.4 EC	2	0.45	0.016	14	1.2	64
1984		4	0.45	0.016	14	1.5	64
		5	0.45	0.016	14	1.4	64
		6	0.45	0.016	14	1.7	64
USA	2.4 EC	6	0.45	0.016	7	2.3	65
1984					14	1.9	65
					21	2.0	65
					28	0.95	65
USA	2.4 EC	6	0.11	0.003	14	0.70	37
1985		6	0.22	0.006	14	0.96	37
		6	0.45	0.012	14	1.0	37
		6	0.90	0.024	14	3.2	37
USA	2.4 EC	2	0.45	0.012	14	0.30	72
1985		4	0.45	0.012	14	0.63	72

Country, Year	Application				PHI, days	Residue, mg/kg	Ref.
	Form.	No.	kg ai/ha	kg ai/hl			
		6	0.45	0.012	14	0.74	72
		8	0.45	0.012	14	0.95	72
USA/85	2.4 EC	8	0.45	0.012	21	0.09	72
USA	2.4 EC	6	0.45	0.012	1	1.5	73
1985					7	2.2	73
					14	1.6	73
					21	1.3	73
					28	0.85	73
					35	0.95	73
USA/86	2.4 EC	6	0.45	0.048	14	1.1	T-6709*
USA/86	2.4 EC	6	0.45	0.012	14	2.9	T-6711
USA/86	2.4 EC	6	0.45	0.035	14	1.8	T-6712
USA/86	2.4 EC	6	0.45	0.016	14	2.4	T-6713
USA/87	2.4 EC	6	0.45	0.048	14	1.8	T-6886
USA/87	2.4 EC	6	0.45	0.24**	14	0.3	T-6972
USA/87	2.4 EC	6	0.45	0.96**	14	0.9	T-6973

* All "T" references are in Fujie 1988 (No. 22).
** Aerial application, hence very low volume per ha.

Grapes. The data are shown in Table 4 and most of them refer to mature grapes, an exception being the results from Hungary at a 70-day pre-harvest interval. The US study T-6415 did not involve a variation of the date of the last application but the samples were all described as mature bunches so presumably the range of pre-harvest intervals was too narrow to have a significant effect on maturity. The other three relevant studies in the USA (T-6409, 6414 and 6835) were designed in such a way that the grapes were all harvested at a similar state of maturity.

Table 4. Residues of fenpropathrin in grapes: whole fruit.

Country, Year	Application				PHI, days	Residue, mg/kg	Ref.
	Form.	No.	kg ai/ha	kg ai/hl			
France	10% EC	1	0.075	0.015	0	0.08	7
1980					7	0.04	7
					14	0.02	7
					21	0.01	7

Country, Year	Application				PHI, days	Residue, mg/kg	Ref.
	Form.	No.	kg ai/ha	kg ai/hl			
France	10% EC	1	0.075	0.015	0	0.41	7
1980					8	0.23	7
					14	0.23	7

Country, Year	Application				PHI, days	Residue, mg/kg	Ref.
	Form.	No.	kg ai/ha	kg ai/hl			
					21	0.12	7
France	10% EC	1	0.075	0.019	7	0.15	6
1978					14	0.10	6
					24	0.06	6
France	10% EC	1	0.05	0.009	63	0.01	6
1978		1	0.075	0.014	63	0.02	6
Hungary	10% EC	1	0.08		0	0.08	83
1983					1	0.09	83
					4	0.11	83
					6	0.10	83
					11	0.09	83
					14	0.09	83
					21	0.07	83
					29	0.07	83
					70	0.04	83
Hungary	10% EC	2	0.1	0.01	0	0.19	30
1984					1	0.16	30
					3	0.12	30
					8	0.06	30
					14	0.02	30
					21	0.01	30
					28	<0.005	30
					35	<0.005	30
USA/82	2.4 EC	1	0.45	0.024	21	0.11	4
USA/83	2.4 EC	2	0.22 0.11	0.021 0.011	25 25	0.14 0.10	T-5952*
USA/84	2.4 EC	4	0.22	0.022	21	0.37	T-6077*
USA/84	2.4 EC	4	0.22	0.015	21	0.75	T-6078*
USA/84	2.4 EC	2	0.22	0.009	14	0.57	T-6079*
USA/84	2.4 EC	1	0.22	0.024	95	0.22	T-6081*

Country, Year	Application				PHI, days	Residue, mg/kg	Ref.
	Form.	No.	kg ai/ha	kg ai/hl			
USA/85	2.4 EC	4	0.22	0.022	1	0.42	T-6409*
					7	0.65	
					14	0.44	
					21	0.52	
					28	0.51	
					35	0.83	
USA/85	2.4 EC	4	0.056	0.003	21	0.11	T-6410*
		4	0.11	0.006	21	0.13	
		4	0.22	0.012	21	0.45	
		4	0.45	0.024	21	1.2	
USA/85	2.4 EC	1 2	0.22 0.22	0.012 0.012	21 21	0.28 0.58	T-6411*
		3	0.22	0.012	21	0.89	
		4	0.22	0.012	21	1.5	
USA/85	2.4 EC	4	0.22	0.022	21	0.74	T-6412*
USA/85	2.4 EC	4	0.22	0.016	21	3.1	T-6413*
USA/85	2.4 EC	1	0.22	0.024	1	2.3	T-6414*
					7	2.0	
					14	1.7	
					21	1.4	
					28	0.5	
					35	0.5	
USA/85	2.4 EC	4	0.22	0.024	1	1.1	T-6415*
					7	0.91	
					14	0.67	
					21	1.1	
					28	0.73	
					35	0.90	
USA/85	2.4 EC	4	0.056	0.006	21	0.15	T-6416*
		4	0.11	0.012	21	0.27	

Country, Year	Application				PHI, days	Residue, mg/kg	Ref.
	Form.	No.	kg ai/ha	kg ai/hl			
		4	0.22	0.024	21	1.1	
		4	0.45	0.048	21	2.6	
USA/85	2.4 EC	1	0.22	0.024	21	0.21	T-6417*
		2	0.22	0.024	21	1.3	
		3	0.22	0.024	21	1.5	
		4	0.22	0.024	21	1.5	
USA/86	2.4 EC	4	0.22	0.024	19	1.1	T-6725*
USA/86	2.4 EC	4	0.22	0.024	14	1.4	T-6726*
USA/86	2.4 EC	4	0.22	0.022	21	1.0	T-6728*
USA/86	2.4 EC	4	0.22	0.024	21	1.4	T-6731*
USA/86	2.4 EC	4	0.22	0.024	21	5.6	T-6829*
USA/86	2.4 EC	4	0.41	0.023	1	0.99	T-6835*
					7	2.6	
					14	1.7	
					21	1.3	
					28	1.2	
					35	2.0	
USA/90	2.4 EC	4	0.22	0.098	21	0.81	T-7544**
USA/90	2.4 EC	4	0.22	0.094	21	0.53	T-7545**
		4	0.45	0.192	21	0.84	

* These "T"references are subdivisions of Fujie, 1990c (No. 25)
** These "T"references are subdivisions of Fujie, 1992 (No. 26)

Cotton seed. The available results are shown in Table 5.

Table 5. Residues of fenpropathrin in cotton seed in the USA. All trials with 2.4 EC.

Year	Application			PHI, days	Residue, mg/kg	Ref.
	No.	kg ai/ha	kg ai/hl			
1975	11	0.22	0.24	20	0.03	75
1975	11	0.22	0.24	20	0.02	76

Year	Application			PHI, days	Residue, mg/kg	Ref.
	No.	kg ai/ha	kg ai/hl			
	8	0.11	0.24	38	<0.01	57
1975	8	0.22	0.47	38	<0.01	57

Year	Application			PHI, days	Residue, mg/kg	Ref.
	No.	kg ai/ha	kg ai/hl			
	17	0.11	0.24	44	0.03	2
1975	17	0.22	0.47	44	0.07	2
	17	0.44	0.94	44	0.02	2
1975	8	0.22	0.47	22	<0.01	84
	3	0.28	0.6	62	<0.05	74
1974	3	0.56	1.6	62	<0.05	74
	3	0.28	0.6	62	<0.01	41
1974	3	0.56	1.2	62	<0.01	41
	8	0.22	0.079	35	<0.01	T-6023*
1983	8	0.45	0.16	35	<0.01	
1983	8	0.11	0.094	33	<0.01	T-6024
1984	10	0.22	0.47**	18	<0.01	T-6069
1984	9	0.22	0.24	21	0.02	T-6070
1984	10	0.22	0.24	18	0.03	T-6071
1984	10	0.22	0.24	21	<0.01	T-6072
1984	10	0.22	variable***	21	0.01	T-6073
1984	11	0.22	0.094	34	0.26	T-6074
1984	10	0.22	0.22	21	<0.01	T-6075
1984	10	0.22	0.24	20	0.29	T-6076
	10	0.22	0.079	3	0.13	T-6418
1985	10	0.15		10	0.15	
	14	0.03		14	0.03	
	21	0.12		21	0.12	
	28	<0.01		28	<0.01	
	35	<0.01		35	<0.01	
	10	0.11	0.039	21	0.02	T-6419
1985	10	0.22	0.079	21	0.01	
	10	0.45	0.161	21	0.03	
1985	10	0.22	0.24**	21	0.02	T-6420

Year	Application			PHI, days	Residue, mg/kg	Ref.
	No.	kg ai/ha	kg ai/hl			
1985	10	0.22	0.47**	21	<0.01	T-6421
1985	10	0.22	0.52	21	0.08	T-6422
1985	10	0.22	0.47	3	3.3	T-6423
				7	0.76	
				14	0.20	
				21	0.02	
				28	0.01	
				35	<0.01	
1985	10	0.22	0.24	21	<0.01	T-6424
1985	10	0.22	0.24	22	<0.01	T-6425
1985	10	0.22	0.47	30	0.01	T-6426
	10	0.11	0.12	21	0.09	T-6427
1985	10	0.22	0.24	3	0.52	
				7	0.47	
				14	0.36	
				21	0.32	
				36	0.31	
	10	0.45	0.48	7	1.20	
1986	8	0.22	0.08	21	1.0	T-6715
1986	8	0.22	0.08	20	0.53	T-6716
1986	8	0.22	0.24	21	0.07	T-6717
1986	8	0.22	0.47	21	0.07	T-6718
1987	8	0.22	1.18**	21	0.27	T-6967
1987	8	0.22	1.18**	21	<0.01	T-6968
1989	5	0.34	0.73	21	0.02	T-7376
1989	5	0.34	0.073	21	0.01	T-7377
1989	5	0.34	0.125	20	0.28	T-7378
1989	5	0.34	0.073	21	0.06	T-7379
		0.34	0.73	21	0.06	T-7380

Year	Application			PHI, days	Residue, mg/kg	Ref.
	No.	kg ai/ha	kg ai/hl			
1989	5	0.34	3.64**	21	0.03	T-7381
1989	5	0.34	3.64**	21	0.04	T-7382

* The "T" references are subdivisions of Fujie, 1990a
** Aerial application
*** The volumes applied varied from 430 to 700 l/ha

Gherkins. The data on gherkins shown in Table 6 were developed in greenhouse trials.

Table 6. Residues of fenpropathrin in greenhouse gherkins

Country, Year	Application				PHI, days	Residue, mg/kg	Ref.
	Form.	No.	kg ai/ha	kg ai/hl			
Denmark	5% EC	1		0.0075	1	0.05	42
1984					2	0.05	
					4	0.01	
					7	<0.01	
Germany	10% EC	3	0.08	0.009	0	0.04	93
1983					1	0.03	
					3	<0.01	
					5	0.01	
					7	0.01	
Germany	10% EC	3	0.08	0.009	0	0.10	94
1983					1	<0.01	
					3	<0.01	
					5	<0.01	
					7	<0.01	
Germany	10% EC	3	0.08	0.009	0	0.06	95
1983					1	0.07	
					3	0.02	
					5	0.03	
					7	0.02	
Germany	10% EC	3	0.08	0.009	0	0.08	96
1983					1	0.15	
					3	0.10	
					5	0.07	
					7	0.03	

Egg plants. The results in Table 7 were submitted by Spain.

Table 7. Residues of fenpropathrin in egg plants, reference 97, all trials with 10% EC. No information on year.

Country,	Application	PHI,	Residue,

Year	No.	kg ai/hl	days	mg/kg
Japan	3	0.01	1	0.12
			3	0.04
			7	0.005
Japan	3	0.01	1	0.19
			3	0.16
			7	0.09
Japan	5	0.01	1	0.12
			3	0.04
			7	0.06
Japan	5	0.01	1	0.18
			3	0.16
			7	0.07
France	1	0.015		fruit
			0	0.07
			7	0.06
			14	<0.01
			21	<0.01
				pulp
			0	<0.01
			7	<0.01
			14	<0.01
			21	<0.01
France	2	0.015		fruit
			0	0.08
			7	0.01
			14	<0.01
			21	<0.01
				pulp
			0	<0.01
			7	<0.01
			14	<0.01
			21	<0.01

Sweet peppers. The results of 7 Spanish trials, 4 Japanese trials and 1 trial from Denmark are summarized in Table 8.

Table 8. Residues of fenpropathrin in sweet peppers, reference 97, all trials with 10% EC.

Country year	Application	PHI, days	Residue, mg/kg

	No.	kg ai/ha	kg ai/hl		
Japan*	3		0.01	1	0.91
outdoor				3	0.86
				7	0.32
Japan*	5		0.01	1	0.88
outdoor				3	0.58
				7	0.42
Japan*	3		0.01	1	0.92
outdoor				3	0.68
				7	0.24
Japan*	5		0.01	1	1.18
outdoor				3	0.76
				7	0.48
Denmark*	1		0.0075	0	0.13
outdoor				2	0.16
				5	0.21
				6	0.17
				13	0.12
Spain	3	0.18	0.01	0	0.30
1986				4	0.20
outdoor				7	0.08
				14	0.03
Spain	3	0.18	0.01	0	0.20
1986				4	0.170
outdoor				7	0.07
				14	0.04
Spain	3	0.18	0.01	0	0.28
1986				4	0.17
outdoor				7	0.07
Spain	1	0.15	0.01	2	0.52
1988				7	0.04

Country year	Application			PHI, days	Residue, mg/kg
	No.	kg ai/ha	kg ai/hl		
indoor				11	0.16
Spain	1	0.15	0.01	2	0.38
1988				7	0.25
indoor				11	0.45
				15	0.17
Spain	1	0.15	0.01	2	0.39
1988				7	0.15
indoor				11	0.15
				15	0.21
Spain	1	0.15	0.01	2	0.34
1988				7	0.38
indoor				11	0.11
				15	0.15

* No information on year. Trials were submitted by Spain.

Tomatoes. The results of trials from Denmark and Germany are summarized in Table 9.

Table 9. Residues of fenpropathrin in tomatoes.

Country, Year	Application				PHI, days	Residue, mg/kg	Ref.
	Form.	No.	kg ai/ha	kg ai/hl			
Denmark	5% EC	1		0.0075	1	0.08	42
1984					3	0.08	
Indoor					5	0.17	
					7	0.09	
					14	0.05	
Germany	10% EC	3	0.08	0.009	0	<0.01 <0.01	85;1*
1983					1	<0.01 <0.01	
Indoor					3	<0.01 <0.01	
					5	0.01 0.01	

Country, Year	Application				PHI, days	Residue, mg/kg	Ref.
	Form.	No.	kg ai/ha	kg ai/hl			
					7	0.03 0.03	
Germany	10% EC	3	0.08	0.009	0	0.26 0.21	86;1
1983					1	0.28 0.22	
Indoor					3	0.19 0.15	
					5	0.51 0.41	
					7	0.07 0.06	
Germany	10% EC	3	0.08	0.009	0	0.12 0.12	87;1
1983					1	0.18 0.19	
Indoor					3	0.16 0.17	
					5	0.13 0.14	
					7	0.22 0.23	
Germany	10% EC	3	0.08	0.009	0	0.11 0.11	88;1
1983					1	0.09 0.09	
Indoor					3	<0.01 <0.01	
					5	0.02 0.02	
					7	0.03 0.03	
Germany	10% EC	3	0.08	0.009	0	0.08 0.07	89;1
1983					1	0.21 0.18	
Indoor					3	0.17 0.15	
					5	0.08 0.07	
					7	0.08 0.07	
Germany	10% EC	3	0.08	0.009	0	0.73 0.73	90;1
1983					1	0.56 0.56	
Indoor					3	0.58 0.58	
					5	0.47 0.47	
					7	0.22 0.22	
Germany	10% EC	3	0.08	0.009	0	0.19 0.15	91;1
1983					1	0.29 0.23	
Indoor					3	0.46 0.37	
					5	0.49 0.39	
					7	0.30 0.24	

Country, Year	Application				PHI, days	Residue, mg/kg	Ref.
	Form.	No.	kg ai/ha	kg ai/hl			
Germany	10% EC	3	0.08	0.009	0	0.19 0.15	92;1
1983					1	0.17 0.13	
Indoor					3	0.13 0.10	
					5	0.13 0.10	
					7	0.08 0.06	
Hungary	10% EC	1	0.05	0.005	0	0.01	18
1984					1	0.07	
Indoor					2	0.03	
					3	0.03	
					4	0.04	
					7	0.02	
Hungary	10% EC	1	0.05	0.005	0	0.04	19
1984					1	0.05	
Indoor					2	0.01	
					3	0.07	
					4	0.04	
					7	0.02	
Japan	10% EC	3	0.25	0.01	1	0.58	34
1986					3	0.60	
Indoor					7	0.58	
Japan	10% EC	5	0.25	0.01	1	1.1	34
1986					3	0.86	
Indoor					7	0.74	
Japan	10%EC	3	0.25	0.01	1	0.42	34
1986					3	0.37	
Indoor					7	0.25	
Japan	10% EC	5	0.25	0.01	1	0.67	34
1986					3	0.60	
Indoor					7	0.55	
Germany	10% EC	3		0.009	0	0.15	97
Outdoor					1	0.23	

Country, Year	Application				PHI, days	Residue, mg/kg	Ref.
	Form.	No.	kg ai/ha	kg ai/hl			
**					3	0.37	
					5	0.39	
					7	0.24	
Germany	10% EC	3		0.009	0	0.15	97
Outdoor					1	0.13	
**					3	0.1	
					5	0.1	
					7	0.06	
Germany	10% EC	3		0.009	0	0.12	97
Outdoor					1	0.19	
**					3	0.17	
					5	0.14	
					7	0.23	
Germany	10% EC	2		0.009	0	<0.01	97
Outdoor					1	<0.01	
**					3	<0.01	
					5	0.01	
					7	0.03	
Germany	10% EC	2		0.009	0	0.21	97
Outdoor					1	0.22	
**					3	0.15	
					5	0.41	
					7	0.06	
Denmark	10% EC	1		0.0075	1	0.08	97
Outdoor					3	0.08	
**					5	0.17	
					7	0.09	
					14	0.05	
Italy** Outdoor	10% EC	2		0.015	21	0.01	97

* From two independent laboratories

** No information on year. Trials used and submitted by Spain.

Animal transfer studies

Cattle. Lactating dairy cattle were administered fenpropathrin at rates equivalent to 25, 75 and 250 ppm in the feed based on the daily average food consumption of 18.54 kg/cow. The fenpropathrin was technical grade of 92.5% purity, unlabelled, and administered in gelatin capsules. There were four cows in each group and two as controls. Milk samples were taken periodically up to 28 days, when three animals of each group and one of the controls were slaughtered. Administration of fenpropathrin then ceased and the remaining animals were killed after a three-day period on untreated feed. Samples of liver, kidney, fat and muscle were collected for analysis from all of the animals. Residues of fenpropathrin itself in the milk reached a plateau after three days. Average residues in the whole milk of the four cows of each group were 0.04, 0.17, and 0.33 mg/l for the three dose levels. On the 28th day, these levels were 0.04, 0.13 and 0.32 mg/l. At the end of the three-day depuration period, residues had fallen to <0.01, 0.02 and 0.04 mg/l for the three levels. In a bulk pasteurized milk sample from the high-dose cows on the 26th and 27th days containing 0.25 mg/l in the whole milk residues were largely confined to the cream where the level reached 3.7 mg/l.

Levels of fenpropathrin in the tissues at terminal slaughter and after the depuration period (average of three cows) are shown in Table 10.

Table 10. Levels of fenpropathrin in cattle tissues following oral ingestion

Level in feed	Residues, mg/kg			
mg/kg	muscle	kidney	liver	fat
After 28 days on treated feed				
25	0.02	0.03	<0.01	0.33
75	0.06	0.04	<0.01	1.0
250	0.20	0.16	0.01	3.8
After three-day depuration period				
25	0.01	0.01	<0.01	0.31
75	0.10	0.06	<0.01	0.83
250	0.12	0.14	<0.01	2.6

Samples of milk and tissues taken from the cows at the highest feeding level killed after 28 days were analyzed for TMPA, PBacid and PBacid-glycine; measurable levels were not found in the lower-dose groups. None of these metabolites was detected in milk or muscle. Average levels in kidney were TMPA 0.1, PBacid 0.07 and PBacid glycine 0.04 mg/kg. Corresponding levels in the liver were 0.03, 0.09 and 0.02 mg/kg. In samples taken from the single cow killed after three days depuration the values were 0.07, 0.09 and 0.04 mg/kg in kidney and <0.02, <0.02 and 0.02 mg/kg in liver, demonstrating rapid losses from liver but less so from kidney. Fat analyses were not carried out as previous studies had shown that over 90% of the residues in fat were present as the parent compound (Fujie *et al*., 1986a).

The residues in whole milk at the plateau level were approximately 0.15% of the level in the feed and were reasonably consistent between the three dose levels. If cows were fed on a diet consisting entirely of dried apple pomace at the residue level found in the processing studies of 45 mg/kg, it could be argued that the maximum level in milk would be 0.07 mg/l. Assuming that these residues would all be present in the fat and that the fat content of the milk would be 4%, such a level would be equivalent to 1.75 mg/kg in the milk fat.

Residues in body fat at the end of the study were about 1.4% of the level in the feed. Using the apple pomace figure of 45 mg/kg, it is reasonable to conclude that residues in meat fat would not exceed 0.6 mg/kg.

Residues in meat (muscle) were about 0.08% of the feed level so that animals fed on apple pomace at 45 mg/kg would not be expected to have more than 0.05 mg/kg in muscle or kidney.

It should be recognized, of course, that it is unlikely that animals would be fed on diets consisting exclusively of apple pomace so that these estimated upper limits are very unlikely to be seen in practice, especially since the apple pomace figure itself is probably a considerable overestimate of what would actually occur.

Hens. Laying hens were fed diets containing unlabelled fenpropathrin of 94.5% purity at nominal levels of 2.5, 7.5 and 25 mg/kg of the technical product for a period of 28 days. Actual average contents were 2.45, 7.10, and 23.6 mg/kg. There were 20 hens in each treatment group including the control animals. Eggs were collected daily and those from days 1, 2, 4, 7, 21 and 28 were analyzed as whole eggs minus shell. All the hens were killed after 28 days and composite samples of liver, gizzard, fat and muscle were prepared for analysis.

In all analyses the lower limit of determination for fenpropathrin was 0.01 mg/kg. Residues in all tissues except fat were below this level at the end of the study. Average levels of fenpropathrin in the fat reached 0.02, 0.05 and 0.14 mg/kg for the three feeding levels.

Residues were found in the eggs only at the highest feeding level. A level of 0.02 mg/kg was reached on the seventh day and remained essentially constant until the end of the study.

Both tissues and eggs from hens in the highest dose group were also analyzed for TMPA, PBacid, and PBacid-glycine. The limit of determination for each compound was 0.02 mg/kg and none of the metabolites was found in eggs or tissues except TMPA at 0.04 mg/kg and PBacid-glycine at 0.02 mg/kg (average of replicates), both in the liver. Fat samples were not analyzed for metabolites, since they were considered unlikely to have accumulated measurable levels of these hydrophilic compounds (Fujie *et al*., 1986b).

It is unlikely that poultry would receive feed items containing appreciable residues of fenpropathrin with the possible exception of cotton seed meal. From Table 11 it may be seen that with a maximum level of 1 mg/kg in raw cotton seed, it is unlikely that residues in meal would exceed 0.1 mg/kg. In the present study the level in fat reached only 0.02 mg/kg even at a total feed level of 2.5 mg/kg so that measurable residues would not be expected in either the meat or eggs of hens fed on cotton seed meal.

FATE OF RESIDUES

Nomenclature of metabolites (*see also* Figure 1 on following page)

3-phenoxybenzoic acid (PBacid)

3-phenoxybenzyl alcohol (PBalc.)

3-phenoxybenzaldehyde (PBald.)

2,2,3,3-tetramethylcyclopropanecarboxylic acid (TMPA)

2-hydroxymethyl-2,3,3-trimethylcyclopropanecarboxylic acid (TMPA-CH_2OH)

5-hydroxymethyl-6,6-dimethyl-3-oxabicyclo[3.1.0]hexan-2-one (TMPA-CH_2OH-lactone)

In animals

Goats. Fenpropathrin labelled with ^{14}C in the benzyl ring or the C-1 position of the cyclopropyl ring was administered to lactating goats at a nominal rate of 50 mg/kg feed/day for five days. The animals were slaughtered and samples of kidney, liver, heart, loin and rear leg muscle and omental and perirenal fat taken within 4 hours of the last dose. The goats were milked every morning and evening and all milk was reserved for analysis.

The total radioactivity in the milk reached a steady state by the evening milking on the third day when average residues in the whole milk were 0.11 mg/l for the cyclopropyl label and 0.25 mg/l for the benzyl label, all expressed as fenpropathrin. Less that 3% of the activity in the milk was found in the butterfat. Of the total administered activity of 250 mg of fenpropathrin, 0.73 mg of fenpropathrin equivalent was recovered in the milk from the benzyl label and 0.43 mg from the cyclopropyl label (averages for the two goats in each treatment), so that milk represented only a minor route of excretion.

Figure 1. Metabolism and metabolic degradation of fenpropathrin in soils and plants.

L, photodegradation; S, soil; P, plant

*, ■, ⅄, 14C-labelled positions

The retention in body tissues was only moderate: a total of 2.8 mg of fenpropathrin equivalents for the benzyl label and 3.7 mg for the cyclopropyl label as compared with the total administered amount of 250 mg. Most of the retained activity was found in the liver, kidney and fat. Levels in these three organs were in the range of 0.4-0.7 mg/kg for both labels. Muscle levels were in the range of 0.02-0.04 mg/kg (Ku and Doran, 1990a).

Between 20% and 40% of the radiocarbon in the milk from animals receiving the benzyl label was associated with the parent compound, with nearly all of the remainder being present as the glycine conjugate of PBacid which reached levels of 0.03-0.15 mg/l. There were minor amounts of the hydroxylated derivatives of PBacid (0.003-0.01 mg/kg) and also of fenpropathrin itself (0.02-0.12 mg/kg). With the cyclopropyl label, 56 75% of the activity was associated with the parent material with moderate amounts of TMPA and its hydroxymethyl (<0.002-0.003 mg/kg), carboxy (<0.002-0.003 mg/kg) and lactone (<0.002-0.003 mg/kg) derivatives. In this case, however, the total recovery was only about 70-80% and the concentration of TMPA and all its derivatives did not exceed 0.01 mg/l.

The identity of the compounds associated with the radioactivity in the tissues was somewhat similar except that in the liver and kidney there were only traces left as parent material. In the case of the benzyl label, most of the activity was in the form of PBacid (kidney 0.05-0.08 mg/kg; liver 0.03-0.06 mg/kg) and its glycine conjugate (kidney 0.21-0.38 mg/kg; liver 0.06-0.11 mg/kg) and in the case of the cyclopropyl label TMPA and its derivatives predominated. The hydroxymethyl TMPA lactone (TMPA CH_2OH lactone) was practically absent from the fat but was prominent in muscle, liver and kidney, accounting for up to 40% of the activity in the kidney, equivalent to about 0.2 mg/kg (Ku and Doran, 1990a).

Cows. Two lactating cows were fed on a diet containing 0.11 mg/kg of fenpropathrin labelled in the benzyl ring with ^{14}C. The cows were milked twice daily and all milk, faeces and urine monitored for radioactivity. After 21 days the animals were slaughtered and radioactivity measured in muscle, fat and liver.

It was found that the equilibrium between intake and excretion in the urine and faeces was reached after about 5 days and that excretion thereafter averaged 96% of the amount ingested. No radioactivity was detected in any of the muscle, blood or fat samples; the limit of determination varied from 0.004 to 0.008 mg/kg of fenpropathrin equivalent. Residues in the milk samples were extremely small and very difficult to measure. The author estimated them to be between 0.0002 and 0.0003 mg/l of fenpropathrin equivalent (Crayford, 1975).

Hens. Fenpropathrin, labelled in either the cyclopropyl or the benzyl ring was administered to laying hens daily for 10 days. The product was given in the form of capsules at a nominal rate of either 0.5 or 5 mg/kg body weight. There were four treatment groups of 10 hens in each group and two control groups. Eggs were collected every morning and evening and excreta every morning. The hens were all killed within four hours of the last dose and kidneys, liver, heart, gizzard (and contents), ovaries, muscle and skin were retained for analysis.

The total doses for the four groups over the ten-day period were as follows:

Low dose, benzyl label 78 mg
High dose, benzyl label 820 mg
Low dose, cyclopropyl label 75 mg
High dose, cyclopropyl label 803 mg

The recovery of total radioactivity from excreta, eggs and tissues was between 75 and 82% of the total applied dose. Between 98.9 and 99.6% of the recovered activity was found in the faeces irrespective of the label. Approximately 0.05% of the applied benzyl label was

found in the eggs and 0.2% of the cyclopropyl label. At about the 6th or 7th day of the study residue levels in the eggs reached a plateau of about 0.023 and 0.22 mg/kg fenpropathrin equivalent for the two doses of the benzyl label and about 0.05 and 0.4 mg/kg for those of the cyclopropyl label. In the body tissues the highest levels of radioactivity were found in the kidney and gizzard followed by the liver, showing about 3-4 mg/kg for the two high doses in the kidney and gizzard and 1.5-3 mg/kg in the liver without major differences between the two labels. Levels in the low-dose hens were about one-tenth of those in the higher dosed birds (Ku and Doran, 1990b).

The products associated with the activity in the solvent extracts from the benzyl label were mainly PBacid, 4-OH PBacid and its glycine conjugate, and 3-hydroxybenzoic acid (3-OH-Bacid), which was not encountered in the other animal studies. Only negligible amounts of the activity in the liver and kidney remained as the parent (1-2%) but fat residues contained nearly 50% of the unchanged compound. The occurrence of 3-OH-Bacid in the kidney, accounting for 35% of the kidney activity, demonstrates that cleavage of the ether linkage of PBacid must have occurred. It also appears that 4′-hydroxylation of PBacid occurred readily in the liver and kidney. Nevertheless, even in the case of the high-dose birds, residues of any single component seldom exceeded 1 mg/kg except 3-OH-Bacid and 4′-OH-PBacid in the kidneys. The pattern of distribution of activity among the metabolites was similar in the case of the cyclopropyl labelled group except that the main metabolites, as would be expected, were TMPA and its derivatives. There were mainly TMPA CH2 OH the carboxy compound (TMPA COOH), and TMPA CH_2OH lactone. Fenpropathrin was only a very minor component of the residues in the liver, kidney, heart and meat but reached 63% of the residues in fat. In the high-dose group with this label no component exceeded 1 mg/kg except TMPA itself in the kidney.

As would be expected from the comparatively high proportion of 3-OH-Bacid in the kidney, this metabolite accounted for a major proportion of the activity in the excreta from the benzyl-labelled group and this together with 4′-OH PBacid accounted for nearly 65% of the excreted activity on the eighth day. Fenpropathrin constituted only about 10% of the excreted activity. Most of the remainder was made up of PBacid and its glycine conjugate with small amounts of OH-fenp. In the case of the cyclopropyl label, TMPA and its derivatives (TMPA CH^2OH, TMPA COOH and TMPA CH_2OH lactone) accounted for half of the activity, the rest being made up of the parent and two of its hydroxylated derivatives, 4′-OH-fenp. and fenp.-CH_2OH (Ku and Doran, 1990b).

In plants

The degradation of fenpropathrin in plants has been studied in cotton, tomatoes, beans, and apples. The compounds involved are shown in Figure 1. The general pattern of degradation in all the plant studies has been rupture of the ester linkage to produce 3-phenoxybenzoic acid (PBacid) and the corresponding alcohol (PBalc.) and aldehyde (PBald.). From the acid side of the molecule, the main metabolite is 2,2,3,3-tetramethylcyclopropanecarboxylic acid (TMPA). The position is complicated, however, by subsequent hydroxylation either of these fragments or of the intact molecule. Thus, TMPA can give rise to 2 hydroxymethyl-2,3,3-trimethylcyclopropanecarboxylic acid (TMPA-CH_2OH) and 5-hydroxymethyl-6,6-dimethyl-3-oxabicyclo[3.1.0]hexan-2-one (TMPA-CH_2OH lactone). PBacid can be hydroxylated at the 4′ position and the parent molecule at various positions on the phenoxy ring to produce, for example, Æ-cyano-3-(2′- or 4′-hydroxyphenoxy)benzyl-2,2,3,3-tetramethylcyclopropanecarboxylate (2′ or 4′-OH-Fenp.).

The results of the studies on individual plants are summarized under separate headings below.

Cotton. The degradation of fenpropathrin in cotton was shown to follow the familiar pattern of ester hydrolysis and conjugation of the resulting PBacid and TMPA. In these studies, cotton plants were grown either in the greenhouse in the UK or outdoors in boxes in Spain. Known amounts of fenpropathrin (740-2000 ìg), labelled with ^{14}C in either the benzyl or cyclocyclopropyl rings were applied at various times to the leaves or bolls of the plants (total amounts 2000 and 4690 ìg). In separate outdoor experiments in Spain, only soils were treated in order to examine uptake.

The plant parts were extracted with acetonitrile/water and the main products in the extracts were found to be fenpropathrin itself with small amounts of TMPA and PBacid together with some polar material which, from the evidence presented, is likely to have consisted primarily of conjugates of either PBacid or TMPA. In the leaves at harvest (the interval between treatment and harvest was 66 days for the benzyl label and 111 days for the cyclocyclopropyl label) the total remaining activity included 70% parent in the case of the cyclocyclopropyl label and 55% parent in the case of the benzyl label. Most of the remaining activity was probably accounted for by PBacid and TMPA, mainly in conjugated forms.

Examination of the plants grown on soils treated with 0.5 kg/ha of fenpropathrin showed only extremely low uptake of radioactivity, demonstrating very limited tendency for translocation. See Table 11 (Hitchings and Roberts, 1977).

Table 11. Analyses of cotton and soil treated with [^{14}C]-fenpropathrin.

Sample	Radioactivity, mg/kg fenpropathrin equivalent			
	benzyl label		cyclopropyl label	
	extracted	unextracted	extracted	unextracted
soil at applicn.	0.86	0.03	0.78	0.034
soil at harvest	0.02	0.09	0.1	0.048
cotton leaves	0.002	0.003	0.014	0.004
cotton stems	0.004	0.01	0.017	0.01
cotton boll case	0.01	0.02	<0.01	0.01
cotton lint	-	0.01	-	0.02
cotton seed kernel	-	0.03	-	0.05
cotton seed hull	-	0.01	-	0.01

Tomatoes. In studies in California by the Chevron Chemical Company, tomato plants were treated four times with fenpropathrin (0.224 kg ai/ha) labelled with ^{14}C in either the cyclopropyl or the benzyl ring. Fruit and leaves were extracted at harvest (PHI 19 days) with a variety of solvents and the components of the residue characterized. In the fruit the residue was too low to allow full characterization, but some two thirds was present as unchanged fenpropathrin with a further 28% as conjugated metabolites. In the leaves, only 30% of the total residue was present as parent and just under 60% as conjugated metabolites. In the case of the benzyl label, the most prominent metabolites were conjugates of PBacid and its 4′-hydroxy derivative (4′-OH-PBacid), although these only constituted a minor proportion of the total residue. The main metabolites reported in the case of the cyclocyclopropyl label were conjugates of TMPA and hydroxymethyl-TMPA

(TMPA-CH_2OH), part of which was also conjugated (Chen and Abell, 1985, 1986b).

Beans. Somewhat similar results were reported by the same authors for pinto beans. The residue after a PHI of 15 days in the beans themselves was too low (0.07 mg/kg) for full characterization; some 93% of the label remaining in the plants at harvest was found in the leaves. In the leaves, 46-47% of the remaining activity consisted of the parent compound. In the case of the benzyl label the main metabolites were conjugates of PBacid and 4′-OH-PBacid, together with conjugates of PBalc. and PBald. In the case of the cyclocyclopropyl label, the main metabolites were conjugates of TMPA and its two stereoisomeric mono-hydroxy derivatives (TMPA-CH_2OH) (Chen and Abell, 1985, 1986c).

Apples. In a study on apples samples from young trees were analyzed at harvest, 14 days after the last of three treatments at the comparatively high rate of approximately 0.448 kg/ha. Practically all of the residue found in the fruit (92-94%) was present as the parent compound. The parent compound was also the major component found in the rest of the plant (61-66% of the total activity). In apples the pattern of metabolites was more complex than in the preceding crops, although few individual metabolites accounted for more than 2% of the total residue. The most prominent metabolite from the benzyl label was conjugated PBalc. and from the cyclopropyl label TMPA, together with the usual complement of hydroxylated derivatives. Both labelled compounds also yielded small amounts of hydroxylated derivatives of intact fenpropathrin (2′- or 4′-OH-fenp.), existing in both free and conjugated forms (Chen and Abell, 1985, 1986a.)

Photodegradation on leaf surfaces. The four top leaves of small potted mandarin orange plants were treated with fenpropathrin labelled in the cyano group or the cyclopropyl or benzyl ring, to produce a deposit of 1.1 ì g/cm^2. After a 14-day exposure to sunlight the leaves were assayed for radioactivity. There were substantial evaporative losses but over 80% of the remaining activity (approximately 40% of that applied) was still in the form of the unchanged parent. There were little more than traces of degradation products, among which were identified CONH2 -fenp. and PBacid with a small amount of Æ-cyano-3-hydroxybenzyl 2,2,3,3-tetramethylcyclopropanecarboxylate. None of these reached more than 0.3% of the applied activity.

It is noteworthy that approximately 40% of the activity remaining on the leaves could be extracted with a surface wash; the rest had penetrated into the leaf (Takahashi *et al*., 1983, 1985).

Metabolites in samples from supervised field trials. Some of the fruit samples from supervised field trials were analyzed for PBacid and TMPA. The results are shown below in Table 12. As will be seen, PBacid did not reach the level of determination in any of the samples analyzed. TMPA slightly exceeded detectable levels in pears, but still constituted only a negligible proportion of the total residues. These results confirm that residues in these crops resulting from treatment with fenpropathrin are adequately determined by analysis for fenpropathrin alone.

Table 12. The occurrence of metabolites in fruits treated with fenpropathrin; residues in mg/kg.

Parent	PBacid	TMPA	Reference
Apples			
3.6	<0.02	<0.02	24
1.5	<0.02	<0.02	24
3.7	<0.02	<0.02	24

Parent	PBacid	TMPA	Reference
2.8	<0.02	<0.02	24
2.4	<0.02	<0.02	24
Pears			
1.0	<0.02	0.04	37
3.2	<0.02	0.07	37
1.1	<0.02	0.03	22
2.9	<0.02	0.06	22
1.8	<0.02	0.04	22
2.4	<0.02	0.04	22
Grapes			
3.1	<0.02	<0.02	25
1.1	<0.02	<0.02	25
1.1	<0.02	<0.02	25
2.6	<0.02	<0.02	25
1.1	<0.02	<0.02	25
1.4	<0.02	<0.02	25
1.0	<0.02	<0.02	25
5.6	<0.02	<0.02	25

Summary of degradation in crops. The above studies demonstrate that fenpropathrin itself was the primary component of the residues in the fruits of the plants, but degradation products constituted the greater part of the residues present in the leaves. Breakdown products in both fruits and leaves did not differ greatly from those in animals.

This conclusion is supported by the data collected in some of the supervised field trials for PBacid and TMPA which were either below the limit of determination of the method or negligible compared with the levels of parent fenpropathrin. It is therefore considered that crop residues are described adequately by defining them as the parent product alone.

It is also evident that any uptake of residues from the soil is too slow for detectable residues to occur in suceeding crops, especially in view of the comparatively short persistence of the compound in soils as shown in the following section.

In soil

Fenpropathrin readily disappears from soil by two main mechanisms, biodegradation and photochemical degradation of surface deposits. It is relatively immobile because of its strong adsorption and its comparatively short life in normal agricultural soils. The nomenclature of the compounds involved in the degradation pathways is as in Figure 1 and the above text.

Degradation. In the first of the soil degradation studies, 5-6 g of a single soil were treated with fenpropathrin labelled either in the cyano group, the C-1 position of the cyclopropyl

ring or uniformly in the benzyl ring. The soils were then perfused with nutrient solutions buffered to pH 7.0 for 148 or 208 days depending on the label. Liberated carbon dioxide was collected periodically for estimation of radioactivity. The soils and perfusates were solvent-extracted at the end of the study and the extracts analyzed for radioactivity and fractionated by TLC. The unextracted activity was determined by combustion.

Table 13 shows the recoveries obtained from the three phases expressed as a percentage of the activity applied (average of two duplicate perfusions).

Table 13. Recovery of radioactivity in soil perfusion studies.

Phase	% of applied activity		
	CN label	Cyclopropyl label	Benzyl label
Carbon dioxide	36.5	19.5	4.7
Perfusate	9.0	10.5	8.0
Soil extract	14.0	32.5	34.0
Combusted soil	7.0	17.2	22.1
Total recovery	66.5	79.7	68.8

As will be seen, the production of labelled carbon dioxide was greatest from the cyano label and least from the benzyl, indicating the relative readiness of the different parts of the molecule to mineralize although the soil with the cyano label was perfused for 208 days compared with only 148 days for the other two labels. Much of the difference in the production of carbon dioxide was made up by the difference in the total amount of activity retained in the soils.

In the soil extracts from the cyclopropyl label, the parent compound was the main component with small amounts of tetramethylcyclopropanecarboxylic acid (TMPA) and unidentified polar compounds and, in one of the perfusions, the amide of fenpropathrin ($CONH_2$-fenp.). No amide was found from the cyano label. In the case of the benzyl label, the liberation of carbon dioxide was much slower and the rates were erratic. Neither $CONH_2$-fenp. nor PBacid was detected in the soils. In the perfusates, the main components isolated were the parent and unidentified hydrophilic compounds (Noble, 1976).

The degradation of fenpropathrin labelled in either the benzyl or cyclopropyl rings was further studied in a sandy clay soil (Brenes) or a clay (Los Palacios) from Spain or a sandy loam from the UK (Leiston). The soils were treated in glass jars at a rate of 2.86 mg/kg and kept in the dark at a temperature between 23 and 25°C. The moisture contents of the soils were maintained at their original values by the periodic addition of distilled water but in the case of Brenes soil the study was conducted at two moisture levels, 6 and 16%. An additional study using Brenes soil was set up to determine volatiles. Benzyl-labelled fenpropathrin was added to the soil at a rate of 2.5 mg/kg at a moisture content of 19.6%. The whole system was aerated continuously and volatiles and carbon dioxide were absorbed in traps.

In a separate study Leiston soil, in conical flasks, was treated at the rate of 2.5 mg/kg with benzyl- or cyclopropyl-labelled fenpropathrin and stored at 25°C under distilled water with occasional nitrogen purging to maintain anaerobic conditions.

In the aerobic study soils were sampled at 4, 8 and 16 weeks and in the volatiles study at 26 weeks. In the anaerobic study, the soils were sampled at intervals of 32, 60, 120 and 160 days. In all cases they were extracted with acetonitrile/water (7:3). In the volatiles

study, the aspirated air was sampled at intervals throughout the experiment. Unextracted residues were determined in the soils after the solvent extraction. Degradation products were identified by co-chromatography, the carboxylic acids after methylation.

In the aerobic soil after 16 weeks, by far the most important component of the residue was the parent compound but the proportion of the original ^{14}C remaining varied greatly, ranging from 66% in Brenes soil at 6% moisture to 10.2% in Leiston at 16% moisture. Although the moisture capacities of the soils were not stated it would appear that the main factor influencing the degree of degradation was the dryness of the soils i.e. the moisture content in relation to the moisture capacity. The half-life of the fenpropathrin was about 4 weeks on moist soils (Leiston and moist Brenes) but more than 16 weeks on the drier ones (dry Brenes and Los Palacios).

In the drier soils after 16 weeks the most prominent metabolite was PBacid in the case of the benzyl label and TMPA in the case of the cyclopropyl label. The other two metabolites found were $CONH_2$-fenp. and COOH-fenp., but these were scarcely detectable in the soils with high moisture (Brenes at 16% moisture and Leiston). In those two soils PBacid was also much lower but unextracted activity was much higher than in the drier soils, suggesting either entrapment in the soil organic matter or possibly mineralization and incorporation in the soil organic matter pool.

In the study using Brenes soils to detect volatile activity the percentage of the applied radioactivity evolving as CO_2 after 26 weeks was 16.0, with 71.8% as the intact parent compound and 11.6% as unextracted activity. Other degradation products were only present at negligible levels.

The main effect of imposing anaerobic conditions on the soils was to slow the rate of ester hydrolysis to some extent and to impede the subsequent degradation of PBacid and TMPA which tended to accumulate. This indicates that their subsequent degradation in aerobic conditions is essentially oxidative (Roberts and Standen, 1976).

In reviewing evidence for the further degradation of metabolic products, Miyamoto concluded that PBacid, as derived from the degradation of fenpropathrin and other pyrethroids, is rapidly and completely degraded to CO_2 under aerobic conditions. There is somewhat comparable evidence for the degradation of TMPA although it is not quite so extensive as for PBacid. Anaerobic conditions impeded the degradation of both compounds (Miyamoto, 1980).

Two fresh Japanese soils, a sandy clay loam (Azuchi) and a light clay with 15% organic matter (Kodaira), were studied under three different conditions: natural aerobic and anaerobic soil, and autoclaved aerobic soil. The soils were treated with fenpropathrin labelled either in the benzyl ring or at C-1 in the cyclopropyl ring to produce a concentration of 1 mg/kg, and then incubated at 25°C. In the aerobic study with the unsterilized soil incubation was continued for 24 weeks, but in the other cases the incubation period was only 8 weeks. In the aerobic soils the moisture content was maintained at a moisture capacity of approximately 40%. Arrangements were made to trap liberated CO_2 in all cases.

Soil extracts (three extractions with methanol) were assayed at various times during the studies. Some of the soils were retained after extraction for examination of the unextracted activity.

It was found that under aerobic conditions the half-life of fenpropathrin was 11 and 17 days on the Azuchi and Kodaira soils respectively, and after 24 weeks the level of fenpropathrin had declined from 1 mg/kg to 0.025 and 0.040 mg/kg respectively (*see also* Mikami, 1983, for the basis for the calculation of these values). After 8 weeks approximately

0.85 mg/kg remained in the same two soils under anaerobic conditions and approximately 0.93 mg/kg under sterilized conditions. The corresponding figures for the natural aerobic soils after 8 weeks (mean of the two labels) were 0.05 and 0.10 mg/kg, so that anaerobic conditions retarded the degradation of fenpropathrin to a much greater degree than had been observed in anaerobic aquatic soil (Roberts and Standen, 1976). Sterilization also greatly retarded degradation, demonstrating the importance of biological processes.

Whilst there were only minor indications of degradation products in the sterile soils, at least 7 were detected in the methanol extracts of the non-sterile soils, both aerobic and anaerobic. In the aerobic soils with the cyclopropyl-labelled compound the main components of activity were the parent fenpropathrin, dephenyl-fenpropathrin (desph-fenp.), 4′-OH-fenpropathrin, and small amounts of $CONH_2$-fenp. and COOH-fenp. There were also indications of very small amounts of unidentified products. In the case of the benzyl label very low levels of PBacid were observed during the first 4 weeks. Unextractable residues at the end of the study reached 44-45% of the added activity in the Kodaira soil (the one containing a high proportion of organic matter), but only 24-32% in the lower-organic Azuchi soil. No TMPA was reported.

Under anaerobic conditions there was a similar pattern of metabolites but in a much smaller proportion as compared with the parent. Correspondingly, bound activity was also at a much lower level. There was no evidence of accumulation of PBacid, as found by Roberts and Standen.

A major part of the lost activity in the aerobic soils was recovered as labelled CO_2 in fairly similar amounts from both labels: an average between the two of 42% in the Kodaira soil and 55% in the Azuchi.

It was shown that part of the bound activity in the soils could be liberated as CO_2 by subsequent incubation of the extracted soils with fresh untreated soil but the extent of this differed greatly between the two soils. Slightly more than 20% of the bound activity was liberated from the cyclopropyl-labelled Azuchi soil whereas the corresponding figure for the Kodaira soil was only about 3% (Mikami *et al.*, 1983a).

The aerobic part of the above study (Mikami *et al.*, 1983a) was repeated at shorter time intervals. The same soils and conditions were used. The incubation period was only 30 days and soils were extracted 1, 3, 7, 17 and 30 days after treatment.

The half-lives were 30 and 33 days in the Kodaira and Azuchi soils respectively, some 2-3 times as long as in the initial study. The same degradation products were found, i.e. CO_2, desph fenp. and small amounts of 4′-OH-fenp., $CONH_2$-fenp., COOH-fenp. and PBacid. Within the short time-scale of this study the dephenyl compound increased with time in both soils. The same was true of the 4′-OH-fenp., but much less so in the Azuchi soil. The authors observed that the appreciably longer degradation time was probably due to the fact that in this study the soils had been stored for a period of 8 months and had probably suffered a reduction in bioactivity (Mikami and Sakata, 1984).

A study of the fate of 1-cyclopropyl-labelled fenpropathrin was carried out at lower temperatures in a laboratory study in The Netherlands (Van Dijk and Vonk, 1989). The soils used were a humic sand taken from Wageningen and a loam soil from Lelystad which was calcareous. Samples of 50 g of the pre-incubated soils were placed in conical flasks and fenpropathrin was added to produce a final concentration in the soils of 2.5 mg/kg. The flasks were fitted with soda lime traps to assess the liberation of labelled CO_2 and incubated at 15°C in the dark for 41 weeks. Duplicate samples were taken at intervals and extracted with methanol.

The half-life of fenpropathrin was estimated to be 6 weeks in the loam soil but

longer than 36 weeks (the estimate was 40 weeks) in the humic sand, at which point some 57% remained. In the loam soil the main conversion products were CO_2 (about 45% after 41 weeks) and unextractable bound residues (28% after 41 weeks). After 36 weeks most of the extractable fraction consisted of unchanged fenpropathrin (average 6.8%) and minor amounts of 4′-OH-fenp. and desph-fenp. (average 0.8 and 0.4% respectively). The main conversion products in the humic sand after 36 weeks were carbon dioxide (3 and 24% of the applied activity for the duplicates) and unextractable residues (14 and 16%). Most of the extractable fraction consisted of unchanged fenpropathrin but there were small amounts of 4′-OH-fenp. and desph-fenp., averaging approximately 5 and 3% respectively of the applied dose.

In both soils it was evident that the maximum levels of these two metabolites had occurred before the 36th week and in some cases well before. The same was true of the unidentified metabolites whose values seem to have been highest at 6-13 weeks and fell from 3.3% at 13 weeks to 0.7% in the loam and from 3.2% at 6 weeks to 0.9% in the sand.

The authors commented that in their work most of the pesticides they had studied were degraded only slowly in the humic sand, and noted that the soil humidity in their studies was considerably lower than that employed by Mikami *et al*. (1983a). The temperature was also a good deal lower (15°C compared with 25°C in the Mikami study). They cite another of their studies (Vonk and van Dijk, 1988) in which the half-life of fenpropathrin in the loam soil at 10°C was found to be 30 weeks. This marked dependence on temperature in the loam soil was less evident in the sand.

A study of soil degradation was carried out according to the EPA protocol, Section 162-1 of the pesticide assessment guidelines. Fenpropathrin labelled in the benzyl ring was incubated in the dark at 25°C with silt loam soil confirmed to be biologically active by plating and counting colonies. The nominal concentration of fenpropathrin was 10 ì g/g soil; the measured concentration immediately after the addition was 10.2 ì g/g. The soils were maintained at 70-75% field capacity throughout the study and sampled at intervals up to the end of the 365-day incubation. The samples were extracted with methanol (3-4 times) and then combusted to determine the unextracted activity.

After 365 days, 18.4% of the dose remained as parent with accumulated volatiles accounting for 59.9% (99.8% of which was CO_2) and unextractable residues for 17.8%. During the whole course of the study the maximum levels of the metabolites were 1.25% PBacid, 0.21% $CONH_2$-fenp., 0.55% desph-fenp., 0.19% 4′-OH-fenp. and 0.39% COOH-fenp., all in terms of the initial dose. In addition there were maxima of 0.07, 0.28, 0.34 and 0.16% of unidentified products. The ^{14}C mass balance for the whole period ranged from 98.7% to 107.1% with a mean of 102.4% and a value at the end of the study of 98.7%. The half-life was calculated, using a first order model, to be 152 days. The authors considered that their results were in good accord with those of Mikami *et al*. (1983a) (Cranor, 1990).

The degradation of fenpropathrin in a loam soil from California was studied in aerobic conditions for 30 days followed by anaerobic conditions for 60 days. The fenpropathrin was labelled in the benzyl ring and was added to the soils to produce a nominal concentration of 10 mg/kg but the mean concentration after dosing was determined to be 12.1 mg/kg. The fenpropathrin was added to the pre-incubated soils which were then incubated at 25°C for a further 30 days. The soil moisture level was adjusted to approximately 75% of the field capacity and maintained at that level by periodic additions. The containers were equipped for the collection and determination of CO_2. After 30 days of aerobic incubation, anaerobic conditions were initiated by adding a small amount of glucose to the soils, covering them with water and flushing the flasks with nitrogen. The soils were extracted periodically with methanol and unextracted activity was determined by combustion analysis.

At the end of the aerobic phase, 85.1% of the activity remained as the parent compound. PBacid (2% of the initial dose), COOH-fenp. (1.3%) and 4′-OH fenp. (0.3%) were also found in the methanol extract. CO_2 was found to the extent of 0.6% of the applied dose. Desph-fenp. and $CONH_2$-fenp. were not detected. (These results were compared with those from another aerobic study which was continued for a year, in which 88% of the parent compound remained after the first 30 days and 8.9% was lost as CO_2. $CONH_2$-fenp. (0.12%), desph-fenp. (0.4%), a trace of 4′-OH-fenp (0.04%) and COOH-fenp. (0.26%) also occurred).

After the 60-day anaerobic phase, 66.0% of the initial dose remained as parent compound, 11.5% as PBacid, 6.4% as COOH-fenp. and 0.7% was liberated as CO_2. $CONH_2$-fenp. and 4′-OH-fenp. were present only as minor metabolites (<1.0%). Unextractable residues at this point were 8.0% of the applied dose and 15.7% of the applied activity was recovered in the supernatant water. The mean mass balance was 98.4 ± 2.8%.

Half-lives of fenpropathrin in the aerobic phase of the study were estimated to be 196 days and in the anaerobic phase 186 days.

Hence, as had been reported by Roberts and Standen in 1976, the main effect of imposing anaerobic aquatic conditions was to impede the degradation of metabolites, especially PBacid. It is not possible to infer the position regarding TMPA in this study owing to the position of the label (Daly and Williams, 1990).

Photodegradation. Studies were carried out in Japan with fenpropathrin labelled in the cyano group, the benzyl ring or the 1-carbon position of the cyclopropyl ring and applied to thin-layer soil plates prepared according to the procedure of Helling and Turner (Science, 162, 1968, 562-3). The three soils were Kodaira light clay, Katano sandy loam and Azuchi sandy clay loam. The fenpropathrin was applied at a rate of 1.1 ì g/cm^2 and the plates exposed to natural sunlight for 10 days during the month of September. The water content of the soils did not fall greatly during the study. Dark controls were run at the same time.

During the study plates were withdrawn at intervals and the soils extracted with methanol/water (5:1) for the determination of extractable activity and TLC fractionation. Unextractable residues were fractionated into activity associated with fulvic acid, humic acid and humins.

Under irradiation, the half-lives of the CN-labelled fenpropathrin were 1, 4 and 5 days in the Kodaira, Azuchi and Katano soils respectively. The fenpropathrin left in the soils at the end of the 14-day period (averages for the three labels in each soil) amounted to 5.1, 29.4 and 32.9% of the amounts applied. The corresponding figures in the dark controls were 74, 85 and 96%; there was insufficient degradation for half-lives to be estimated.

The main degradation product under irradiation with all three labels was $CONH_2$-fenp. which reached a maximum in the three soils after 5, 7 and 7 days. Substantial amounts also occurred in the dark controls. For the most part, other metabolites were present in the irradiated soils in only very small amounts. An exception was PBacid, especially on the Katano soil where it reached a maximum of 11.4% of the total applied activity after 7 days. On that soil "others" and unextracted residues were higher than on the other two; the unextracted activity was mainly associated with the fulvic acid fraction of the soil organic matter. Minor metabolites found were COOH-fenp., desph-fenp., Æ-carbamoyl-3-phenoxybenzyl alcohol (PM-amide) and 3-OH-Bacid. The last three did not occur in the dark controls.

The recovery of total activity in the dark controls was mostly just above 100% after 14 days, but gradually declined in the irradiated soils, presumably owing to mineralization of the labelled moiety. The losses shown in Table 14 were reported.

Table 14. Loss of activity (%) from irradiated soils after 14 days.

Soil	Loss of activity, %		
	Cyclopropyl label	CN label	Benzyl label
Kodaira	13.2	19.8	9.8
Azuchi	15.3	28.4	8.4
Katano	43.4	52.5	23.6

Clearly, loss was greatly enhanced by irradiation. It is evident that the nitrile carbon was the most susceptible followed closely by the 1-carbon of the cyclopropyl group. The benzyl group was evidently a more stable part of the molecule. It is clear that differences between the soils affected the rate of loss. The Katano soil was especially active owing, presumably, to the presence of photosensitizing substances. As shown in another part of this study, humic acid exerted a marked sensitizing effect when added to irradiated solutions of fenpropathrin in distilled water. It would seem likely that irradiation could play an important role in the degradation of surface deposits of fenpropathrin following spray applications which, in many cases, would be expected to remain on the soil surface for a large part of the season (Takahashi *et al*., 1983, 1985).

Adsorption. Attempts were made to determine adsorption coefficients by leaching treated soils but insufficient fenpropathrin passed through the columns for dependable estimates to be made. As an alternative approach, values may be deduced by using Briggs's equations linking the adsorption coefficient with the octanol-water partition coefficient (for fenpropathrin, $\log_{10} P_{ow} = 6.0$). The following Table was presented for two of Briggs's equations representing the lowest and highest values.

Table 15. Calculated values for adsorption coefficients of fenpropathrin.

Soil	Organic matter %	$\log_{10} P_{ow}$	K_d (Equation 2)	K_d (Equation 6)
Kodaira	15.3	6.0	500	2210
Azuchi	2.5	6.0	85	360
Katano	11.0	6.0	370	1590

Assuming that adsorption is confined essentially to the soil organic matter, the corresponding adsorption coefficient for organic matter (K_{om}) would have been (approximately) between 3300 (equation 2) and 14,400 (equation 6) (Mikami, 1983).

Leaching. A study was carried out with a sandy loam from the UK, packed into two glass columns and conditioned by slowly passing water through them until the effluent was clear. Fenpropathrin labelled in the benzyl or cyclopropyl ring was applied to the surfaces of the soil columns and the columns stored for 4 weeks. Two more columns were set up in the same way but not stored, and all four columns were leached at the rate of 0.25 ml/hour for 45 days, producing a total of 270 ml passing through each column, or a depth of about 17 cm. Samples of eluate that contained radioactivity were extracted with ethyl acetate, and extruded segments of soil taken at the end of the study were extracted with acetonitrile/water (7:3 v/v) for analysis.

The leachate from the stored column containing the cyclopropyl-labelled compound showed an appreciable level of activity amounting to 9.1% of that applied. This was shown by TLC to arise from approximately equal quantities of $CONH_2$-fenp. and TMPA.

In the soil columns, the major part of the radioactivity remained in the top 2 cm even in the stored soils. The total recoveries were 80-93% for the cyclopropyl and benzyl labels respectively in the unstored soils but these levels fell to 62 and 46% in the stored columns, indicating appreciable loss. The unextracted activity was between 41 and 47% except in the stored soil with the benzyl label; the low figure of 8% for this soil has no obvious explanation. The main metabolite in all cases was $CONH_2$-fenp., but except in the stored column with the cyclopropyl label the parent fenpropathrin constituted the major part of the residual activity.

It was concluded that neither fenpropathrin nor its degradation products were likely to leach through the soil under field conditions (Roberts, 1976).

A further study was carried out with fenpropathrin labelled either at the 1-cyclopropyl carbon or in the benzyl ring. There were 4 soils, Azuchi, Kodaira, Sapporo and Muko. The last had almost no organic matter or biological activity and consisted of 99% sand. The four soils were packed into columns to a height of 25 cm and the columns eluted with water until the eluate ran clear. More soil was treated with 1 mg/kg of the labelled fenpropathrin and placed on top of the columns either at once or after 4 weeks incubation at 25°C. During incubation provision was made for the determination of liberated CO_2. The columns were leached with a total of 1 litre of distilled water at a rate of 3 ml/hour for 14 days. From the dimensions of the columns, this would have been equivalent to a total depth of approximately 1.4 metres of water, a much greater depth than that in the Roberts study. At the end of the study the soil columns were divided into six segments of 5 cm each and the radioactivity was determined by combustion analysis. The treated soils and the top 5-cm sections were also extracted with methanol and the extract analysed.

In the eluates from the Kodaira, Azuchi and Sapporo soils radioactivity was negligible and no attempt was made to characterize it. There was a considerable level of activity in the eluate from the Muko soil. Where soil treated with cyclopropyl-labelled fenpropathrin without pre-incubation had been added to the column, 21.2% of the applied dose appeared in the eluate. The majority of the activity was from $CONH_2$-fenp. with small amounts from 4′-OH-fenp., desph.-fenp. and COOH-fenp., together with TMPA and others. There was very little fenpropathrin itself. In the columns containing pre-incubated soil treated with the cyclopropyl-labelled compound 37.6% of the applied dose appeared in the eluate. There was somewhat less $CONH_2$-fenp. (8.8%) but this was more than balanced by an increased amount of COOH-fenp. (17.5%) and the same complement of minor metabolites. In the case of the benzyl label, results were available only for the pre-incubated soil where the total activity in the eluate was 47.3% of that applied, with 8.7% as $CONH_2$-fenp. and 26.4% as COOH-fenp. A small amount of PBacid (3%) appeared (instead of TMPA of course).

Most of the activity in the soils remained in the treated layers, with minor amounts penetrating into the columns, mainly into the top 5 cm. In all cases the parent compound was the major component of the residues extracted with methanol and only small amounts of metabolites were present, even in the stored soils; $CONH_2$-fenp. was usually the most prominent. A considerable proportion of the activity remained unextractable in the pre-incubated soils, in one case reaching 35% (Kodaira soil with the cyclopropyl label), but in the Azuchi and Sapporo soils about 20% was more typical. The figure was much lower in the Muko sand (3-5%).

It was concluded that fenpropathrin and its metabolites showed only a very limited tendency to leach in normal soils (Mikami *et al.*, 1983b).

Conclusions. It is clear from these studies that fenpropathrin falling on soil will be degraded by a combination of photochemical and microbiological processes. It is unlikely that fenpropathrin would remain in the soil long enough to give rise to carry-over residues to affect succeeding crops.

The evidence is that metabolites do not accumulate in soil and that the degradation of fenpropathrin labelled in all 3 positions has been accompanied by the liberation of carbon dioxide, indicating that fragments observed to occur during the degradation process are themselves ultimately mineralized. There is no evidence of the accumulation of metabolites under aerobic conditions.

Fenpropathrin is strongly adsorbed by normal agricultural soils and studies in the laboratory using columns have shown that in such soils it is very resistant to leaching. The data give reassurance that when used as recommended, fenpropathrin will not cause contamination of groundwater in normal circumstances.

In storage and processing

Apples. Data for processed products are shown in Table 16. Residues of fenpropathrin in apples are essentially superficial as can be seen from the data on peeled apples in the French studies. As would be expected the extraction of apple juice leaves almost all of the residues in the solids.

Table 16. Residues of fenpropathrin in processed products: apples.

Country	Residues , mg/kg				
	Fruit		Pomace	Juice	Ref.
	raw	peeled	wet dry		
France	0.30	<0.01			10
France	0.36	<0.01			10
France	0.29	<0.01			10
France	0.31	<0.01			10
France	0.19	<0.01			10
France	0.20	<0.01			10
France	0.13	<0.01			10
France	0.13	<0.01			10
France	0.48	<0.01			10
France	0.49	<0.01			10
France	0.60	<0.01			10
France	0.16	<0.01			10
USA	3.6	3.4	34	0.10	71
USA	1.6	4.5	10.2	0.01	39

Pears. The position is similar to that of apples, except that the retention of residues by the pear solids would appear to be stronger than in the case of apples. The data are shown in Table 17.

Table 17. Residues of fenpropathrin in processed products: pears.

Country	Residues , mg/kg

	Fruit		Puree	Pears in	Ref.
	raw	peeled	washed	Syrup	
France	0.10	<0.01			8
France	0.05	<0.01			8
France	0.03	<0.01			8
France	0.65	<0.01			9
France	0.35	<0.01			9
France	0.24	<0.01			9
France	0.17	<0.01			9
France	0.42	<0.01			9
France	0.33	<0.01			9
France	0.13	<0.01			9
France	0.10	<0.01			9
France	0.19	<0.01			9
France	0.17	<0.01			9
France	0.19	<0.01			9
France	0.17	<0.01			9
USA	1.70		<0.01	<0.01	64
USA	1.25	0.01			73
USA	0.89	0.01	0.72		72

Grapes. As can be seen from Table 18 residues of fenpropathrin are retained by solids during grape juice extraction, as with apples and pears.

Table 18. Residues of fenpropathrin in processed products: grapes.

Country	Residues , mg/kg						Ref.
	Fruit	Raisins	Raisin	Pomace	Juice	Wine	
	raw	washed	waste	wet	dry		
France	0.06				n.d.		6
USA	0.37	0.45		0.49	2.5	0.03	26**
USA	0.75	1.5	0.13	0.90	4.9	0.09	26
USA	0.74	0.65	3.2	0.85	1.2	n.d.*	n.d. 26
USA	0.42	0.43					26
USA	3.1				0.01	n.d.	26
USA	2.6			2.8	10.5	0.01	26
USA	1.0	0.87					26
USA	1.4			0.99	2.6	n.d.	n.d. 26
USA	5.6			4.1	9.4	0.06	n.d. 26
USA	1.3				4.1	0.04	n.d. 26
USA	0.84	3.2	6.0	1.1	2.1	0.13	26

* n.d.= not detectable, <0.01 mg/kg
** The reference 26 (Fujie, 1992) includes the trials T-6077, T-6078, T-6412, T-6409, T-6413, T-6416, T-6728, T-6731, T-6829, T-6413, T-7545

Cotton seed. Three samples of seed from field trials were subjected to simulated laboratory processing as described below (ref. T-6427, 210-214 of Fujie, 1990a) and the residues surviving in various fractions were determined. It can be seen from Table 19 that residues in

soapstock were at about twice the level in the raw seed and that residues in the refined oil were in the region of three times those in the seed. Assuming a maximum level in raw cotton seed of 1 mg/kg, it can reasonably be concluded that residues will not exceed 2 mg/kg in soapstock and 3 mg/kg in refined oil.

Processing procedure. A Carver impact huller is used to obtain the fractions (kernels and hulls). The kernels are flaked in a Ferrell-Ross "flake-n-roll" to 0.008 of an inch thickness. The flakes are washed three times with hexane at a temperature of approximately 145°C. This extraction process takes 3 hours. The oil is recovered with a precision laboratory evaporator. During this process the oil reaches a maximum temperature of 75°C. Warm air is forced through the extractor to dissolve the cotton seed flakes. The oil is refined by the following steps:

1. NaOH is added to the oil while it is stirred at 250 RPM at a temperature of 20-24 °C for 15 minutes.
2. The oil is heated to 63-67°C for 12 minutes and the stirring reduced to 70 RPM.
3. The oil is then allowed to settle for 60 minutes at a temperature of 60-65°C.
4. The oil is refrigerated overnight or at least for 12 hours.
5. After refrigeration the oil is filtered to obtain the refined oil and soapstock fractions.

Table 19. Residues of fenpropathrin in processed products: cotton seed processed in the USA. Fujie, 1990a (ref. 23).

Residues, mg/kg						Ref.
Seed	Meal	Crude oil	Refined oil	Hulls	Soap-stock	
0.02	0.01	0.06	0.06	0.02	0.01	T-6070
0.03	0.02	0.07	0.09	0.03	0.05	T-6071
1.2	0.09	2.3	2.6	1.0	1.6	T-6427

Stability of residues in stored analytical samples

Fenpropathrin. Samples of apples, cotton seed, grapes, oranges and pears were stored at -20°C for periods up to 12 months and analyzed for residues of fenpropathrin to determine the extent of loss during storage. The results are shown in Table 20.

Table 20. Effects of frozen storage on residues of fenpropathrin.

Interval (months)	% of initial level				
	Apples	Cotton seed	Grapes	Oranges	Pears
3	82	133		93	99
6	78	116	89	120	77
9	91		87		78
12	82	100	87	88	89

No clear trend emerges from these figures which fall within the normal spread of recoveries and it was concluded that there were no measurable losses from any of these items during one year of storage at -20°C. Similar conclusions were drawn from studies with grape juice,

grape pomace, raisins and raisin waste (Fujie, 1992).

Stability studies were also carried out on eggs and cattle kidneys over periods of 156 and 71 days respectively. Again there was no evidence of decline in the residue levels during storage (Fujie *et al*., 1986b).

It may reasonably be inferred that the same conclusions could be drawn for other food and feed items.

Metabolites. Samples of macerated oranges and pears were spiked with known amounts of either PBacid or TMPA and analyzed after 7.5 or 11.2 months of storage. The results were compared with recoveries from freshly spiked macerated samples of the same substrates. See Table 21.

Table 21. Storage stability of metabolites.

Fruit	Interval (months)	% recovery from spiked samples			
		Frozen		Fresh	
		TMPA	PBacid	TMPA	PBacid
Orange	7.5	72	77	92	82
		75	71		
	11.2	62	79	67	75
		64	78		
Pears	7.5	55	62	65	77
		62	66		
	11.2	57	70	72	76
		54	59		
Mean		63	70	74	78
Coefficient of variation		12%	11%	17%	4%

Hence the recoveries of TMPA and PBacid from fresh and frozen samples show no significant differences and demonstrate that both metabolites are stable in orange and pear macerates during the storage intervals of the study (Fujie, 1990c).

It is reasonable to infer a similar stability in other substrates.

Residues in the edible portion of food commodities

Crop by-products used for animal feed

The main commodities used for animal feeds are the fruit pomaces, cotton seed meal and soapstock. Residues in cotton seed meal and soapstock were much lower than in the fruit pomaces.

Fruit pomaces. Dry grape pomace (see Table 18) contained between 2 and 7 times the residue level in the original grapes. If the highest level in raw grapes is 5 mg/kg, the highest level to be expected in dry grape pomace would be 35 mg/kg. In the case of raisin waste,

the highest factor was again 7 so that a similar figure could be anticipated.

In the case of dry apple pomace the two sets of results in Table 16 suggest a maximum concentration factor of 9, so that residues in dry apple pomace would not be expected to exceed 45 mg/kg on the
basis of a maximum residue level in whole apples of 5 mg/kg.

METHODS OF RESIDUE ANALYSIS

Parent compound. A number of analytical procedures have been developed by Sumitomo. These depend on solvent extraction of the substrate, a clean-up by either silica gel or Florisil column chromatography and the determination of the extracted residue by GLC using electron capture detection. The main variations dictated by different substrates are concerned with extraction and clean-up procedures. Fruits and vegetables may be homogenized with water, shaken with acetone and extracted according to accepted procedures with dichloromethane, using sodium chloride to minimize emulsification. After drying with anhydrous sodium sulphate and clean-up by silica gel column chromatography, the solvent is evaporated at <40°C and the residue dissolved in acetone before estimation by GLC with EC detection. Other extraction procedures involve direct extraction of the homogenized material without suspension in water or homogenization with methanol instead of water.

In the case of cotton seed oil dissolution of the sample in n-hexane is followed by extraction with acetonitrile, which is removed by evaporation and the residue dissolved in acetone for measurement.

A somewhat similar procedure is recommended for animal fats, whereas in the case of meat or offal (kidney was specifically studied) the sample is homogenized in acetone and the suspension extracted with n-hexane. After drying with anhydrous sodium sulphate the solution is extracted with acetonitrile, the solvent evaporatedd and the residue redissolved in n hexane for estimation.

Milk is mixed with an equal volume of acetone and centrifuged. The combined supernatant layers are extracted with n-hexane, the extract is dried with anhydrous sodium sulphate and the solvent evaporated. The residue is redissolved in acetonitrile, cleaned up by silica gel chromatography, and dissolved in hexane before determination by GLC.

Soils are extracted with combined water/methanol and, after filtration, the extract is partitioned with dichloromethane. The solution is dried with anhydrous sodium sulphate and taken to dryness by rotary evaporation. The residue is re-dissolved in n-hexane containing 10% diethyl ether and cleaned up by silica gel chromatography. The solvent is removed by evaporation and the residue dissolved in acetone for estimation by GLC.

In the case of water samples large volumes, up to 10 l depending on the lower limit of determination required, are extracted with n-hexane and the extract dried with sodium sulphate and cleaned up by Florisil column chromatography.

The relationship between the amount of fenpropathrin and peak area is linear over the usual range of 0-0.8 ng of fenpropathrin with a lower level of determination of 0.04 ng. Recoveries from most substrates have been reported to be between 90 and 100% although this fell to 80% in water where very large volumes were involved. The lower limit of determination in most crop samples is 0.01 mg/kg although in some cases it is possible to achieve 0.005 mg/kg, depending on the success of the clean-up steps. In whole milk a lower limit of determination of 0.001 mg/kg is usual, whereas in water limits down to 0.001

µg/l have been achieved (Ohnishi and Suzuki, 1981, 1982a,b, 1983a,b,c; Ohnishi *et al*., 1987; Kadooka *et al*., 1991; Hirota, 1990).

The procedures for crop samples are basically similar to those adopted by the Shell Company, except that in their procedure the crop samples are extracted dry in the presence of anhydrous sodium sulphate and homogenized with a 1:1 mixture of acetone and petroleum spirit. The extract is dried and cleaned up by Florisil column chromatography, the solvent is evaporated and the solute dissolved in petroleum spirit before its analysis by GLC. It is recommended that a standard solution should be chromatographed with each two sample solutions to make certain that there is a continuous correction of small changes in the response of the equipment. The method also includes a TLC procedure for confirming the identity of the fenpropathrin (Anon, 1976b).

Methods developed in the USA are similar in principle to those developed in Japan. In summary, macerated crop samples (20g) are blended with 100g sodium sulphate and 150 ml acetone/hexane (1:2), re-extracted, and the extracts dried with sodium sulphate. The extracts are then shaken with water and partitioned with hexane. The resulting hexane extract is evaporated to dryness, and the residue redissolved in hexane for clean-up by silica gel chromatography. The column is eluted with 1:5 ether/hexane, the eluate evaporated to dryness and the residue redissolved in methanol. The methanol solution is further cleaned up on a C-18 "SepPak", the eluate is taken to dryness, and the residue redissolved in hexane for determination of fenpropathrin by gas chromatography with an electron capture detector.

The response of the system is linear with analytical standard solutions covering the range 0.06-0.12 µg/ml, but the linearity must be checked each day. The limit of determination of the method for crop samples is approximately 0.01 mg/kg for a 20-g sample which is equivalent to a concentration in the injected solution of 0.01 µg/ml. A fortified control sample should be analyzed with each set of unknown samples. Recoveries from fortified samples should be between 70 and 120%; recoveries outside these limits require a repeat of the analysis.

This method has been developed further to cover residues in oily samples such as cotton seed. The macerated sample (20g) is moistened with 20 ml water and blended with 100 g sodium sulphate and 200 ml acetone/hexane (1:2), re-extracted, and the extract concentrated to about 20 ml, made up to 50 ml with more hexane and partitioned into acetonitrile. The solution is evaporated to dryness and the residue redissolved in hexane for clean-up and measurement.

Oil samples are dissolved in hexane, partitioned into acetonitrile and the solution evaporated to dryness. The residue is redissolved in hexane and subjected to an alumina column clean-up. The fenpropathrin is eluted from the column with hexane containing 10% ether. The eluate is evaporated to dryness and the residue redissolved in hexane for GLC.

For soapstock, a 5-g sample is dissolved in water, diluted further in a separating funnel and extracted twice with dichloromethane, using phosphoric acid and sodium chloride to minimize emulsification. The combined extracts are taken to dryness, redissolved in hexane and partitioned with acetonitrile which in turn is evaporated and the residue redissolved in hexane for clean-up on an alumina column (Leary and Abell, 1986c; Fujie, 1990a).

The method also allows analysis of milk, eggs and animal tissues. The samples (homogenized as appropriate) are extracted with acetone/hexane (1:2), and the extract diluted with water. The lower aqueous layer is extracted with hexane. The hexane extracts are dried over sodium sulphate and the solvent removed. The residue is redissolved in

hexane and cleaned up by silica gel chromatography. With fatty samples, the hexane solutions are first extracted with acetonitrile then re-extracted with hexane before clean-up and subsequent determination by GLC with an electron capture detector (Fujie *et al.*, 1986b).

A further variation of the method was employed in the analysis of some of the grape samples, where a nitrogen-phosphorus flame ionization detector was used as an alternative to electron-capture (Fujie, 1992).

Metabolites. For the determination of the metabolites PBacid and TMPA, macerated crop samples are extracted with methanol/water, the pH adjusted to 8.3 and the solution extracted with hexane to remove parent fenpropathrin. It is then acidified to liberate the acids which are derivatized with pentafluorobenzyl bromide. An aliquot of the reaction mixture is then cleaned up by passing through a silica "SepPak" cartridge and analyzed by gas chromatography using a mass-selective detector.

Recoveries from crop samples averaged 69% for TMPA and 76% for PBacid but the acceptable recovery range was estimated to be 60-120% and thus wider than for fenpropathrin owing to the complexity of the procedure (Fujie, 1988).

NATIONAL MAXIMUM RESIDUE LIMITS

The following national MRLs were reported to the Meeting.

Country	Commodity	MRL, mg/kg
Austria	Apple	1
	Cucumber	
	(incl. Squash, Summer)	0.1
	Egg plant	0.1
	Grapes	1
	Pepper, Sweet	0.1
	Pear	1
	Tomato	0.1
Belgium	Apple	0.5
	Cucumber	
	(incl. Squash,summer)	1
	Egg plant	1
	Pepper, Sweet	1
	Pear	0.5
	Tomato	1
France	Apple	0.5
	Grapes	0.5

Country	Commodity	MRL, mg/kg
	Peach	0.5
Germany	Dwarf French beans	1
	Climbing French beans	1
	Cucumber	0.2
	Tomato	1
Hungary	Apple	0.3
	Cucumber	0.1
	Grapes	0.2
	Pears	0.3
	Tomato	0.2
Italy	Apple	1
	Citrus fruits	1
	Cucumber	
	(incl. Squash, Summer)	1
	Grapes	1
	Peach	1
	Pear	1
	Tomato	1
Japan	Apple	1
	Citrus	
	peel	10
	fruits	2
	Cucumber	2
	Egg plant	2
	Pepper, Sweet	2
	Peach	2
	Pear	1
	Strawberry	2
	Tea, Green, Black	30
	Tomato	2
Netherlands	Apple	0.5
	Cucumber	

Country	Commodity	MRL, mg/kg
	(incl. Squash,Summer)	1
	Egg plant	1
	Pepper, Sweet	1
	Pear	0.5
	Tomato	1
Spain	Apple	0.1
	Cotton seed	0.05
	Egg plant	0.5
	Pear	0.1
	Tomato	0.5
Switzerland	Beans (Greenhouse)	0.5
	Beans (Field)	0.02
	Cucumber	0.02
	Pome fruits	0.02
	Stone fruits	0.02
USA	Apple	5*
	Cotton seed	1*
	Pear	5*

* proposed tolerances in USA

APPRAISAL

Residue data from supervised trials on apples, cotton seed, gherkins, grapes, pears and tomatoes were supplied to the Meeting. No data on cucumber were received.

The major biotransformation reactions of fenpropathrin in animals consist in oxidation at the methyl groups of the acid moiety and at the 2_ and 4_ positions of the alcohol moiety, cleavage of the ester linkage and conjugation of the resultant carboxylic acids and alcohols with glucuronic acid, sulphuric acid and glycine.

Studies in plants with radio-labelled fenpropathrin demonstrate that in fruit fenpropathrin itself is the primary component of the residues, whereas in leaves degradation products constitute the greater part of the residues. The major metabolic reaction of fenpropathrin in plants has been found to be the rupture of the ester linkage followed by oxidation to produce 3-phenoxybenzoic acid (PB acid) and the corresponding alcohol and aldehyde. From the acid side of the molecule, the main metabolite is 2,2,3,3-tetra-methylcyclopropanecarboxylic acid (TMPA) and this compound can give rise to 2-hydroxymethyl-2,3,3-trimethylcyclopropanecarboxylic acid (TMPA-CH_2OH) and 5-hydroxymethyl-6,6-dimethyl 3-oxabicyclo-[3.1.0]hexan-2-one (TMPA-CH_2OH lactone) by

subsequent hydroxylation. Also PB acid can be hydroxylated at the 4′ position and the parent molecule at the 2′ or 4′ position. The hydroxylated derivatives exist in both free and conjugated forms. Breakdown products in plants did not differ greatly from those in animals. The residues of the main metabolites PB acid and TMPA in samples from supervised field trials constituted only a negligible proportion of the total residues. It is therefore considered appropriate to define the residue in crops as the parent compound.

The fate of fenpropathrin in the soil will be influenced by a combination of photo degradation and microbiological processes. It is unlikely that fenpropathrin will remain in the soil long enough for residues to survive and affect succeeding crops. Metabolites do not accumulate in soil. Fenpropathrin is strongly adsorbed by soils, and when used as recommended will not contaminate ground water. Examination of plants grown on treated soils showed only extremely small uptake of radioactivity.

The residue data from supervised trials were evaluated as follows.

Apple. Results of 19 US trials with a maximum application rate of 0.45 kg ai/ha, a 14-day PHI and a maximum of 8 applications showed that the residues were below 5 mg/kg in whole fruit (minimum 0.06 mg/kg, maximum 4.5 mg/kg, estimated maximum residue level 5 mg/kg).

Pear. The maximum level observed in pears treated according to anticipated approved uses was 3.2 mg/kg in whole fruit in the State of Washington, USA, where the spray concentration was 0.024%, the application rate 0.9 kg ai/ha, the PHI 14 days and the crops were subjected to a total of 6 applications. In 15 supervised US trials within GAP based on 0.45 kg ai/ha, 0.012%, a 14-day PHI and 8 applications all residues were below 5 mg/kg (minimum 0.58 mg/kg, maximum 2.9 mg/kg; estimated maximum residue level 5 mg/kg).

Grapes. The maximum GAP was in US trials. There were 4 trials within GAP (0.45 kg ai/ha, a 21-day PHI and 4 applications; minimum 0.84 mg/kg, maximum 2.6 mg/kg; estimated maximum residue level 5 mg/kg) and 18 trials using 0.22 kg ai/ha, with a PHI of 21 days and also 4 applications. It is considered that residue levels from applications based on accepted use recommendations would normally fall below 5 mg/kg.

Gherkin. Residues in samples from 4 supervised German trials using an application rate of 0.08 kg ai/ha , a 3-day PHI and 3 applications did not exceed 0.1 mg/kg (minimum <0.01 mg/kg, maximum 0.1 mg/kg; estimated maximum residue level 0.2 mg/kg).

Peppers, Sweet. Residues from outdoors supervised trials based on 3 applications of 0.01% and a 0-1-day PHI in Japan and Spain ranged from 0.2 mg/kg to 1.2 mg/kg (estimated maximum residue level 1 mg/kg). Spanish residues (indoors, 7-day PHI) ranged from 0.04 to 0.38 mg/kg and for a 2-day PHI from 0.34 to 0.52 mg/kg.

Tomato. The highest levels were seen in four Japanese studies, because GAP in Japan allows an application rate of 0.25 kg ai/ha and a one-day PHI. One figure exceeded 1 mg/kg. Residue results of 5 outdoor and 8 indoor supervised trials in Germany with a lower application rate of 0.08 kg ai/ha show that 3 days after the last application residues were all below 0.6 mg/kg. (Outdoors: minimum <0.01 mg/kg, maximum 0.37 mg/kg. Indoors: minimum <0.01 mg/kg, maximum 0.46 mg/kg; estimated maximum residue level 1 mg/kg).

Egg plant. Residues from 4 Japanese trials based on 3 - 5 applications of 0.01% and a 1-day PHI were low (minimum 0.12 mg/kg, maximum 0.19 mg/kg; estimated maximum residue level 0.2 mg/kg).

Cotton seed. A well-known factor that can influence the level of residues in cotton seed is whether an appreciable number of bolls have opened at the time of the last application. If not, residues in the seed are usually very low but if there is direct contact between the

insecticide spray and the seed, residues can reach measurable levels. In considering the MRL needed it is important that it should be high enough to include cases where the last application was to plants with a comparatively high proportion of open bolls. It was possible to use 26 trials with an application rate of 0.22 kg ai/ha, 8 - 11 applications and a PHI of 18 - 22 days (minimum residues <0.01 mg/kg, maximum 1 mg/kg; estimated maximum residue level 1 mg/kg).

Residues in food of animal origin

Cattle. Residues in whole milk when a plateau level had been reached were approximately 0.15% of the level in the feed. If cows were fed on a diet consisting entirely of dried apple pomace at the postulated maximum residue level of 45 mg/kg (see processing of apples, below), it could be argued that the maximum level in milk would be 0.07 mg/l. Assuming that these residues would all be present in the fat and that the fat content of the milk would be 4%, such a level would be equivalent to 1.8 mg/kg in the milk fat. An animal transfer study showed levels in body fat to be approximately 1.4% of the level in the feed. Using the apple pomace figure of 45 mg/kg, it is reasonable to conclude that residues in meat fat would not exceed 0.6 mg/kg. Based on similar arguments and the data from the same studies, residues in meat (muscle) were about 0.08% of the feed level so that animals fed on apple pomace at 45 mg/kg would not be expected to have more than 0.05 mg/kg in muscle, kidney or liver.

Poultry. Poultry are unlikely to receive dietary items containing appreciable residues of fenpropathrin with the possible exception of cotton seed meal. With a maximum level of 1 mg/kg in raw cotton seed, it is unlikely that residues in meal would exceed 0.1 mg/kg. With a total feed level of 2.5 mg/kg, the level in fat reached only 0.02 mg/kg so that measurable residues would not be expected in the eggs, meat or edible offal of poultry fed on cotton seed meal.

In processing

In fruits the residues are essentially surface residues. As would be expected juice extraction leaves the great majority of the residues in the solids. In the case of dry apple pomace the data suggest a maximum concentration factor of 9, so that residues in dry apple pomace would not be expected to exceed 45 mg/kg on the basis of a maximum residue level in whole apples of 5 mg/kg.

As would be expected, raisins have higher residues than the raw grapes. The highest concentration factor in the trials is about 3. Using this factor and assuming that residues in raw grapes will not exceed 5 mg/kg, it would seem reasonable to estimate that residues in raisins would not exceed 15 mg/kg.

Dry grape pomace contained between 2 and 7 times the residue level in the original grapes. If the highest level in raw grapes is 5 mg/kg, the highest level to be expected in dry grape pomace would be 35 mg/kg.

Processing grape juice into wine appears to reduce residue levels still further and although strictly comparable data are only rarely available, residues of fenpropathrin have not been found above the limit of determination in wine, whereas in juice the highest level found was 0.06 mg/kg, which disappeared during vinification. In this particular case residues in the raw grapes were up to 5.6 mg/kg, so that even at this high level measurable residues did not survive in the wine.

As would be expected from the lipophilic nature of fenpropathrin, residues in oil obtained from cotton seed are higher than in the raw seed by roughly the inverse proportion of oil weight to seed weight. The residues in the meal ranged from 0.01 to 0.09

mg/kg. Residues in soapstock were about twice the level in the raw seed and residues in the refined oil were in the region of three times the seed level. Assuming a maximum level in raw cotton seed of 1 mg/kg, it can reasonably be concluded that residues in soapstock will not exceed 2 mg/kg and in oil 3 mg/kg.

Stability of stored analytical samples

In stability studies carried out on apples, pears, grapes, oranges, cotton seed, eggs and kidney of cattle over periods from 3-12 months there was no evidence of a decline in residue levels of fenpropathrin during storage at -20°C.

Methods of residue analysis

Methods of analysis used GLC with an EC detector after solvent extraction of the substrate and clean-up by either silica gel or Florisil column chromatography. The limit of determination in most crop samples is between 0.005 and 0.01 mg/kg.

RECOMMENDATIONS

On the basis of the residue data from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing Maximum Residue Limits.

Definition of the residue: fenpropathrin (fat soluble)

Commodity		Recommended MRL (mg/kg)		PHI on which based, days
CCN	Name	New	Previous	
MO 0812	Cattle, Edible offal of	0.05	-	
MM 0812	Cattle meat	0.5 (fat)	-	
ML 0812	Cattle milk	0.1 F	-	
SO 0691	Cotton seed	1	-	18-22
OC 0691	Cotton seed oil, crude	3	-	
PE 0112	Eggs	0.01*	-	
VO 0440	Egg plant	0.2	-	1
VC 0425	Gherkin	0.2	-	3
FB 0269	Grapes	5	-	21
VO 0445	Peppers, Sweet	1	-	0-2
FP 0009	Pome fruit	5	-	14
PO 0111	Poultry, Edible offal of	0.01*	-	
PM 0111	Poultry meat	0.02 (fat)	-	
VO 0448	Tomato	1	-	3

* Limit of determination

FURTHER WORK OR INFORMATION

None.

REFERENCES

1. Almond, R.H., Dawson, J. and Brown, D.C. 1984. The determination of concentrations of S 3206 in tomatoes . Huntingdon Report SMO 185/4, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR-41-0094. Unpublished.

2. Anon 1976a. 1975-Residue data for SD-41706 (Fenpropathrin) in cotton seed from plants treated with seventeen applications of SD-41706, a South Carolina study. Shell Dev Co TIR 24 285 75, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 61-0007. Unpublished.

3. Anon 1976b. Determination of Residues of WL 41706 in crops. Shell Research WAMS 232-1, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FA-81-0006. Unpublished.

4. Anon 1982. Residue data for Danitol (S-3206) in Grapes. Chevron study T 5864, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR-21-0159. Unpublished.

5. Anon 1984. Report on supervised trials for residue analysis (Apples). Hungarian Ministry of Agriculture, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 41 0107. Unpublished.

6. Bosio, P.G. 1979. Residues of Fenpropathrin in grapes and wine from France - 1978 trials. Shell Chimie report BEER 79 005, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR-81-0278. Unpublished.

7. Bosio, P.G. 1980. Residues of S 3206 in grapes from France - 1980 trials. Shell Chimie report BEER 80 014, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR-01-0066. Unpublished.

8. Bosio, P.G. 1981a. Residues of S 3206 in pears from France - 1980 trials. Shell Chimie report BEER 80 013, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR-01-0064. Unpublished.

9. Bosio, P.G. 1981b. Residues of fenpropathrin in pears from France -1981 trials. Shell Chimie report BEER 81 009, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 11 0275. Unpublished.

10. Bosio, P.G. 1981c. Residues of fenpropathrin in apples. Shell Chimie Report BEER 81.006, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR-11-0273. Unpublished.

11. Bosio, P.G. 1984. Residues of fenpropathrin in apples from France treated with Danitol EC formulation - 1983 trials. Shell Chimie report BEER 84 011, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR-31-0274 and FR-41-0314. Unpublished.

12. Chen, Y.S. and Abell, J. 1985. The metabolism of fenpropathrin in plants. Chevron report 12th April 1985, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FM 41-0031. Unpublished.

13. Chen, Y.S. and Abell, J. 1986a. The metabolism of fenpropathrin in apple plants. Chevron report 20th February 1986, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FM 51 0022. Unpublished.

14. Chen, Y.S. and Abell, J. 1986b. The metabolism of fenpropathrin in tomato plants. Chevron report 20th February 1986, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FM 51 0023. Unpublished.

15. Chen, Y.S. and Abell, J. 1986c. The metabolism of fenpropathrin in pinto bean plants.

Chevron report 20th February 1986, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FM-51-0024. Unpublished.

16. Cranor, W. 1990. Aerobic soil metabolism of benzyl ^{14}C fenpropathrin. ABC Laboratories Inc project 34586, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FM-91-0039. Unpublished.

17. Crayford, J.V. 1975. The excretion and residues of radioactivity in cows fed (^{14}C) WL 41706 in their diet. Shell Research TLGR 0096 75, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR-51-0051. Unpublished.

18. Csatlos, I. 1984a. Report on supervised trials for residue analysis. Hungarian Ministry of Agriculture Report, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 41 0099. Unpublished.

19. Csatlos, I. 1984b. Report on supervised trials for residue analysis. Hungarian Ministry of Agriculture Report, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR-41-0100. Unpublished.

20. Daly, D. and Williams, N. 1990. Anaerobic soil metabolism of ^{14}C fenpropathrin. ABC Laboratories Inc project 38182-2, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FM-01-0043. Unpublished.

21. van Dijk, C.P. and Vonk, J.W. 1989. Degradation of fenpropathrin in soil at 15°C. TNO (Delft) study 18649,
submitted to FAO by Sumitomo Chemical Company Limited under the reference number FM-91-0037. Unpublished.

22. Fujie, G.H. 1988. Addendum to: Magnitude of the residue and residue reduction of fenpropathrin and metabolites in pears. Chevron report 186/pears, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR-81-0304. Unpublished.

23. Fujie, G.H. 1990a. Magnitude of the residue of fenpropathrin in fuzzy cotton seed and cotton seed processing products. Chevron Report R194/cotton, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR-01-0306. Unpublished.

24. Fujie, G.H. 1990b. Magnitude of the residue and residue reduction of fenpropathrin in and metabolites in apples (Addendum). Chevron Report R185/apple/a, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR-81-0305. Unpublished.

25. Fujie, G.H. 1990c. Magnitude of the residues and residue reduction of fenpropathrin and metabolites in grapes and grape processing products. Chevron Report R187/Grape, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 01 0308. Unpublished.

26. Fujie, G.H. 1992. Addendum to magnitude of the residues and residue reduction of fenpropathrin and metabolites in grapes and grape processing products (MRID No.41734002). Chevron Report 1714/90/gra, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR-01-0335. Unpublished.

27. Fujie, G.H., Leary, J.B. and Abell, J. 1986a. Meat and milk residue feeding study with fenpropathrin technical (SX-1713) in dairy cattle. Bio Life Associates, Project No 86 MM3,

submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 61 0174. Unpublished.

28. Fujie, G.H., Leary, J.B. and Abell, J. 1986b. Poultry and egg residue feeding study with fenpropathrin technical (SX-1713) in laying hens. Hazleton Laboratories America Inc. Project 6183-103, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR-61-0175. Unpublished.

29. Fujie, G.H., Leary, J.B. and Abell, J. 1986c. Stability of fenpropathrin residues in crops stored at -20C. Chevron Report, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR-61-0190. Unpublished.

30. Gabriella, K. 1984. Report on supervised trials for residue analysis. Hungarian Ministry of Agriculture Report, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 41 0105. Unpublished.

31. Hirota, M. and Yamada, H. 1990. Residue analytical methods for fenpropathrin in river water. Sumitomo Chemical Company Method Report ER-MT-9022, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FA-00-0063. Unpublished.

32. Hitchings, E.J. and Roberts, T.R. 1977. The metabolism of the insecticide WL 41706, fenpropanate, in cotton. Shell Research report BLGR 0045 77, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FM-81-0005. Unpublished.

33. Kadooka, O., Hirota, M., Ohnishi, J. and Yamada, H. 1991. Residue analytical method for fenpropathrin in soil. Sumitomo Chemical Company Method Report ER-MT-9102, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FA-10-0074. Unpublished.

34. Kato, S. 1986. Analysis of magnitude of residues - fenpropathrin in tomatoes, 1986 trial in Japan. Japan Plant Protection Laboratory report, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR-61-0299. Unpublished.

35. Ku, H.S. and Doran, T.J. 1990a. A study to determine the nature of the residue in milk, meat and tissue from lactating goats dosed with ^{14}C fenpropathrin. Ricerca Inc report project 89 0109, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FM-01-0041. Unpublished.

36. Ku, H.S. and Doran, T.J. 1990b. A study to determine the nature of the residue in poultry and eggs from chickens dosed with ^{14}C fenpropathrin. Ricerca Inc report project 89 0084, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FM 01 0042. Unpublished.

37. Lai, J.C. 1985. Residue analysis of fenpropathrin in pears. Chevron report Test number 6394, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 51 0199. Unpublished.

38. Lai, J.C. and Fujie, G.H. 1985a. Residues of fenpropathrin in apples. Chevron report 6404, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR-51-0220. Unpublished.

39. Lai, J.C. and Fujie, G.H. 1985b. Residues of fenpropathrin in apples - 1985 study in New York USA. Chevron report 6406, submitted to FAO by Sumitomo Chemical Company Limited

under the reference nubmer FR-51-0222. Unpublished.

40. Lantos, J. 1984. Report on supervised trials for residue analysis. Hungarian Ministry of Agriculture, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 41 0106. Unpublished.

41. Lau, S.C. and Krudop, W.L. 1975. Residue levels of SD 41706 in cotton seed resulting from the application of SD 41706 to cotton - a Louisiana study. Shell Devco TIR 26 155 74 D, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 41 0017. Unpublished.

42. Macdonald, I.A., Dawson, J. and Richardson, R. 1984. The determination of concentrations of fenpropathrin in crops (Trial in Denmark, 1984). Huntingdon Report NAL 7/2, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 41 0112. Unpublished.

43. Maclellan, M.R. 1985. Standard processing procedures for residue determination of Danitol in apples. Letter from New York Experiment Station to Chevron, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR-51-0214. Unpublished.

44. Mikami, N. 1983. The response to EPA's requirement concerning soil studies of fenpropathrin (Danitol); data on the half-life and K_d calculations at 0.33 bar soil moisture. Sumitomo study, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FM 30 0013. Unpublished.

45. Mikami, N. and Sakata, S. 1984. The response to EPA's requirement concerning soil studies of fenpropathrin (Danitol); data on degradation in soils under aerobic conditions at shorter time points. Sumitomo report, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FM-40-0014. Unpublished.

46. Mikami, N., Sakata, S., Yamada, Y. and Miyamoto, J. 1983a. Degradation of fenpropathrin in soil. Sumitomo report, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FM 30 0008. Unpublished.

47. Mikami, N., Yoshimura, J., Yamada, H. and Miyamoto, J. 1983b. Leaching behaviour of fenpropathrin in the soil. Sumitomo report, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FM-30-0007. Unpublished.

48. Miyamoto, J. 1980. Comments on the metabolic fate of 3-phenoxybenzoic acid and 2,2,3,3-tetramethylcyclopro- panecarboxylic acid in the soil. Sumitomo report, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FM-00-0006. Unpublished.

49. Noble, S.R. 1976. Soil percolation and aqueous die-away studies with WL 41706. Shell Research report WKGR 0127 76, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 61 0013. Unpublished.

50. Ohnishi, J., Hirota, M., Suzuki, Y. and Matsuda, T. 1987. Residue analytical methods for fenpropathrin in cotton leaf and cotton oil. Sumitomo Chemical Company Method Report ER-MT-8707, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FA-70-0043. Unpublished.

51. Ohnishi, J. and Suzuki, Y. 1981. Method of residue analysis of fenpropathrin in apple. Sumitomo Chemical Company Method Report, submitted to FAO by Sumitomo Chemical

Company Limited under the reference number FA-10-0068. Unpublished.

52. Ohnishi, J. and Suzuki, Y. 1982a. Method of residue analysis of S-3206 (fenpropathrin) in cabbage, sweet corn, okra pods, cucumbers and tomatoes. Sumitomo Chemical Company Method Report, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FA 20 0014. Unpublished.

53. Ohnishi, J. and Suzuki, Y. 1982b. Method of residue analysis of fenpropathrin in pears. Sumitomo Chemical Company Method Report, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FA-20-0020. Unpublished.

54. Ohnishi, J. and Suzuki, Y. 1983a. Method of residue analysis of fenpropathrin in milk. Sumitomo Chemical Company Method Report, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FA-30-0024. Unpublished.

55. Ohnishi, J. and Suzuki, Y. 1983b. Method of residue analysis of fenpropathrin in kidney of cows. Sumitomo Chemical Company Method Report, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FA-30-0025. Unpublished.

56. Ohnishi, J. and Suzuki, Y. 1983c. Method of residue analysis of fenpropathrin in subcutaneous fat of cows. Sumitomo Chemical Company Method Report, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FA-30-0026. Unpublished.

57. Price, R.G. 1975. 1975 - Residue data for SD 41706 in cotton seed from cotton plants treated with 8 applications of SD 41706 - an Oklahoma study. Shell Devco TIR 24 283 75, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 61 0006. Unpublished.

58. Roberts, T.R. 1976. The leaching behaviour of WL 41706 in laboratory soil columns. Shell Research Report WKGR 0070 76, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 61 0012. Unpublished.

59. Roberts, T.R. and Standen, M.E. 1976. The degradation of the insecticide WL 41706 in soil under laboratory conditions. Shell Research Report WKGR 0061 76, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR-61-0014. Unpublished.

60. Robinson, K.A. 1984a. Residue analysis of fenpropathrin in pears. Chevron report Test number 6062, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 41 0192. Unpublished.

61. Robinson, K.A. 1984b. Residue analysis of fenpropathrin in pears. Chevron report Test number 6063, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 41 0193. Unpublished.

62. Robinson, K.A. 1984c. Residue analysis of fenpropathrin in pears. Chevron report Test number 6065, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 41 0195. Unpublished.

63. Robinson, K.A. 1984d. Residue analysis of fenpropathrin in pears. Chevron report Test number 6066, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 41 0196. Unpublished.

64. Robinson, K.A. 1984e. Residue analysis of fenpropathrin in pears. Chevron report Test

number 6067, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 41 0197. Unpublished.

65. Robinson, K.A. 1984f. Residue analysis of fenpropathrin in pears. Chevron report Test number 6153, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 41 0198. Unpublished.

66. Robinson, K.A. 1984g. Residue analysis of fenpropathrin in apples. Chevron report Test number 6156, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 41 0206. Unpublished.

67. Robinson, K.A. 1984h. Residue analysis of fenpropathrin in apples. Chevron report Test number 6158, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 41 0207. Unpublished.

68. Robinson, K.A. 1984i. Residue analysis of fenpropathrin in apples. Chevron report Test number 6159, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 41 0208. Unpublished.

69. Robinson, K.A. 1984j. Residue analysis of fenpropathrin in apples. Chevron report Test number 6150, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 41 0211. Unpublished.

70. Robinson, K.A. 1984k. Residue analysis of fenpropathrin in apples. Chevron report Test number 6151, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 41 0212. Unpublished.

71. Robinson, K.A. 1984l. Residue of fenpropathrin in apples. Chevron report Test number 6055, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 41 0205. Unpublished.

72. Robinson, K.A. 1985a. Residue analysis of fenpropathrin in pears. Chevron report Test number 6395, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 51 0200. Unpublished.

73. Robinson, K.A. 1985b. Residue analysis of fenpropathrin in pears. Chevron report Test number 6396, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 51 0201. Unpublished.

74. Schultz, D.R. 1975. Determination of WL 41706 residues in cotton seed. Shell Dev Co TIR 26 155 74 A, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 41 0016. Unpublished.

75. Skelsey, J.J. 1975a. 1975 - Residue data for SD 41706 in cotton seed from cotton plants treated with 11 applications of SD 41706 - a California study. Shell Devco TIR 24 276 75, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 61 0004. Unpublished.

76. Skelsey, J.J. 1975b. 1975 - Residue data for SD 41706 in cotton seed from cotton plants treated with 11 applications of SD 41706 - a California study. Shell Devco TIR 24 279 75, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 61 0005. Unpublished.

77. Slagowski, J. 1984a. Residue analysis of fenpropathrin in apples. Chevron report Test number 6161, submitted to FAO by Sumitomo Chemical Company Limited under the

reference number FR 41 0210. Unpublished.

78. Slagowski, J. 1984b. Residues of fenpropathrin in apples - 1984 study in Washington, USA. Chevron report Test number 6154, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR-41-0215. Unpublished.

79. Slagowski, J. 1985a. Residue analysis of fenpropathrin in pears. Chevron report Test number 6064, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 51 0194. Unpublished.

80. Slagowski, J. 1985b. Residues of fenpropathrin in apples - 1984 study in Michigan, USA. Chevron report Test number 6403, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 51 0219. Unpublished.

81. Takahashi, N., Mikami, N., Yamada, H. and Miyamoto, J. 1983. Photodegradation of fenpropathrin in water and on soil and plant foliage. Sumitomo study, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FM-30-0011. Unpublished.

82. Takahashi, N., Mikami, N., Yamada, H. and Miyamoto, J. 1985. Photodegradation of the pyrethroid insecticide fenpropathrin in water, on soils and on plant foliage. Pestic. Sci. 16: 119-31, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FM-50-0055.

83. Teleki, M 1983. Examination of Danitol 10E in vine. Hungarian study, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR-31-0082. Unpublished.

84. Test, A.G. 1975. 1975 - Residue data for SD 41706 in cotton seed from cotton plants treated with 8 applications of SD 41706 - a Mississippi study. Shell Devco TIR 24 303 75, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 61 0008. Unpublished.

85. Undeutsch 1983a. Residue analysis of fenpropathrin in tomatoes. Shell Agrar Study number R52-83, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 31 0240G. Unpublished.

86. Undeutsch 1983b. Residue analysis of fenpropathrin in tomatoes. Shell Agrar Study Number R53-83, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 31 0241G. Unpublished.

87. Undeutsch 1983c. Residue analysis of fenpropathrin in tomatoes. Shell Agrar Study Number R54-83, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 31 0242G. Unpublished.

88. Undeutsch 1983d. Residue analysis of fenpropathrin in tomatoes. Shell Agrar Study Number R55-83, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 31 0243G. Unpublished.

89. Undeutsch 1983e. Residue analysis of fenpropathrin in tomatoes. Shell Agrar Study Number R56-83, submitted to FAO by Sumitomo Chemical Company Limited under the refernece number FR 31 0244G. Unpublished.

90. Undeutsch 1983f. Residue analysis of fenpropathrin in tomatoes. Shell Agrar Study Number R57-83, submitted to FAO by Sumitomo Chemical Company Limited under the

reference number FR 31 0245G. Unpublished.

91. Undeutsch 1983g. Residue analysis of fenpropathrin in tomatoes. Shell Agrar Study Number R114-83, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 31 0246G. Unpublished.

92. Undeutsch 1983h. Residue analysis of fenpropathrin in tomatoes. Shell Agrar Study Number R115-83, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 31 0247G. Unpublished.

93. Undeutsch 1983i. Residue analysis of fenpropathrin in snake gherkins. Shell Agrar Study Number R47-83, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 31 0253G. Unpublished.

94. Undeutsch 1983j. Residue analysis of fenpropathrin in gherkins. Shell Agrar Study Number R48-83, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 31 0254G. Unpublished.

95. Undeutsch 1983k. Residue analysis of fenpropathrin in gherkins. Shell Agrar Study Number R49-83, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 31 0255G. Unpublished.

96. Undeutsch 1983l. Residue analysis of fenpropathrin in gherkins. Shell Agrar Study Number R51-83, submitted to FAO by Sumitomo Chemical Company Limited under the reference number FR 31 0257G. Unpublished.

97. Anonym 1993. CCPR Pesticide Documentation. 186 Fenpropathrin. Spanish GAP and residues studies in eggplant, pepper, tomato, apples. By Subdirection General de Sanidad Vegetal, Spain.

FLUCYTHRINATE (152)

EXPLANATION

Flucythrinate was first reviewed for residues by the 1985 JMPR, at which a number of MRLs were recommended. Additional data for a number of crops and more information on likely residues in animal products were desirable. New data were reviewed in 1987 and additional MRLs recommended. The 1988 and 1989 Meetings addressed issues relevant to MRLs for cabbage. The 1990 JMPR considered summary data submitted by the government of Spain from residue trials on citrus fruit, cucumbers, green beans and peppers and a separate submission of summary information on animal transfer studies. Full detials of the trials and transfer studies were required by 1992 so that maximum residue levels could be estimated.

Submissions to the 1992 JMPR in response to these requirements could not be reviewed at that Meeting. Much of the same, as well as other, information was submitted to the present Meeting, either by the manufacturer or the Spanish government, and other government comments were provided on animal MRLs. Some of the same data had been reviewed by the 1985 JMPR.

USE PATTERNS

New or updated information on relevant crops is summarized in Table 1. Where conflicting information was provided, the most recent is given preference.

Table 1. Nationally approved or registered uses of flucythrinate on selected crops.

Country/ crop	Application			PHI (days)	Notes
	Form.	Rate g ai/ha (g ai/hl)	No.		
Spain					
cabbages	EC	(4-6)*	**	3	* Except for maize, a separate submission on "registered uses to be considered by the EEC" lists 4-6 and 8-10 g ai/hl as the uses.
cotton seed	EC	40-60*** (4-6)	-	7	
cucurbits	EC ~	32-80 (4-10)	-	3	** Number was not indicated. "Each at 15-20 days" implies multiple applications at this interval.
green beans	EC	32-80 (4-10)	-	3	
lemons	EC	40 (4)	-	7	*** A separate submission indicated 20-30 g ai/ha.
maize	EC	1000-1200 (30-40)	-	7	
peppers	EC	32-80 (4-10)	-	3	
tomatoes	EC	32-80 (4-10)	-	3	
Japan					
Citrus fruits	EC	(3-4.4)*	4	21	* to run off
	WP	(3.3-5)*	4	21	

RESIDUES RESULTING FROM SUPERVISED TRIALS

Plants

The 1990 JMPR required detailed reports of trials on citrus fruits, cucumbers, green beans and peppers for which summary data had been provided. As indicated below, examination of the detailed reports by the Meeting revealed that most of them were the same as those reviewed by the 1985 JMPR. Although not requested, studies were also provided for brassicas, cotton seed and tomatoes. The trials on tomatoes, but apparently not those on brassicas and cotton seed, had been previously reviewed by the JMPR.

Beans, green. The trial details (Report Carse R-007, Spain, 1993; Cyanamid, 1993) revealed that these are the 1981 Egyptian trials reviewed by the 1985 JMPR. The Meeting was informed that the use is not GAP in Egypt, but is covered by Spanish GAP. Maximum residues were 0.14 and 0.22 mg/kg after three days from treatments within Spanish GAP.

Brassicas. The 0.2 mg/kg CXL for flowerhead brassicas (broccoli, Chinese broccoli, cauliflower) was recommended by the 1985 JMPR and the 0.5 mg/kg CXL for head cabbages (cabbage, green or red cabbage, oxhead cabbage, white cabbage, Savoy cabbage, yellow cabbage) by the 1987 JMPR. One national delegation had proposed higher limits on the basis of the data provided, but the relevant national uses were subsequently withdrawn and the current limits adopted. Although there appear to be no outstanding issues, data were submitted (Spain, 1993) on Brussels sprouts (two 1980 Netherlands trials, $\leq$0.06 mg/kg from treatments according to Spanish GAP, Reports TI-80-4 and TI-80-8/1414), broccoli (one 1982 German trial, $\leq$0.14 mg/kg, Report 1622), red cabbage (Report 1601), white cabbage (three 1981 German trials, 0.05 mg/kg from Spanish GAP, Report 1605) and Savoy cabbage (ten 1981 German trials, $\leq$0.37 mg/kg from Spanish GAP, Report 1549).

The trials on Brussels sprouts and broccoli appear to be the same as those reviewed by the 1985 JMPR.

Savoy cabbages. Data from ten 1981 supervised trials in Germany appear not to have been previously reviewed. Maximum residues were $\leq$0.29 mg/kg 3 to 5 days or more after 3 applications of an EC formulation at 30 g ai/ha (5 g ai/hl), except in one trial with residues of 0.28, 0.37, and 0.33 mg/kg after 4, 7, and 14 days respectively. All the trials were according to Spanish GAP and the residues are within the current 0.5 mg/kg limit for head cabbages.

Red and white cabbages. Three supervised trials in Germany in 1981 on each variety resulted in residues of 0.05 mg/kg 0 to 21 days after three EC applications at 30 g ai/ha (5-7 g ai/hl).

Citrus fruit. No new results were provided. Data from 4 Japanese trials (two on limes and 2 on mandarins) and 2 Egyptian trials on oranges (Report R-020) had been reviewed by the 1985 JMPR, but the Meeting was informed that the use is not GAP in Egypt. The Egyptian data on oranges included residues at 7 days, which is the Spanish PHI for lemons but from treatment at twice the Spanish GAP rate. The maximum residue was 1 mg/kg after 7 or 56 days. Residues in the Japanese trials from treatment in accordance with Japanese GAP were 0.2 to 1.5 mg/kg after 21 days, 0.2 to 1.7 mg/kg after 30 days and 0.2 to 1.5 mg/kg after 42/45 days. The required additional details of the trials reviewed in 1990 and details of the analytical method JT-001 used in the Japanese trials were also provided (Cyanamid, 1993).

Cotton seed. Summary data from supervised trials in 1978 in six States in the USA were submitted, which appear not to have been reviewed by the 1985 or 1987 Meetings (Spain, 1993). Reported residues were <0.05 mg/kg 29-166 days after 9 to 15 treatments at 60-120 g ai/ha. The current CXL is 0.1 mg/kg.

Cucumbers. The 1990 JMPR required the detailed reports which were summarized. The detailed report (Carse 003, Cyanamid, 1993) is of the

Egyptian trials reviewed by the 1985 JMPR.

Maize, maize forage and fodder. Residues in these crops are discussed below in the context of feed items relevant to MRLs for animal products.

Peppers. The detailed report required by the 1990 Meeting refers to the Italian trials reviewed by the 1985 JMPR (Report 1370/IT/GR/80, Spain, 1993, Cyanamid, 1993).

Tomatoes. The current 0.2 mg/kg CXL was recommended by the 1985 JMPR on the basis of data from Egypt, Finland, Italy, New Zealand and the United States and a 3-day PHI. Reports R-004-C/2 411/81, (1981 Egyptian trials) and 1290-IT/5R/80 (1980 Italian trials) were provided to the Meeting. These were reviewed by the 1985 JMPR (Spain, 1993).

Animals

The 1985 JMPR estimated temporary maximum residue levels at 0.5 mg/kg in the fat of meat for cattle and goats, 0.1 mg/kg in milk, and 0.05 mg/kg in eggs, although the basis for those estimates was not explained (the estimate for eggs was withdrawn by the 1990 Meeting). Of the animal feed items most relevant to these estimates, the 1985 JMPR recommended limits of 0.2 mg/kg for barley, oats and wheat, 5 mg/kg for their dry fodders, 0.2 mg/kg for green maize forage on the basis of European data, 0.5 mg/kg for pome fruit and 2 mg/kg for sugar beet tops. No limit was recommended for maize grain or fodder but the 1987 Meeting recommended 0.05 mg/kg for maize grain and 1 mg/kg for maize fodder on the basis of US data. All US uses have since been withdrawn.

The 1985 JMPR reviewed two goat metabolism studies and a cattle feeding study. In the first goat study goats fed for 7 days with [^{14}C]-alcohol- or [^{14}C]-acid-labelled flucythrinate up to 0.5 mg/kg in the feed showed flucythrinate equivalents of <0.01 mg/kg in the milk and <0.05 mg/kg in the tissues. In the second study, goats were fed for 7 days with both labels at 30 or 100 ppm in the diet. Milk and blood residues reached a plateau after 2 to 3 days. Maximum residues of flucythrinate equivalent from the 30 ppm feeding (alcohol label) in fat and milk were 1 mg/kg and 0.25 mg/kg respectively. The parent compound was the predominant residue in fat, muscle and blood, but hydrolysis products, lactones and conjugates predominated in kidney and liver.

In the cattle feeding study, with 13 and 39 ppm in the diet, residues in milk became steady after 4 days, a similar time to that found in goats. The 1985 JMPR did not record tissue residues, but cited average milk residues of 0.22 mg/kg for the lower feeding level. It was not indicated whether tissue residues had been provided.

The 1985 JMPR listed as desirable "information on actual residues found in meat, fat, milk and eggs after feeding treated animal feed crops or other crops of which wastes are used as animal feeds, e.g., straws of cereal grains, vines and/or straws of legume vegetables, etc." Over several years some delegations to the CCPR have questioned the temporary limits for cattle and goats, believing that adequate data on the residues in animals arising from feeding crops with expected residue levels were needed. Maize forage and fodder limits have been held at step 7B since 1989, awaiting reconsideration on the basis of storage stability studies. They were made temporary by the 1991 CCPR pending the submission of animal feeding studies. The 1990 JMPR received summary information on animal transfer studies, but did not evaluate it in the absence of the full reports.

The Meeting was provided with country comments on the issue, as well as comments from one of the two producers of flucythrinate. The Netherlands did not consider the relatively high feeding levels (30 or 13 mg/kg) to be suitable for estimating the maximum residue levels expected from the feeding of 5 mg/kg in cereal straws or 2 mg/kg in sugar beet leaves (Netherlands, 1992). Similar objections were raised by France (France, 1992).

One of the producers of flucythrinate repeated an argument already put forward in 1989 and 1990 in support of limits in animal products on the basis of information previously provided (Cyanamid, 1992). The 1990 JMPR had considered this information and had required submission of the detailed reports from which it was summarized by 1992.

As in 1989 and 1990, the latest submission did not consist in the detailed reports of the transfer studies, but extractions therefrom and a discussion of the issues. The data seemed to be from the studies reviewed by the 1985 JMPR, but included more detail than was given in the 1985 monograph. The Meeting did not have the original reports for reference. Although a full assessment is not possible in their absence, the Meeting noted the following points. The comments explain that the higher feeding levels were intended to accommodate worse-case levels expected at the time for proposed US uses (since withdrawn) and to provide sufficiently high levels of residues for metabolite identification. The producer points out that flucythrinate residues are fat-soluble, that residues increase linearly according to the dose and that residues are much higher in milk and fat than in other tissues. Tables 2 and 3 below were provided to support this view and, by pooling the data to support linear extrapolation formulas for fat of $y = 0.037x - 0.11$ ($r^2=0.98$) and for milk $y = 0.0075x + 0.06$ ($r^2=0.98$).

Table 2. Residues of [^{14}C]flucythrinate in goat tissues (quoted from Cyanamid, 1992).

Tissue	Mean residue levels		Residue ratio
	30 ppm feeding	100 ppm feeding	
Tenderloin muscle	0.041	0.085	2.1
Leg muscle	<0.05	0.075	
Kidney	0.013	0.057	4.4
Fat	0.86	3.63	4.2
Liver	0.021	0.079	3.8
Milk (4-7) days)	0.28	0.8	2.9
Blood (4-7 days)	0.07	0.28	4
Dose ratio: 30:100 = 1:3.3		Mean residue ratio 1:3.6	

Table 3. Residues of flucythrinate in cattle tissues (quoted from Cyanamid, 1992).

Tissue	Mean residue level		Residue ratio
	13 ppm feeding	39 ppm feeding	
Tenderloin muscle	<0.05-0.06	<0.05-0.075	
Omental fat	0.5	1.5	3
Back fat	0.3	0.99	3.3
Liver	<0.05	<0.05-0.052	
Kidney	<0.05-0.054	<0.05-0.086	
Milk (4-8 days)	0.22	0.37	1.7
Dose ratio: 13:39 = 1.3		Mean residue ratio 1:2.7	

NATIONAL MAXIMUM RESIDUE LIMITS

National MRLs for Spain were reported as 0.5 mg/kg for pome fruit, stone fruit, lemon, tomato, pepper, cucurbits, green beans, artichokes and cabbages.

APPRAISAL

Flucythrinate was reviewed for residues by the 1985, 1987, 1988, 1989 and 1990 Meetings. The 1985 JMPR listed additional data for a number of crops and information regarding likely residues in animal products as desirable. MRLs for these commodities have been retained at step 7, as have limits for maize forage and fodder. The 1990 JMPR required full documentation of data which were submitted only in summary form for citrus, cucumber, green beans and peppers. That Meeting did not evaluate summary information submitted on residues in animal products in the absence of the full reports. The present Meeting reviewed submissions made in response to the 1990 requirements, additional data for crops with and without current or proposed MRLs, and a re-submission of summary information on animal residues.

Green beans. Submission, as requested, of detailed reports on green bean trials provided only in summary to the 1990 JMPR reveals that the data are the same as the Egyptian data reviewed by the 1985 JMPR. The two values of 0.14 and 0.22 mg/kg after 3 days are consistent with reported Spanish GAP, but the Meeting concluded (as apparently did the 1985 JMPR) that the data were insufficient to support an MRL for green beans.

Brassica vegetables. There are no outstanding issues concerning the CXLs of 0.5 mg/kg for head cabbages and 0.2 mg/kg for flowerhead brassicas (broccoli, cauliflower). The Meeting received data on broccoli and Brussels sprouts which were reviewed by the 1985 JMPR and which did not need further review. New data were also received for red, white and Savoy cabbages. With maximum residues of 0.37 mg/kg from applications within Spanish GAP, no change in the current limits was required.

Citrus. Data reviewed by the 1985 JMPR from trials in Egypt (not GAP) and Japan were re-submitted, together with additional detail and GAP information for Japan and Spain. While the data suggest that residues are unlikely to exceed 2 mg/kg from GAP, the Meeting concluded (as implicitly did the 1985 JMPR) that data reflecting GAP in additional countries are needed to support an MRL for a major crop such as citrus. Processing studies would also be needed.

Cotton seed. Summary data reported from supervised trials in the United States and apparently not previously reviewed by the JMPR indicate residues well below the 0.1 mg/kg CXL. The Meeting concluded that there was no need to request the complete studies nor to revise previous estimates.

Cucumber. Submission of detailed reports as requested by the 1990 Meeting reveals that the data are the same as the Egyptian data reviewed by the 1985 JMPR. While the detailed reports indicate that the maximum residues of <0.05 mg/kg are consistent with reported Spanish GAP (3-day PHI) for cucurbits, the manufacturer could not confirm that the use is GAP on cucumbers anywhere. The Meeting concluded (as apparently did the 1985 JMPR) that the data were too limited to support an MRL.

Peppers. Submission, as requested by the 1990 JMPR, of detailed reports indicate that the summary data provided to that Meeting are the same as the Italian data reviewed by the 1985 JMPR. While the detailed reports indicate that the 0.14 and 0.13 mg/kg values after 4 days should be within Spanish GAP, the Meeting concluded (as apparently did the 1985 JMPR) that the data were too limited for estimating MRLs.

Tomatoes. There were no questions on the 0.2 mg/kg CXL recommended by the 1985 JMPR. The re-submitted studies had been previously reviewed.

Cattle meat and milk; goat meat. The current 0.5 mg/kg limits for the meat of cattle and goats and the 0.1 mg/kg limit for cattle milk have been retained at Step 7 by the CCPR pending submission of adequate animal feeding studies representative of feeding levels likely to occur in practice. Government comments provided to the Meeting proposed deletion of these limits with the view that available information was based on feeding levels irrelevant to actual animal intakes. The manufacturers again submitted discussion points and summary information previously provided to the 1990 JMPR, which required submission of the detailed reports from which

the summary information was taken. This information was not available to the Meeting.

The Meeting considered likely levels in commodities which could be used as animal feed items and agreed that the maximum dietary intake for cattle was unlikely to exceed 5 mg/kg, and in practice would probably be lower. Assuming this level, using the available feeding data summary, and assuming that residues in the animal products vary linearly with their levels in the feed from the 13 to 100 ppm levels fed experimentally down to the postulated 5 ppm, maximum residues of the order of 0.08 mg/kg in the fat of meat of cattle and goats and 0.1 mg/kg in milk could be estimated. This would suggest that the previously estimated levels of 0.5 mg/kg in the fat of meat and 0.1 mg/kg in milk would be adequate.

The summary information and comments provided to the Meeting provide greater insight into likely residues in animal products. However in the absence of the detailed reports from which the transfer data were summarized, and in view of the fact that the fat-solubility of flucythrinate leaves the potential for residues in animal tissues and milk, the Meeting recommended that the temporary limits for the meat and milk of cattle and goat meat should be withdrawn.

RECOMMENDATIONS

On the basis of the data on residues from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits or should be withdrawn.

Definition of residue: flucythrinate

Commodity		Recommended MRL (mg/kg)	
CCN	Name	New	Previous
MM 0812	Cattle meat	W[1]	0.5 (fat) T
ML 0812	Cattle milk	W	0.1 F T
MM 0814	Goat meat	W	0.5 (fat) T

[1] Withdrawn

REFERENCES

Cyanamid, 1992. Flucythrinate Residue Tolerances (discussion on MRLs for animal products); Appendix I, Supplementary Flucythrinate Residue Data; Appendix II, Summary of Flucythrinate Residue Data; Table 1 (Exhibits 17 and 18).

Cyanamid, 1993. Submissions of American Cyanamid Company for a variety of crops and animal product issues, analytical methods and GAP:

Citrus - Japanese supervised trials reports TTR-83-011 and TTR-83-012 and information on Japanese GAP.

Method JT-001 used in the Japanese supervised field trials and further details of the trials.

Citrus	1 -	Report Carse R-20, Egyptian trials.
Citrus	2 -	Report TTR-83-011 (corrected submission), Japanese data.
Citrus	3 -	Report TTR-83-012 (corrected submission), Japanese data.
GAP	4-6	Japanese labels (translated)
Cucumber	7 -	Report Carse 003, 1981 Egyptian trials
Green beans	8 -	Report Carse R-007, 1982 Egyptian trials.
GAP	9 -	Spanish labels (translated)
Peppers	10 -	Report 1370, 1981 Italian trials.

GAP 11 - Italian label (translated)

France, 1992. Comments from France. Flucythrinate (re: residues in animal products). Fax to FAO August 25, 1992.

Netherlands, 1992. Information of The Netherlands on pesticides to be considered by the 1992 JMPR, Flucythrinate, June 1992 (Ministerie van WVC, Directoraat Generaal van de Volksgezondheid.

Spain, 1993. Spanish government submission of GAP information and supervised trials data for a number of crops:

Peppers - Report 1370/ IT/GR/80
Tomato - Report R-004/ C/2 411/81 Egypt; Report 1290/ IT/5R/80 Italy
Brussels sprouts - Report 1414/ TI-80-4
White Cabbage - Report 1605
Green Beans - Report Carse R-007
Broccoli - Report 1622
Red Cabbage - Report 1601
Cotton seed - summary of data from 1978 trials in the USA (California, Louisiana, South Carolina, Georgia, North Carolina and Arizona).
Ministerio de Agricultura, Pesca y Alimentación. 1993.

FLUSILAZOLE (165)

EXPLANATION

Flusilazole was previously reviewed for residues by the 1989, 1990 and 1991 Meetings. An outstanding requirement from the 1991 evaluation was "Information on GAP and additional supervised trials data (including data on major metabolites) for nectarines and peaches reflecting that GAP from additional countries." The current MRLs are temporary pending the availability of this information.

Desirable information comprised:

1. Additional information on GAP for flusilazole on grapes in Europe.

2. Details of the wheat grain freezer storage stability study (Guinivan, 1987) and information on the relevance thereof to the storage of samples in the supervised trials.

3. Information on the stability of flusilazole metabolites (especially the silanol IN F7321) in cereal grains and/or plant parts under freezer storage conditions.

4. Submission of the hen metabolism study by Smyser (1990) which had been cited but not provided.

5. Information on residues of the major flusilazole metabolites (especially the silanol) in processed fractions of cereal grains from field-treated cereals containing measurable residues.

6. Submission of final reports of soil studies of which the interim reports were reviewed by the 1989 JMPR.

The Meeting received and reviewed information on all these items. Additional residue data were also submitted on pome fruit, grapes, barley, rye and wheat, although there were no outstanding requirements for trials on these commodities (they all have CXLs). Data on sugar cane were provided for the first time.

USE PATTERN

Information on current GAP in the use of flusilazole was provided for stone fruit, cereals, pome fruit, grapes and sugar cane, in some cases from countries whose previously submitted GAP has changed (Table 1). In cases where conflicting information was provided, that supported by labels or English translations of labels was given preference. The Meeting was informed that the product is not registered in The Netherlands owing to environmental persistence.

Table 1. Registered and approved uses of flusilazole on selected crops.

Crop Country	Application	PHI, days	Notes

	Form.	g ai/hl (g ai/ha)	No.	Int., days		
Cereals						
Belgium barley* rye & winter wheat	SC[1]	(160-200) (160)	 >1 >1		42	* winter or spring
Germany wheat	EC[2]	40-80 (160)	2		49	
winter wheat, barley, rye	SC[3]	≤75 (300)	1		56	
Italy barley, wheat	20DF	(120-160)	-	-	-	proposed use
Grapes						
Australia	DF	2 or (10)** (15)*** (20)****	*	14-21	14	* as needed ** early season *** mid-season ****late season (pre-flowering to bunch closing)
France	EW	(30)	2-3	14	*	*unspecified
Spain	40EC	1.6-3.2* 3.2-4.8*** (16-48)	5**		14	* normal infestation ** 10 cm buds to ripening, 2 appl. after flowering *** heavy infestation
Pome fruits						
Spain	40EC	2.4-4.8 (3.6-7.2)	4	10-14	14	
Australia Apple	DF	2-3*	>1**	10-14*	14	* pest pressure-dependent ** until term. growth ceases
Pear	DF	2	2	10-14	14	
Stone fruits						
France peach, nectarine	EW	4	3-4	12-14*	12-14*	* label interval, whether between or after appls. not specified

Table 1 (contd.).

Crop Country	Application				PHI, days	Notes
	Form.	g ai/hl (g ai/ha)	No.	Int., days		
Stone fruits						
Italy plum, peach, apricot, cherry	20DF	4	2-3		10	proposed use
Spain peach, apricot	40EC	4[4]	>1*	-	7	* start at petal fall
Greece peach	40EC	3-4 (600-800)	-	*	*	* pending use, no PHI proposed (PHIs of other countries will be considered)
Sugar cane Australia	Liq. 1.6 gai/L	0.2	*	*	*	* Set treatment in spray or dip planter

[1]Mixed formulation, 80 g flusilazole and 200 g chlorothalonil/l
[2]Mixed formulation, 160 g flusilazole and 350 g tridemorph/l
[3]Mixed formulation, 250 g flusilazole and 125 g carbendazim/l
[4]From manufacturer's label. Spanish summary gives 2 applications at 2.4-4.8 g ai/hl (26-53 g ai/ha).

RESIDUES RESULTING FROM SUPERVISED TRIALS

Cereals. Results of additional supervised trials were provided although there were no outstanding requirements for them and although there are CXLs for barley, rye and wheat at 0.1 mg/kg and for their straws and fodders (dry) at 2 mg/kg. Results are summarized in Table 2. All flusilazole residues in the grain were below the CXL (maximum 0.05 mg/kg in wheat and spring barley) and in the straw were ≤1.7 mg/kg except one residue of 2.3 mg/kg in wheat straw. This residue was found 56 days after the last of 3 post-emergence broadcast applications of a mixed suspension concentrate formulation (the first at 200 g ai/ha and the other two at 160 g ai/ha) in 1986 UK trials (Du Pont, 1993). Both the application rate and PHI are consistent with current GAP in other European countries and with UK GAP summarized by the 1989 JMPR.

The Meeting noted from data submitted, but not included in Table 2, that residues in straw and particularly forage could significantly exceed the 2 mg/kg CXL for dry fodders and straws at shorter PHIs and generally higher application rates than those which are reported as European GAP.

Table 2. Residues of flusilazole and metabolites on cereals resulting from supervised trials.

Crop Country Year	Application			Residue, mg/kg				Ref.[4]
	Rate g ai/ha	No.	PHI (days)	Flusilazole	F-7321[1]	H-7169[2]	TA[3]	
Wheat Italy 1987	175	1	34	0.05	<0.01			1
Winter wheat grain Germany 1986-8, USA 1983-4, UK 1986 straw	70-300	1-3	26-140 42-138	0.02, ≤0.014 (91)[5] 2.3, 1.7, 1.4, ≤1.1 (73)	0.02, ≤0.01 (7) 9.9, ≤6.6 (16)	≤0.01 (8) ≤0.3 (16)	2.5, ≤0.6 (10)	2
Spring wheat grain USA 1983-4 straw	70-140	2	19	0.013, <0.01 0.34, 0.3				3
Spring barley grain USA 1983-4 UK 1986 straw USA UK	70-140 160 70-160 160	2 2	12 63-69 12 63-69	0.01, 0.05 0.05, 0.01 1.2, 1.1, 1, 0.5	 3.9, 3.4 (2), 3.1	 ≤ 0.12 (4)	≤0.13 (4)	4 5
Winter barley grain Germany 1986-8, USA 1983-4, UK 1986 straw	140-300	1-2	44-88	0.02, ≤0.01 (17) 1.1-1.6 (5) ≤0.7 (20)	 7.7, 5.9, ≤4.8 (12)		≤0.31 (12)	6
Winter rye grain Germany 1986-8 straw	160-300 300	1-2	82-83 120	<0.01 (3) <0.01				7

[1] F7321: bis(4-fluorophenyl)(methyl)silanol.
[2] H7169: [bis(4-fluorophenyl)(methyl)silyl]methanol.
[3] TA: 3-(1H-1,2,4-triazol-1-yl)alanine.
[4] Numbers refer to Du Pont, 1993, Table 2.
[5] Number in parentheses following a residue indicates the number of residues at that level.

Grapes. The 1991 JMPR considered additional information on European GAP desirable because it had been suggested that the 1989 estimate of 0.5 mg/kg was unnecessarily high. It was based on German trials but the GAP of other European countries, and was obviously rounded up since the 1989 Meeting concluded that residues would not be likely to exceed 0.3 mg/kg. There was an outstanding question of whether the 5-8 applications in the German trials was GAP in Europe (the use was not registered in Germany). However, there were no requirements for additional data and the 0.5 mg/kg limit is now a CXL.

Information provided to the Meeting included the updated GAP of Spain (essentially unchanged), and France (essentially unchanged, still no PHI specified) and new information on GAP in Australia (Table 1). It confirms the appropriateness of the 14-day PHI used by the 1989 JMPR. Spain (1993) also provided data from trials in the USA (4 studies, 14 trials) and summary data on 6 grape (and 2 processing) trials (apparently French, no details, formulation unspecified). Results of one Australian trial were available (Australia, 1993). Residues in grapes did not exceed 0.15 mg/kg in the French trials (8 applications of an unspecified formulation at 20 g ai/ha, PHI unspecified), 0.08 mg/kg in the US trials (no US GAP) or 0.22 mg/kg from treatments reflecting GAP in the Australian trial.

Pome fruit. The CXL is 0.2 mg/kg. Although there were no outstanding requirements, the Meeting was provided with current GAP for Spain, Australian trials data (Australia, 1993) and a summary from Spain of trials in Italy, Germany, Belgium and France (Spain, 1993). In the Australian trials maximum residues in apples were 0.2 mg/kg at the Australian 14-day PHI from a double application rate and 0.07 mg/kg at the recommended rate. No residues (<0.02 mg/kg) were detected in pears after 21 days at 1 to 1.5 times the GAP application rate of 2 g ai/hl and up to 0.03 mg/kg at a threefold rate. The highest residues reported in the Spanish submission were 0.16 mg/kg from GAP and exaggerated application rates.

Stone fruit. The 1991 JMPR recommended a temporary MRL of 0.1 mg/kg for peaches and nectarines, based on GAP in New Zealand and Spain and data from France and New Zealand, pending the availability of additional data on residues of flusilazole and its metabolites from treatments according to GAP. New or updated information on GAP for peaches and nectarines or stone fruit in France, Italy, Spain and Greece is summarized in Table 1. Two or more applications at 3-4 g ai/hl and PHIs of 7 to 14 days appear to be usual for these countries, although a PHI is not specified for France or Greece. These practices are comparable to those recorded by the 1991 JMPR for New Zealand and Spain and are in accord with the 7-day PHI on which the 1991 MRL recommendation was based.

Data from supervised trials in Italy, Australia, France, the USA, and Greece were provided (Table 3) although no information on GAP was available for the USA or Australia. Two of the three French trials on peaches which were reported were reviewed by the 1991 JMPR and are not included in the Table. In two additional 1986-87 French studies on peaches (not in Table 3) no residues (<0.05 mg/kg) of triazolylalanine were detected 1 to 26 days after as many as 9 treatments with an EC formulation at application rates up to 30 g ai/ha (Du Pont, 1993, Reports BG-88-01 and BG-88-07).

Table 3. Residues of flusilazole and metabolites in stone fruits resulting from supervised trials. Underlined residues are from treatments according to GAP.

Crop Country Year	Application			Residues, mg/kg at intervals after last application	Ref.
	Form	No.	Rate g ai/ha (g ai/hl)		
Apricots			Days	0 1347 10 106	
France 1989	CE	4	30 (3)	<0.01	1
1991	10EC	8	24 (4)	0.30.20.090.080.05	2
				Controls ≤0.01	
1991	10EC	4	40 (14.3)	0.030.040.040.050.03	2
				Controls <0.01	
1991	10EC	6	40 (14.3)	0.080.10.070.060.06	2
				Controls 0.01	
Cherries					

Crop Country Year	Application			Residues, mg/kg at intervals after last application	Ref.
	Form	No.	Rate g ai/ha (g ai/hl)		
Australia 1989	20DF	3[1]	(2, 3, 4, or 8)	Not detected (<0.05 mg/kg) at 43 days	3
Peaches					
Australia 1990	20 DF	3[1]	(2, 3, 4, or 8)	Not detected (<0.05 mg/kg) at 148 days	3
				Daysflusilazole IN-F7321[2]IN-H7169[3]	
France 1986	40EC	3-4	23 (3)	1 0.060.03<0.01	4
(nectarines)			30 (4)	1 0.040.01<0.01	
			23 (3)	17 0.070.01<0.01	
			30 (4)	17 0.050.01<0.01	
				Controls <0.01 <0.01<0.01	
		9	10.5 (3)	8 0.02<0.01<0.01	4
			14 (4)	8 0.550.07<0.01	
				Controls 0.1,<0.01<0.01	
				0.05	
Italy 1988	20DF	3[4]	60 (4)	<0.01 at 102 days	5
				Control <0.01	
Greece			Days	0 3 7 14 21	
1992	40EC	5	40 (4)	0.1, 0.10.2, 0.2 0.05	6
			80 (8)	0.3, 0.20.07, 0.05 0.04	
				Controls 0.01	
1992	40EC	6	40 (4)	0.3, 0.2 0.090.02 0.03	6
			80 (8)	0.2, 0.2 0.070.05 0.04	
				Control <0.01	
USA 1984				5 days12 days 5 applcns. 3 applcns.	
	EC	3 or 5	70 (1.2) 140 (2.4) 280 (4.8) 140 (2.4)	0.050.1 0.20.06 0.30.3 0.10.1	7
			Days	712 14 15 22 29	8
U.S.A. 1984	40EC	3	70 (1.2)	0.040.030.02<0.01 0.01 0.01	
			140 (1.2)	0.050.070.05 0.05 0.020.03	
			280 (4.8)	0.20.10.1 0.09 0.050.03	
				Controls <0.01	
			Days	0 4 6 7 14 21	8
1984	40EC	5	70 (1.2)	0.050.060.030.04 0.050.03	
			140 (2.4)	0.050.080.090.09 0.080.06	
			280 (4.8)	0.20.50.30.2 0.2-	
Plums					
Australia 1990	20DF	3[5]	(2 to 8)	No residues (<0.05 mg/kg) at 119 days	
Australia 1989	20DF	3[5]	(7.5 to 30)	No residues (<0.05 mg/kg) at 121 days	3

Crop Country Year	Application			Residues, mg/kg at intervals after last application	Ref.
	Form	No.	Rate g ai/ha (g ai/hl)		
1989	20DF	3[5]	(2, 3, or 8)	No residues (<0.05 mg/kg) at 117 days	3
			(4)	0.07 at 117 days	

[1] The last at petal fall
[2] F7321: bis(4-fluorophenyl)(methyl)silanol
[3] H7169: [bis(4-fluorophenyl)(methyl)silyl]methanol
[4] Applications at bloom, beginning of fruit set and end of fruit set

Sugar cane. No limit has been proposed for sugar cane. The Meeting was provided with information on Australian GAP and data from trials in Australia in which sugar cane was grown from cane sets dipped at concentrations of 1, 2 (GAP rate), 5 and 10 mg flusilazole/l (Australia, 1993). No residues (<0.02 mg/kg) of flusilazole were detected in cane juice extracted 11 months after the dip treatments. The stalks were not analyzed.

FATE OF RESIDUES

In animals

The 1991 JMPR requested the submission of the report of a hen metabolism study which was cited and referenced, but not provided, in a 1991 submission to FAO (Wustner, 1991). In response the manufacturer re-submitted all available reports of hen metabolism studies, including the one requested (Du Pont, 1993).

The reports:
1. AMR-245-84 (Bodden and Kneeland, 1984); phenyl label. Reviewed by the 1989 JMPR (omitted from 1989 monograph references).
2. Supplement to AMR-245-84 (Stadalius, 1984). Not previously provided. 3. AMR-638-86-1 (Lin, 1988a); phenyl label. Reviewed by 1989 JMPR.
4. Supplement to AMR-638-86-1 (Lin, 1988b). Not previously provided.
5. AMR-638-86-2 (Lin, 1988c); triazole label. Reviewed by the 1989 JMPR.
6. Supplement to ARM-638-86-2 (Lin, 1988d). Not previously provided.
7. AMR-638-86, Supplement 2 (Smyser, 1990). The requested report.

Report AMR-245-84, reviewed by the 1989 JMPR, left questions on the nature of the residues in poultry. The situation was clarified by AMR-638-1 and -2 which were also reviewed in 1989. The Supplement to AMR-245-84 is merely a response to a US EPA requirement for a hen metabolism study with the triazole label and cites AMR-638-86-2 as fulfilling this requirement. The supplements to AMR-638-86-1 and -2, not previously provided to the JMPR, respond to EPA requirements for more details of the raw data and the calculation of the distribution of residues in the original reports. This leaves only the requested report for consideration by the Meeting.

The report (Smyser, 1990) is a response to EPA requirements for chromatograms showing the co-chromatography of metabolites and analytical standards, and for the expression of metabolite concentrations as a percenage of the total residue in Reports AMR-638-86-1 and -2. Table 4 of the 1991 JMPR monograph, taken from AMR-638-86-1, showed the metabolites in liver, kidney, muscle and fat (but not eggs) as a percentage of the total residues found with the phenyl label. The resiudes from the triazole-labeld compound were not shown. They are given in Table 4 below, which is taken from AMR-638-86-2.

Tabe 4. HPLC quantification of flusilazole and its metabolites in extracts of tissues from laying hens administered [triazole-3-^{14}c]flusilazole for 14 days at 3 ppm in the diet (Smyser, 1990).

Component	Component as % of total radioactivity in						
	Liver	Kidney	Thigh muscle	Breast muscle	Fat	Eggs[1] 4-day	12-day
Flusilazole	5	5	-	1	68	1	2
Triazole	76	79	75	86	14	83	77
Thymine	8	7	11	6	3	5	9
Other	6	9	3	2	15[2]	11	2

[1] 4 and 12 days chosen as representative of the 2-, 4-, 6-, 8-, 10-, and 12-day samples which were analysed.
[2] Radioactivity associated with three HPLC peaks. No single component exceeded 2%.

Triazole is the predominant residue in all samples except fat where flusilazole is greater.

When chickens were fed for 14 days at a 3 ppm dietary level with phenyl-labelled flusilazole, residues in 12-day eggs (chosen to be representative) were as follows (Smyser, 1990):

	% of total radioactivity
IN-F7321 (methyl silanol)	32
IN-37738 (silyl phenol)	5
Phosphate conjugate of IN-37738 at the 3-hydroxy position	3
[(4-fluorophenyl)methyl]silanediol	38
Flusilazole	4
P5 (unidentified)	5
P11 (at least 3 unidentified metabolites	3
Lipophilics	7
Other	3

In plants

No new information.

In soils

The 1989 and 1991 Meetings requested submission of the final reports on two field soil dissipation studies reviewed by the 1989 JMPR at an interim stage (AMR-556-86, Stadalius, 1986; AMR-791-87, Fujinari, 1986b). The completed study AMR 556-86 (Smyser, 1993) was submitted, but the Meeting was informed that report AMR-791-87 had not yet been completed.

In the AMR-556-86 study [phenyl(U)-^{14}C]flusilazole was applied to Delaware silt loam soil (in 38 cm x 10 cm i.d. steel cylinders driven into the ground) at the nominal rate of 105 g ai/ha four times a year for 3 years. That rate would be comparable to current GAP for many crops and higher than that for others. The 1989 JMPR noted that the half-life of flusilazole was <12 months and that flusilazole and its silanol metabolite (IN-F7321) were the main identified residues. It also noted that the radioactive residues increased with the number of applications, with 90% of the total radioactivity in the top 0 to 8 cm of the soil and with maximum soil residues of flusilazole

of 0.22 mg/kg after 2 years. Only 62% of the applied radioactivity was recovered after 2 years. Losses were attributed to photolysis and microbial attack (1989 JMPR monograph).

The interim report summarized results for 697 days and the final report for the entire 1092 days of the completed study. The percentages of the radioactivity recovered in the three cylinders at different soil depths in each of the three years, with a cumulative rainfall 325 cm, was as follows (from Table V of the report):

Soil depth (cm)	year 1	year 2	year 3
Day->	0-368	326-697	694-1092
0-8	97.3-100	92.6-99	92.6-98.6
8-16[a]	ND-0.9	0.7-5.6	0.9-4.8
16-24[b]	ND-0.5	0.2-1.4	0.4-2.3
24-36[c]	ND	ND-0.3[d]	0.1-0.9

[a] peak residue on day 633
[b] peak residue on day 737 (12th and last application)
[c] Nominal 38 cm cylinders, but 2-3 cm left above ground
[d] Radioactivity first detected after 368 days

Calculations indicate a flusilazole average half-life of 251 days. The statistical evaluation of the data given in the report predicts that flusilazole residues would reach a steady state of approximately 57% of the yearly application under worse-case application conditions.

The residues of flusilazole and IN-F7321 (expressed as mg/kg flusilazole in the 0-8 cm soil segment after each of 12 applications (from Table VI of the report) were:

Application	Day	Flusilazole	IN-F7321	Total
1	0		0.06	<0.01
0.06				
2	14		0.12	<0.01
0.12				
3	28	0.16	<0.01	0.16
4	42	0.22	0.01	0.23
5	326	0.22	0.04	0.26
6	339	0.26	0.04	0.3
7	354	0.31	0.04	0.35
8	368	0.31	0.05	0.36
9	694	0.26	0.05	0.31
10	708	0.36	0.08	0.44
11	722	0.39	0.06	0.45
12	737	0.34	0.07	0.41

In the 8-16 cm soil segments radioactivity did not exceed 0.01 mg/kg flusilazole equivalents until after 483 days (after the 6th application at 0.016 mg/kg), with maximum residues of 0.03 mg/kg after the 12th application. In the 16-24 cm segments residues did not exceed 0.01 mg/kg until immediately after the 12th application at 737 days (0.015 mg/kg), but were below 0.01 mg/kg after 844 days. In the 24-36 cm segments no residues exceeded 0.01 mg/kg throughout the study although, as noted in the Table showing percentage radioactivity above, some radioactivity was recorded in this segment after 368 days.

In water/sediment systems

No new information.

In storage and processing

Storage No information.

Processing. The 1991 Meeting requested information on residues of the major flusilazole metabolites (especially IN-F7321, the silanol) in processed fractions field-treated cereals with measurable residues in the grain. A processing study on barley (Guinivan and Desmond, 1993) was provided.

A 91-kg sample of barley grain was taken at maturity 47 days after the second of two over-crop tractor spray applications (at a two-week interval) to a 6 x 156 m barley plot at 420 g ai/ha (3 times the proposed US GAP rate). Bulk samples were shipped at ambient temperatures (a small sample also frozen) for simulated commercial processing at Texas A&M University. Samples were stored frozen until processing approximately 3 months later. The pre-milling steps included aspiration, screening, dehusking and husk removal. Pearled grain was milled (broken and sieved) to remove bran and further milled (reduction and sieving) for shorts, low-grade flour and patent flour.

Fractions were analyzed for flusilazole and its major phenyl metabolites by Du Pont method AMR 2126-91 (Koch, 1993; see "Methods of residue analysis") 16-20 months after processing. Validation data and sample chromatograms were provided. Percentage recoveries from the various fractions (mostly at 0.05 to 0.2 mg/kg fortifications) were flusilazole 90±14, IN-7321 89±19, IN-G7072 107 ±22, IN-37722 95±22, and IN-37738 91±15. The results are summarized in Table 5 (an the metabolites defined in its footnotes). Flusilazole residues were not concentrated in milling fractions which showed ratios of residue in fraction to residue in grain of 0.8 in bran, 0.6 in shorts 0.4 in low grade and 0.5 in patent flour. They were concentrated in normal pre-milling fractions: 9.2-fold in light impurities and 2.4-fold in huskes. A similar pattern was for metabolite IN-F7321. Factors could not be estimated for the other metabolites because the grain did not have measurable residues, although some concentration in the light impurities from pre-milling were observed.

Table 5. Residues of flusilazole and metabolites in barley grain and its processed fractions from simulated commercial processing (Guinivan and Desmond, 1993).

Sample	Residue, mg/kg				
	Flusila-zole	IN-7321[1]	IN-G7072[2]	IN-37722[3]	IN-37738[4]
Treated grain					
Shipped at ambient temp.	0.12, 0.14	0.05, 0.05	<0.05,	<0.05	<0.05
Shipped frozen	0.13, 0.06	0.1, 0.13	<0.05	<0.05	<0.05
Pre-milling fractions					
Light impurities	1.2, 0.82	0.26, 0.58	0.02, 0.11	0.09, 0.12	<0.05, 0.12
Screenings	0.09	0.05	<0.05	<0.05	<0.05
Husks	0.33, 0.19	0.18, 0.24	<0.05, 0.07	0.04, <0.05	0.03, 0.04
Milling					

Sample	Residue, mg/kg				
	Flusila-zole	IN-7321[1]	IN-G7072[2]	IN-37722[3]	IN-37738[4]
fractions					
Bran	0.09	0.07	<0.05	<0.05	<0.05
Shorts	0.07, 0.06	0.04, 0.07	<0.05	<0.05	<0.05
Low-grade flour	0.04	0.03	<0.05	<0.05	<0.05
Patent flour	0.09, 0.03	<0.05, <0.05	<0.05	<0.05	<0.05

[1] IN-7321 = bis(4-fluorophenyl)(methyl)silanol (I)
[2] IN-G7072 (disiloxane) = 1,1,3,3-tetrakis(4-fluorophenyl)-1,3-dimethyldisiloxane (V)
[3] IN-37722 = 2-fluoro-5-[(4-fluorophenyl)(methyl)(1-*H*-1,2,4-triazol-1-ylmethyl)silyl]phenol (IV)
[4] IN-37738 = 2-fluoro-5-[(4-fluorophenyl)(hydroxy)(methyl)silyl]phenol (XII).

Roman numerals refer to structures in Fig. 1, 1989 monograph.

Stability of pesticide residues in stored analytical samples

The 1989 JPMR required the submission of freezer storage stability studies for flusilazole and its metabolites in wheat. The 1991 JMPR reviewed a summary of a stability of flusilazole which reportedly demonstrated that residues in wheat grain were stable up to 36.5 months. The 1991 Meeting considered details of this summary as well as information on freezer storage stability of metabolites to be desirable. Instead of providing details of the previous summary, the manufacturer supplied an entirely new study (Desmond, 1993). Two 2-g samples of wheat or straw were fortified with each compoud and stored for intervals up to 11 months at -20◦C. Results (apparently uncorrected for procedural recoveries) are summarized in Table 6.

The study began before the development of analytical method AMR 2126-91. Although sample chromatograms were provided, they were not sufficiently labelled to be of much use. Control values were recorded as <0.05 mg/kg for all compounds in each matrix. Freshly fortified samples were anaylysed at the same time as the stored samples and results were generally similar for fresh and stored samples throughout the test period and showed similar variation. Differing recoveries therefore appear to have been more the result of analytical variation than of the effects of the storage.

Table 6. Average recoveries [1] of flusilazole and metabolites from wheat grain and straw after laboratory fortification and freezer storage (Desmond, 1993).

Storage Interval (Months)[2]	Percentage recovery from sample, stored/freshly fortified

	Flusilazole 0.27 mg/kg	IN-F7321[3] 0.26 mg/kg	IN-G7072[4] 0.26 mg/kg	IN-37722[5] 0.35 mg/kg	IN-37738[6] 0.27 mg/kg
Grain					
0	87/85	127/123	81/73	69/77	95/93
3	87/100	94/100	89/104	74/80	92/78
4	91/85	140 /123	79/73	74/77	102 /93
5	83/93	79/81	96/108	83/91	78/78
6	70/74	106 /58	73/77	68/86	119 /81
9	78/89	83/92	87/100	77/77	69/63
11	95/96	73/81	106/100	106/126	87/100
Straw					
0	81/85	79/77	92/92	97/69	85/81
3	100 /100	83/85	96/104	91/100	107/107
4	83/85	95/77	88/102	83/69	98/81
5	78/93	94/96	75/88	70/66	96/89
6	85/85	79/119	115/104	103 /109	96/137
9	80/85	125 /135	100/112	83/77	115 /115
11	74/78	81/62	89/92	107/80	83/63

[1]Values for stored samples are averages of two samples. Values for fresh fortification recoveries are single determinations.
[2]Interval from start of freezer storage to extraction.
[3]IN-7321 = bis(4-fluorophenyl)(methyl)silanol
[4]IN-G7072 (disiloxane) = 1,1,3,3-tetrakis(4-fluorophenyl)-1,3-dimethyldisiloxane
[5]IN-37722 = 2-fluoro-5-[(4-fluorophenyl)(methyl)(1-*H*-1,2,4-triazol-1-ylmethyl)silyl]phenol
[6]IN-37738 = 2-fluoro-5-[(4-fluorophenyl)(hydroxy)(methyl)silyl]phenol

Residues in the edible portion of food commodities

The reduction of flusilazole residues in barley milling fractions is discussed in "Fate of residues in processing" above.

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

No new information.

METHODS OF RESIDUE ANALYSIS

Most of the methods used for residue analyses have been described in earlier monographs, except du Pont methods AMR-115-85 and AMR 2126-91. Method AMR-115-85 is probably a modification of AMR-115-83, previously described. AMR-2126-91 (Koch, 1993) was used for the cereal analyses. It is capable of determining flusilazole and its major phenyl metabolites in wheat grain, straw and forage. It consists in homogenization of the hydrated sample with NaOH and 10% dichloromethane/ethyl acetate, centrifugation, concentration and the determination of flusilazole and the metabolite IN-G7072 by GLC with a mass-selective detector (GC-MSD). Another aliquot is reacted with diazomethane and analyzed by GC-MSD for IN-F7321, IN-37738 and IN-37722. In the case of forage an acetonitrile partition step is added after the initial extraction.

Average percentage recoveries from wheat samples fortified with flusilazole or metabolites at levels from 0.03 to 0.7 mg/kg average recoveries were:

	Forage	Grain	Straw
Flusilazole	97±21%	92±19%	81±13%

IN-G7072	94±11	107±19	100±18
IN-F7321	100±18	97±14	100±12
IN-37738	82±11	83±9	100±21
IN-37722	90±14	99±23	92±13

Sample chromatograms suggest that determinations of 0.05 mg/kg of flusilazole and the metabolites should be feasible in these samples, except possibly IN-F7321 in forage (owing to an interference peak) and flusilazole, IN-G7072 and IN-37722 in straw where 0.1 mg/kg may be more realistic for routine analyses. These judgements are based on a comparison of sample and control chromatograms at the lowest fortification levels. Detection is possible at lower levels.

During the application of the method in grain processing studies (see above) analytical recoveries of 90 ± 14% were reported for flusilazole in grain and its processed fractions at fortification levels mostly ranging from 0.05 to 0.2 mg/kg. Similar or better results were reported for the metabolites. Chromatograms suggest that quantification should be generally achievable at about 0.05 mg/kg for flusilazole and 0.1 mg/kg for the metabolites in these samples.

NATIONAL MAXIMUM RESIDUE LIMITS

The following national MRLs were reported to the Meeting:

Crop	Country	MRL, mg/kg
Banana	Australia	0.2
	The Netherlands	0.05 (under consideration)
Grapes	Australia	0.5
	Spain	0.05 (incl. wine grapes)
Pome fruit	Australia	0.2
	Spain	0.2
Stone fruit	Spain	0.1 (peach/apricot) 0.05 (other stone fruit)
Sugar cane	Australia	0.02
Commodities other than banana	The Netherlands	0* (0.05)

APPRAISAL

Flusilazole was previously reviewed for residues by the 1989, 1990 and 1991 Meetings. The present Meeting reviewed information provided in response to the 1991 JMPR requirement for additional GAP and residue data to confirm the 0.1 mg/kg temporary estimate for peaches and nectarines, and information listed as desirable on grapes, details of wheat grain freezer storage studies, stability of metabolites in freezer-stored grain samples, hen metabolism, metabolites in grain processed fractions and soil studies. Additional residue data on pome fruit, grapes and cereals (although there were no outstanding residue data requirements on these commodities) and new data on sugar cane were also provided.

Fate of residues in animals. Several reports on hen metabolism were provided. Some had been submitted before and some, including the requested study by Smyser, were new. Only the Smyser report included data in need of review by the Meeting.

The report basically combines and summarizes information in two previously reviewed reports and provides further clarification of the residues of metabolites, especially in terms of the percentage of the total radioactivity in poultry tissues and eggs, for both the phenyl and triazole labels. It confirms previous JMPR conclusions that bis(4-fluorophenyl)(methyl)silanol (IN-F7321, the methyl silanol) and 4-fluorophenyl(methyl)silanediol are the predominant residues in poultry tissues

and eggs arising from the phenyllabelled compound and that triazole is the main residue from the triazole label, except in fat where flusilazole is the primary residue from the triazole label.

The report does not effectively answer questions raised by the 1989 and 1991 Meetings concerning differences in residues found between ruminant and poultry metabolism and feeding studies. The Meeting noted and agreed with the 1991 JMPR conclusion that these differences probably result largely from the more detailed residue characterization and identification in the poultry studies than in the ruminant studies. The Meeting also agreed with the 1991 JMPR that although all questions have not been completely answered, the nature of the residue in animal products can be considered to be reasonably well understood in view of the low residues expected (especially for flusilazole) in animal products.

Soil dissipation. The Meeting reviewed the final report of a 3-year soil dissipation study (4 applications per year) for which an interim report was reviewed by the 1989 JMPR. It confirms the 1989 observations that over 92% of the radioactivity is confined to the top 8 cm of soil over the test period, and that the predominant residues in this segment are flusilazole and its silanol metabolite IN-F7321. The author cites statistical evaluation of the data to support the view that residues will reach a steady level at 57% of yearly application levels after repeated application levels under worst-case conditions.

The report cites the steady-state conclusion, the strong adsorption to the top layers of soil, the lack of residues exceeding 0.01 mg/kg in the 24-36 cm soil depths and the weak leaching potential indicated in other studies as evidence that residues in ground water were unlikely. While the data indicate that over 92% of the radioactivity remains in the top 8 cm of the silt loam soil investigated, and indeed that residue levels are extremely low in the 24-36 cm depths, it also shows an increasing penetration by low levels of radioactivity over the test period in this soil type. The identity of these residues in the deeper soil segments was not indicated.

While the adsorption of this persistent pesticide to soil is strong, the 1989 JMPR had noted that uptake of low residue levels can occur in rotational crops and that the leaching potential would be less for silt loams (as in this study) than for more sandy soils. Because the silt loam study was under worst-case conditions (bare ground, repeated applications) and was consistent with reassuring findings of a number of other relevant studies, the Meeting accepted that ground water residues from silt loam soils were unlikely.

Freezer storage stability. Instead of details of a previous 36.5-month study for the parent compound only, the Meeting was provided with a new 11-month freezer storage study of flusilazole and its metabolites in wheat grain and straw. While the results suggest that about 30% of 0.3 mg/kg residues of the parent compound and its phenyl metabolites in grain and straw are lost after various storage intervals up to 11 months, the variability in the recoveries of freshly fortified samples indicates that the apparent losses are probably as much the result of analytical variability as actual storage losses. The Meeting concluded that the data demonstrated adequate stability of flusilazole and the metabolites IN-7321, 1,1,3,3-tetrakis(4-fluorophenyl)-1,3-dimethyldisiloxane (IN-G7072), 2-fluoro-5-[(4-fluorophenyl)(methyl)(1-*H*-1,2,4-triazol-1-ylmethyl)silyl]phenol (IN-37722) and 2-fluoro-5-[(4-fluorophenyl)(hydroxy)(methyl)silyl]phenol (IN-37738) (presumably unconjugated) over 11 months under the conditions of the study.

The 11-month storage interval compares with sampling-to-laboratory-receipt intervals ranging from 2 to 15 months in cereal grain trials from which data were reviewed by the 1989 JMPR. The Meeting did not know the actual sampling-to-analysis intervals for the data reviewed in 1989, although according to the 1989 monograph all samples were generally stored at -20°C.

Cereals. The original 1989 JMPR estimates of maximum residue levels of 0.1

and 2 mg/kg respectively for cereal grains and straws or fodders (dry) were based on maximum residues of 0.07 mg/kg in grain and 1.7 mg/kg in straw. Although there were no outstanding requirements for additional supervised trials data, the Meeting received extensive additional cereal grain, plant, forage and straw data from Europe and North America. Because no need for MRL revisions was indicated, the Meeting only briefly summarized the submitted data on grain and straw. It concluded that there was no need to revise the recently adopted limits of 0.1 mg/kg in the grains and 2 mg/kg in the straws and fodders (dry) of barley, rye and wheat at present. This conclusion may need to be reconsidered at a future Meeting in the light of future GAP information.

Cereal grain processing. The 1991 JMPR reviewed a wheat processing study submitted in response to a 1989 requirement. While no concentration in milled fractions was observed, samples were not analysed for metabolites (especially IN-F7321) and such analysis had been recorded as desirable. A barley grain processing study provided to the Meeting confirmed that no concentration of flusilazole or the major metabolite IN-F7321 occurred in milling fractions.

Grapes. Limited additional information on GAP in Europe and Australia and additional grape data submitted in response to the 1991 requests showed maximum residues reflecting GAP of 0.22 mg/kg compared to the recently adopted CXL of 0.5 mg/kg. A delegation to the CCPR had suggested that a 0.2 mg/kg limit was sufficient. The Meeting confirmed the 1989 JMPR conclusion that residues were unlikely to exceed 0.3 mg/kg.

Pome fruit. Additional GAP information and residue data did not require a revision of the current 0.2 mg/kg limit.

Stone fruit. The 0.1 mg/kg limit for peaches and nectarines recommended by the 1991 JMPR was temporary pending the submission of additional GAP and residue data. It had been based on data from New Zealand and France and GAP from New Zealand and Spain. The Meeting received information on current GAP from Spain, France, Greece (pending) and Italy, and residue data on nectarines from France and on peaches from Australia, Italy, Greece, and the United States. French apricot data were also provided as supporting information. No GAP information was available for Australia or the United States. One to 4 applications at 3-4 g ai/hl and a PHI of 7 to 10 days appears to be usual for countries with established GAP, although in two cases the maximum number of permitted applications was not indicated.

At a 7-day PHI, the new French data or those summarized by the 1991 JMPR which reflect GAP rates showed maximum residues of flusilazole *per se* in peaches of 0.09 mg/kg (1991) or 0.08 mg/kg (1993), except in one trial in the 1993 submission where a residue of 0.55 mg/kg after 8 days was reported from 9 applications at GAP rates. Maximum apricot residues reflecting GAP rates were 0.08 mg/kg after 7 days. Maximum residues in the US trials were 0.09 mg/kg at a 2.4 g ai/hl spray concentration after 7 or 14 days (0.2 mg/kg after 5 days) and 0.3 mg/kg at a 4.8 g ai/hl rate after 12 days. At a pending GAP rate, maximum residues after 7 days in the Greek trials were 0.09 mg/kg. Residues were not detected in the Australian or Italian trials (<0.05 mg/kg and <0.01 mg/kg respectively), but that is not unexpected in view of the long PHIs and the type of application. The Meeting concluded that a 0.5 mg/kg limit was supported for peaches. Observing that GAP for apricots and nectarines is similar to that for peaches, the Meeting concluded that the available data could also mutually support 0.5 mg/kg limits for apricots and nectarines at a 7-day PHI.

Limited data for plums and cherries were insufficient to recommend MRLs.

Sugar cane. No residues (<0.02 mg/kg) were detected in the juice from plants grown after dip treatments of sugar cane sets at fivefold application rates. No stalks were analysed. The Meeting concluded that the data were inadequate to support a limit for sugar cane.

RECOMMENDATIONS

On the basis of the data on residues from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits.

Definition of the residue: flusilazole

CCN	Commodity	MRL (mg/kg)		PHI (days), on which recommendation is based
		New	Previous	
FS 0240	Apricot	0.5	-	7
FS 0245	Nectarine	W	0.1 T[1]	7
FS 0247	Peach	0.5	0.1 T[1]	7

[1]Temporary

FURTHER WORK OR INFORMATION

Desirable

1. Submission of analytical method AMR-115-85 cited in Du Pont, 1993, Vol. 1, exhibit 6. Submission of validation information to permit estimation of limits of determination is desirable.

2. On completion, submission of the soil dissipation report AMR-791-87 (Fujinari, 1988). The interim report was reviewed by the 1989 JMPR.

REFERENCES

Australia, 1993. Submission by the government of Australia to the 1993 JMPR. Appendix 24 - Volume 1 of 1 (grapes); Volume 2 of 2 (stone fruit); Volume 2 of 3 (pome fruit); Volume II of III (sugar cane); Appendix 25 -Australian DF label (grapes, apples, pears) and "Liquid Fungicide" label (sugar cane).

Bodden, R. and Kneeland, L. 1984. Metabolism of ^{14}C-DPX-H6573 in Laying Hens. Unpublished Du Pont Report AM-245-84 (see Du Pont 1993, Vol. 5).

Desmond, P.J. 1993. Freezer Storage Stability Study of Flusilazole Fungicide (DPX-H6573) and its Metabolites (IN-F6321, IN-G7072, IN-37722 and IN-37738) in Wheat Grain and Straw. Unpublished Du Pont Report No. AMR 2127-91 completed in response to US EPA data requirements. (See Du Pont, 1993, Vol. II, exhibit 2.

Du Pont, 1993. Supplemental Information on Flusilazole Prepared for the Food and Agriculture Organization:

March 10, 1993 Submission
Vol. I - Residue data and labelling for peaches, nectarines and apricots.
Vol. II - Residue data for cereals.
Vol. III - Residue data for cereals.
Vol. IV - Residue data for cereals.
Vol. V - Chicken metabolism.

April 30, 1993 Submission
Vol. VI - Residue data for peaches and soils and current labelling.

Individual reports are referenced in Table 2 as refs. 1-7 and Table 3 as refs. 1-8 as follows.

Table 2 (Cereals)

Ref.	Subject	Volume	Section	Country
	wheat			
1.	BF-66.630-03-88-19	III	3	Italy
	winter wheat			
2.	BG-BF-88-03	II	5	
	Germany			
	BG-BF-88-04	II	6	Germany
	BE-A-11-89-32-BG	IV	2	UK
	BG-BF-5122-09-89-06	III	3	Germany
	BG-BF-5122-09-89-07	III	4	Germany
	BF-66.630-03/08-88-11	IV	1	Germany
	BF-66.630-03/08-88-01	IV	3	UK
	BF-66.630-03/08-88-08	IV	2	
	Germany			
	BE-A-11-90-02-BG-BF	IV	4	Germany
	BE-A-11-90-03-BG-BF	IV	5	Germany
	AMR-1811-90	II	4	USA
	spring wheat			
3.	AMR-1811-90	II	4	USA
	spring barley			
4.	BE-A-11-89-32-BG	IV	2	
	UK			
	BF-66.630-03-88-01	IV	3	
	UK			
5.	AMR-1811-90	II	4	USA
	winter barley			
6.	BG-BF-88-03	II	5	Germany
	BG-BF-88-04	II	6	
	Germany			
	BE-A-11-89-32-BG	IV	2	
	UK			
	BF-66.630-03-88-01	IV	3	UK
	BF-66.630-03/08-88-08	IV	2	Germany
	BF-66.630-03/08-88-11	IV	1	Germany
	BE-A-11-90-02-BG-BF	IV	2	Germany
	BE-A-11-90-03-BG-BF	IV	5	Germany
	AMR-1811-90	II	4	USA

	winter rye		
7.	BG-BF-88-03	II	5
Germany			
	BG-BF-88-04	II	6
Germany			
	BE-A-11-90-03-BG-BF	IV	5
Germany			

Table 3 (Stone Fruit)

Ref.	Report No./exhibit
1	BE-A-11-89-25-BF/12
2	KO1REO1/13
3	See Australia, 1993. Australian peach data also submitted by du Pont as exhibit 6, Report AMR-1950-91.
4	BF-66.630-03-87-05/8
5	BE-A-11-89-15-BF/5
6	BE-A-11-92-22-BG/7 (du Pont, Vol. VI, 3/30/93).
7	Exhibit 14, Jan. 9, 1985, McIntosh
8	Exhibit 15, Jan. 22, 1985, McIntosh

Guinivan, R.A. and Desmond, P.J., 1993. Magnitude of Residue of Flusilazole in the Processed Fractions of Barley. Unpublished Du Pont Report No. AMR 1972-91 completed in response to US EPA data requirements. (See Du Pont, 1993, Vol. II, exhibit 3).

Koch, S., 1993. Method for the Analysis of Flusilazole and its Major Phenyl Metabolites in Wheat. Unpublished Du Pont Report No. AMR 2126-91, March 25, 1993.

Lin, P., 1988a. Metabolism Study of [Phenyl(U)-^{14}C]DPX-H6573 in Laying Hens. Unpublished Du Pont Report AMR-638-86-1 (see Du Pont, 1993, Vol. V).

Lin, P., 1988b. Supplement to: Metabolism Study of [Phenyl(U)-^{14}C]DPX-H6573 in Laying Hens. Unpublished Du Pont Report AMR-638-86-1 (see Du Pont, 1993, Vol. V).

Lin, P., 1988c. Metabolism Study of [Triazole-3-^{14}C]DPX-H6573 in Laying Hens. Unpublished Du Pont Report AMR-638-86-2 (see Du Pont, 1993, Vol. V).

Lin, P., 1988d. Supplement to: Metabolism Study of [Triazole-3-^{14}C]DPX-H6573 in Laying Hens. Unpublished Du Pont Report AMR-638-86-2 (see Du Pont, 1993, Vol. V).

Smyser, B., 1990. Supplement to: Metabolism Study of [Triazole-3-^{14}-C]DPX-H6573 and [Phenyl(U)DPX-H6573 in Laying Hens. Unpublished Du Pont Report AMR-638-86, Supplement No. 2 (see Du Pont 1993, Vol. V).

Smyser, B., 1993. Long-Term Terrestrial Field Dissipation of [Phenyl(U)-^{14}C]DPX-H6573 at Stine Farm, Delaware. Unpublished Du Pont Report No. AMR 556-86 (See Du Pont, 1993, Vol. VI, exhibit 6).

Stadalius, M., 1984. Supplement to: Metabolism of ^{14}C-DPX-H6573 in Laying Hens. Unpublished Du Pont Report AMR-245-84, MRID 40042130 (see Du Pont, 1993, Vol. V).

Spain, 1993. Documentation, Spanish GAP and Residues Studies in Apples (4), Pears (4), Peaches (7), Grapes (8). Spanish GAP for pome fruit, stone fruit and grapes. June 30, 1993 submission of the Ministerio de Agricultura, Pesca y

Alimentación:

Summary Reports only:

Peaches	Apricots	Grapes
BAT-86-02	BE-A-11-89-25-BF	CTB/E3-5122-02/MC/mf
BE-66.630-03-88-03		
CTB/E3.5122.02/MC/ns		
BE-A-11-89-15-BF		

Apples	Pears
BE-A-11-89-16-BF	BE-66.630-03-88-16
BAT-86-08	BE-A-11-91-03-BF
BE-A-11-89-22-BF	BF-66.630-03-88-18
BE-66.630-03-88-17	BAT-86-04

More Detailed Reports Grapes (US trials):

Test	FWM.JLP.83.8?, Manteca, CA	AAB.K88.83.4?, Madera, CA
	FWM.JLP.83.1?, Sanger, CA	FWM.JLH.83.9?, Riverbank, CA

Wustner, D. 1991. Residue Information on DPX-H-6573 (Flusilazole) Prepared for the World Health Organization, February 1991 - letter: Additional Residue Information for DPX-H6573 Flusilazole (Wustner to Kopisch, January 28, 1991). Exhibit 1 included - "Response to JMPR Review of Flusilazole Animal Metabolism and Feeding Studies (Koch to Wustner, February 27, 1991)" and references: Metabolism Study of Triazole-3-^{14}C-DPX-H6573 and Phenyl(U)-^{14}C-DPX-H6573 in Laying Hens (Smyser, B.P. 1990), unpublished E.I. du Pont de Nemours and Co., Inc. Document No. AMR-638-86, Supplement No. 2. This reference (Smyser, 1990) was requested by the 1991 JMPR).

FOLPET (41)

EXPLANATION

Folpet was first evaluated in 1969 and has been reviewed several times since, most recently in 1990. The 1987 JMPR recommended that a detailed review of all aspects of the use of folpet should be carried out at the 1989 Meeting or as soon as possible.

The 1990 Meeting required, by 1992, the results of supervised trials on apples, cherries, cucumbers, grapes, bulb onions, strawberries and tomatoes, as well as information on current GAP relevant to those crops and to the supervised trials. At the 23rd (1991) Session of the CCPR it was decided (ALINORM 91/24A, para 95) to propose withdrawal of the CXLs for blueberries; currants, black, red, white; raspberries, red, black, and watermelon, and to maintain the CXLs for all the other commodities, regarding them as temporary until 1992.

The 24th (1992) Session of the CCPR was informed that folpet was scheduled for toxicological review by the 1993 JMPR because of the temporary ADI (ALINORM 93/24, para 89). The CCPR was informed that residue studies on citrus fruits, lettuce, melons and potatoes were in progress and data would be available for the 1994 JMPR. The CCPR decided to maintain the CXLs as temporary for all commodities.

The 25th (1993) Session of the CCPR was informed that the manufacturer had provided information for all commodities with temporary MRLs except cherries and onions (ALINORM 93/24A, para 66). Folpet was on the agenda of the 1993 JMPR.

The basic manufacturer provided information to the Meeting on the registered uses of folpet and data from supervised trials on fruit and vegetable crops. MRLs for bulb onion and cherries will not be supported by new supervised field trials.

Information on GAP, residue trials and national MRLs was made available by Canada, the EEC, The Netherlands and Spain.

USE PATTERN

Folpet is a broad-spectrum fungicide used on both food and non-food crops. The major uses are on grapes, apples and various vegetables. The registered uses in many countries are summarized in Tables 1 and 2.

Table 1. Registered uses of folpet on fruits and nuts.

Crop	Country	Form	Application				PHI, days[1]
			Type	Rate per application, kg ai/ha	Spray concentration kg ai/hl	Number	
Almond	Greece	WP	foliar	1.5-3.2	0.1-0.12	3-4	7
Apple	Argentina	WP SC	foliar		0.1-0.12	3	20
Apple	Canada	WP	foliar	0.75-1.0			1
Apple	Greece	WP	foliar	2.0-3.2	0.1-0.13	4-5	7
Apple	Israel[2]	WP	foliar	2.0		every 14 days	21
Apple	Portugal	WP	foliar		0.13	10	21
Apple	Spain	WP	foliar	1.5	0.13	4-6	10
Apple	Spain	SC	dip or drench	-	0.1 (dip)	1	Post harvest 2 months
Blackberries	Netherlands	WP	foliar	1.3-1.6	0.13	4-6	4
Cherries	Netherlands	WP	foliar	1.3-2.0	0.13	2	4
Citrus	Israel[2]	WP	foliar		0.15		21
Cranberries	Canada	WP	foliar	5.0	0.25	2	30
Currants	Denmark	WP		1.5			21
Currants, red, white, black	Netherlands	WP	foliar	1.3-1.6	0.13	4-6	10
Grapes	Argentina	WP SC	foliar		0.1-0.13		7 T 20 W
Grapes	Canada	WP	foliar	1.0			1
Grapes	France	SC		1.5	0.15	2	28
Grapes	France			1.5	0.5-0.7	5-7	28
Grapes	Greece	SC	foliar	0.5-1.9	0.1-0.125	3	30
Grapes	Israel[2]	WP	foliar		0.15	every 14 days	8
Grapes	Italy	WP	foliar	0.35-0.40		3	10 T 40 W
Grapes	Portugal	WP	foliar	1.5-2	0.1-0.15	4-5	21 T 42 W
Grapes	Spain	WP	foliar	1.5-2	0.13-0.20	4	21
Medlar	Portugal	WP	foliar		0.125	3-4	21
Olive	Spain	WP	foliar	1.6	0.16		10
Peach	France	WP	foliar		0.15		15
Pear	Portugal	WP	foliar		0.13	10	21
Pear	Spain	SC	dip or drench		0.1 (dip)	1	Post-harvest 2 months
Pome fruit	Denmark	WP	foliar	1.3-2			21
Pome fruit	France			1.5	0.1	4-5	14
Pome fruit	Italy	WP	foliar	0.35-0.4		3	10
Pome fruit	Spain	WP	foliar	2.4	0.16		10
Stone fruit	Denmark	WP	foliar	1.25-2.0			21
Stone fruit	Greece	WP	foliar	1.5-3.0	0.1-0.13	3-4	7
Stone fruit	Italy	WP	foliar	0.35-0.40		3	10
Stone fruit	Spain	WP	foliar	2.4	0.16	3	10
Strawberry	Belgium	WP	pl dr[3]	-	0.048	1	56
Strawberry	Belgium	WP	foliar	0.6			56
Strawberry	Brazil	WP	foliar	0.35-0.54	0.14		1
Strawberry	Canada	WP	foliar	1.0			1
Strawberry	Denmark	WP	foliar	3			21

Crop	Country	Form	Application				PHI, days[1]
			Type	Rate per application, kg ai/ha	Spray concentration kg ai/hl	Number	
Strawberry	France	SC		4			30
Strawberry	Israel[2]	WP	foliar	2.0		weekly	17
Strawberry	Netherlands	WP	foliar	0.85-1.3	0.13	4-6	4
Strawberry	Netherlands	WP	foliar	0.85-1.3	0.13	2G[4]	14
Strawberry	Portugal	WP	foliar		0.1-0.15	3	7
Strawberry	Spain	WP	foliar	1-1.5	0.15	4	21

[1] T: table grapes. W: wine grapes.
[2] Proposed regiestration.
[3] Drench or dip at planting
[4] Glasshouse use.

Table 2. Registered uses of folpet on vegetables and cereals. All foliar applications.

Crop	Country	Form	Application			PHI, days
			Rate per application, kg ai/ha	Spray concentration kg ai/hl	Number	
Barley	France		1.5			21
Beans	Greece	WP	0.6-1.5	0.1-0.25	3-4	7
Beans	Portugal	WP		0.13	1-2	7
Beans, green	Spain	WP	1.6	0.16		21
Brassica vegetables	Italy	WP	0.35-0.40			10
Brassica vegetables	Spain	WP	0.75	0.075		7
Broad beans	Spain	WP	1.6	0.16		10
Bulb vegetables	Italy	WP	0.35-0.40			10
Bulb vegetables	Spain	WP	0.75	0.075		7
Carrots	Greece	WP	0.6-1.3	0.1-0.13	3-4	7
Chickpeas	Spain	WP	1.6	0.16		10
Cucumber	Canada	WP	1.0-2.0			1
Cucumber, gherkin	France		0.5-1			3
Cucumber	Mexico	WP	1.2-1.7			0
Cucurbits	Greece	WP	0.45-1.9	0.075-0.13	3	7
Cucurbits	Italy	WP	0.35-0.40			10
Cucurbits	Spain	WP	0.75	0.075		7
Egg plant	Greece	WP	0.45-1.3	0.075-0.13	3-4	7
Egg plant	Spain	WP	1.6	0.16		10
Leeks	Greece	WP	0.45-1.3	0.025-0.13	3-4	7
Legume vegetables	Italy	WP	0.35-0.40			10
Lettuce	Brazil	WP	0.34-0.54	0.14		1
Lettuce	France	WP	0.64			21, 41[1]
Lettuce	Israel[2]	WP	2.0		weekly	11
Lettuce	Italy	WP	0.35-0.40			10
Lettuce	Portugal	WP		0.13	1-2	14
Lettuce	Spain	WP	0.75-1.5	0.13-0.16	4	21
Melon	Canada	WP	1.0-2.0			1
Melon	France			0.15		8
Melon	Israel[2]	WP	2.0		weekly	7
Melon	Portugal	WP		0.13-0.15	1-3	28
Onion	Greece	WP	0.45-1.3	0.075-0.13	3-4	7
Onion	Portugal	WP		0.1-0.13	2-3	7
Peas	France		1.5	0.1-0.25	3	15
Peas	Greece	WP	0.6-1.5		3-4	7
Peas, green	Spain	WP	1.6	0.16		10
Peppers	Greece	WP	0.45-1.3	0.075-0.13	3-4	7
Potato	France		1.5		7	7-15
Potato	Greece	WP	0.45-1.3	0.075-0.13	3-4	7
Potato	Israel[2]	WP		0.15	weekly	14
Potato	Portugal	WP		0.13	5-6	7
Potato	Spain	WP	1-1.2	0.15	4	10
Potato	Uruguay	WP SC		0.13		0
Pumpkin	Canada	WP	1.0-2.0			1
Root and tuber vegetables	Italy	WP	0.35-0.40		3	10
Root and tuber vegetables	Spain	WP	0.75	0.075		7

Crop	Country	Form	Application			PHI, days
			Rate per application, kg ai/ha	Spray concentration kg ai/hl	Number	
Solanaceae	Italy	WP	0.35-0.40			10
Solanaceae	Spain	WP	0.75	0.075		7
Squash	Canada	WP	1.0-2.0			1
Stem vegetables	Italy	WP	0.35-0.40			10
Stem vegetables	Spain	WP	0.75	0.075		7
Tomato	Canada	WP	2.0			1
Tomato	France		0.5-1			3
Tomato	Greece	WP	0.45-1.3	0.075-0.13	3-4	7
Tomato	Hungary	WP	1.3	0.25		14
Tomato	Israel[2]	WP	2.0		weekly	14
Tomato	Mexico	WP	1.5-2.0			0
Tomato	Portugal	WP		0.13	2, 6 G[3]	7
Tomato	Spain	WP	1-1.5	0.13-0.5	4	10
Tomato	Uruguay	WP SC	1.3			0
Watermelon	Portugal	WP		0.13	1-2	7
Wheat	France		1.5			21
Witloof	Italy	WP	0.35-0.40			10
Witloof	Spain	WP	0.75-1.6	0.075-0.16		7-21

[1] Summar 21 days, winter 41 days.
[2] Proposed registration.
[3] Glasshouse use.

RESIDUES RESULTING FROM SUPERVISED TRIALS

Residues of folpet and phthalimide, a metabolite, found in supervised trials on horticultural crops are shown in Tables 3 to 7.

Table 3. Mandarins, oranges. *Spain, Israel.*
Table 4. Apples. *Chile, France, Israel, Portugal.*
Table 5. Grapes. *Argentina, Chile, France, Israel, Italy, Spain.*
Table 6. Strawberries. *Brazil, Hungary, Israel, Spain, Uruguay.*
Table 7. Melons, squash, lettuce, potatoes, tomatoes. *Brazil, Denmark, France, Greece, Hungary, Israel, Netherlands, Spain, UK, Uruguay.*

Most supervised residue trials were fully or adequately described. Residues in the Tables are not adjusted for analytical recoveries. Since recoveries were mostly in the 80-120% range, this should not influence interpretations. Attention is drawn to cases where recoveries were excessively high or less than 70%.

Where residues were not detected, they are recorded in the Tables as less than the limit of detection or the limit of determination e.g. <0.1 mg/kg. Residues have generally been rounded to 2 significant figures or, near the limit of determination, to 1 significant figure. Residues were not detected in control samples, but controls are not included in the Tables. Most of the trials were based on three or four replicate plots and the analyses of samples from each replicate are recorded in the Tables.

In the laboratory reports from trials in Argentina, Brazil and Uruguay there was some confusion between the units of concentration ng/kg, ìg/kg and mg/kg. Limits of determination were not clearly expressed but appeared to be 0.01 mg/kg and 0.1 mg/kg for folpet and phthalimide respectively in most cases.

Plot sizes in the Argentine trials were grapes 300-500 m^2, apples 4 trees.

Plot sizes in the Brazilian trials were strawberries 61 plants, lettuce 133 plants. Lettuces were irrigated by sprinkler 5 days after transplanting, and then every 3 days. Strawberries were sprinkler-irrigated once a week.

In Chile plot sizes for grapes and apples were 4 rows of 12 m and 18 trees respectively.

In France the experimental plot sizes were grapes 20-120 m^2, apples 3 or 5 trees, tomatoes 22 m^2, tomatoes under glass 18 plants, and cantaloupes 30 plants. Folpet was applied to grapes using a pneumatic sprayer. Analysis of cantaloupes (melons) was on a whole-fruit basis and no residues were detected (<0.01 mg/kg). The laboratory reported low recoveries from tomatoes (36%) and melons (15%) if the samples were spiked before chopping, but good recoveries (tomatoes 107% and 100%, melons 98%) if the samples were spiked immediately before analysis. No laboratory reports were available for the French trials on apples (FR104-91 and FR105-91) or lettuce (FR108/91).

The experimental plot size for the squash trials in Greece was 15 m^2. Folpet was applied to the strawberry plots (300 m^2) in Hungary with a back-pack sprayer, and to the tomato plots (200 m^2) with a 10 m boom spray. Recoveries were generally good except that at the LOD in tomato there was one low recovery of folpet and one of phthalimide and in strawberries, also at the LOD, one folpet result was very high (175%) and one phthalimide was low (<70%).

Folpet was applied to experimental plots of lettuce, potatoes, tomatoes, melons, grapes and strawberries in Israel with a motorised back-pack sprayer, and to apples and citrus with motorised orchard-spraying equipment. Plot sizes for lettuce, potatoes, tomatoes and melons were 50-60 m^2, for strawberries 14 m^2, for apples and citrus 18 trees, and for grapes 18 vines. Recoveries were generally satisfactory except for individual recoveries at the LOD which could be marginally low or too high. In melons recoveries of phthalimide at higher levels (2 mg/kg) were marginally low.

In the Italian grape trials folpet was applied to plots of 160-370 m^2. Recoveries were satisfactory, but those of phthalimide appear to have been tested only at the relatively high level of 2 mg/kg.

A gas-powered knapsack sprayer was used to apply folpet in the Netherlands potato trials. The plot size was 25 m^2. In Denmark a gas-pressurised 3 m boom spray was used for potatoes; the plots were 36 m^2.

In apple trials in Portugal the plot size was 6 trees. The trees were treated using a motor-driven sprayer with hand-gun application.

In Spain folpet was applied with a knapsack sprayer to experimental plots of grapes (75 m^2), strawberries (204 plants), and melons (44 plants). In citrus trials an experimental plot comprised 30 trees. The reports of the citrus trials in Spain in 1991 were vague about the LODs of folpet and phthalimide. The limits were assumed to be 0.02 and 0.1 mg/kg respectively.

In Uruguay strawberry plots were 4.2 $\times$ 20 m and potato plots were 5.6 $\times$ 20 m. Folpet was applied with a knapsack sprayer.

There were 6 replicates in the UK potato trials and each plot was 2 $\times$ 6 m. Folpet was applied with a boom spray.

Table 3. Folpet residues in citrus fruit from supervised trials in Israel and Spain.

Country, year (Variety)	Application				Day	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		Folpet	Phthalimide	
Mandarins								
Spain, 1991	WP	4.5	0.15	1	0 7	1.1, 0.47, 1.4 0.13, 0.14, 0.21	0.1 (2), <0.1 <0.1 (3)	203/91

Country, year (Variety)	Application				Day	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		Folpet	Phthalimide	
(Clementine)					14	0.15, 0.08, 0.14	<0.1 (3)	
					21	<0.02, 0.02, 0.09	<0.1 (3)	
					28	0.02, <0.02, 0.07	<0.1 (3)	
					42	<0.02, <0.02, <0.02	<0.1 (3)	
					56	<0.02, 0.05, 0.05	<0.1 (3)	
Spain, 1988 (Clementine)	SC	6.0	0.13	1	0	0.98, 1.2, 1.2		MAPA 7/5/91
					7	0.93, 0.88, 0.82		
					14	0.77, 0.75, 0.65		
					21	0.75, 0.67, 0.65		
					28	0.50, 0.70, 0.68		
Oranges								
Israel, 1992 (Shamuti)	WP	14	0.14	1	0	3.0, 2.8, 3.1, 3.8	<0.1 (4)	FP/29/92
					7	1.0, 2.0, 1.6, 1.6	<0.1, 0.1, <0.1 (2)	
					23	1.3, 1.0, 1.3, 0.64	<0.1 (4)	
Israel, 1992 (Shamuti)	WP	29	0.29	1	0	7.1, 9.9, 7.6, 4.1	0.1 (3), <0.1	FP/29/92
					7	2.9, 2.2, 1.9, 2.1	<0.1 (4)	
					23	1.5, 2.5, 3.1, 2.6	<0.1 (4)	
Spain, 1988 (Navel)	SC	6.5	0.13	1	0	0.40, 0.38		MAPA 7/5/91
					7	0.20, 0.18, 0.20		
					14	0.10, 0.09, 0.09		
					21	0.09, 0.12, 0.08		
					28	0.05, 0.10, 0.10		
					42	0.10, 0.09, 0.09		
					56	0.08, 0.10, 0.08		
Spain, 1988 (Navel)	SC	6.5	0.13	1	0	0.37, 0.25, 0.39		MAPA 7/5/91
					7	0.20, 0.22, 0.27		
					14	0.11, 0.33, 0.38		
					21	0.18, 0.21, 0.40		
					28	0.19, 0.24, 0.10		
					42	0.17, 0.24, 0.14		
					56	0.14, 0.12, 0.06		
Spain, 1991 (Navelina)	WP	4.5	0.15	1	0	0.67, 0.44, 0.78	<0.1 (3)	204-91
					7	0.60, 0.43, 0.40	0.1, <0.01, 0.1	
					14	0.45, 0.42, 0.42	0.1 (3)	
					21	0.35, 0.39, 0.23	0.1 (2), <0.1	
					28	0.24, 0.21, 0.14	0.1 (2), <0.1	
					42	0.18, 0.14, 0.14	0.1, <0.1 (2)	
					56	0.11, 0.24, 0.18	<0.1, 0.1 (2)	

Table 4. Folpet residues in apples from supervised trials in Chile, Israel, France and Portugal. Underlined residues are from treatments within GAP.

Country, year (Variety)	Application				Day	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		Folpet	Phthalimide	
Chile, 1992 (Red King Oregon)	WP	4.4-7.2	0.24	3	120	0.37, 0.65, 0.28, 0.35	<0.1 (4)	7538/7779
France, 1991 (Golden Delicious)	WP	2.5	1.0	13	3	0.20, 0.20		FR104-91
					22	0.12, 0.10		
					42	0.55		
France, 1991 (Golden Delicious)	WP	5.0	2.0	13	3	0.55, 0.55		FR104-91
					22	0.40, 0.32		
					42	0.25		
France, 1991 (Golden Delicious)	WP	1.3	0.10	10	0	0.23, 0.05, 0.08, 0.02		FR105-91
France, 1991 (Golden Delicious)	WP	2.6	0.20	10	0	0.47, 0.13, 0.13, 0.13		FR105-91
					20	0.005, 0.01 (2), 0.005		
					41	0.005, 0.01 (2), 0.005		
Israel, 1991 (Starking)	WP	2.9	0.14	4	0	2.2, 4.7, 3.4, 2.1	<0.1 (4)	FP/24/91
					23	<u>0.92</u>, <u>0.98</u>, <u>1.4</u>, <u>0.77</u>	<0.1 (4)	
					36	<u>0.82</u>, <u>0.70</u>, <u>0.50</u>, <u>0.56</u>	<0.1 (4)	
				5	7	1.4, 3.0, 1.7, 3.3	<0.1 (4)	
Israel, 1991 (Starking)	WP	5.8	0.29	4	0	8.1, 11, 6.9, 11	0.1 (2), 0.26, 0.23	FP/24/91
					23	1.9, 4.9, 4.3, 4.8	<0.1 (4)	
					36	1.3, 0.49, 0.83, 1.7	<0.1 (4)	
				5	7	5.3, 5.1, 4.8, 7.1	<0.1 (4)	
Portugal, 1991 (Golden Delicious)	WP	1.1-1.3	0.13	10	0	3.0, 1.9, 1.9, 2.9	<0.1 (4)	FP/25/91
					10	1.7, 0.71, 0.85, 1.9	<0.1 (4)	

Country, year (Variety)	Application				Day	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		Folpet	Phthalimide	
					21	0.97, 1.4, 0.94, 1.8	<0.1 (4)	

Table 5. Folpet residues in grapes from supervised trials in Argentina, Chile, France, Israel, Italy and Spain. Underlined residues are from treatments within GAP.

Country, year (Variety)	Application				Day	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		Folpet	Phthalimide	
Argentina, 1992 (Pedro Ximinéz)	WP	1.3	0.12	3	20	0.057, 0.055, 0.063, <0.01	0.10, <0.1, 0.13, <0.1	JV-wine grape
Argentina, 1992 (Black Cherry)	WP	1.7	0.12	5	7	0.26, 0.57, 0.25	<0.1 (3)	SJ-table grape
Chile, 1992 (Thompson)	WP	2.9-3.6	0.24	4	15	25, 23, 19	1.9, 1.7, 1.9	7538/7779
France, 1991 (Merlot Noir)	SC	1.5	1.0	10	0 11 21 33	2.0, 1.1, 0.35, 0.73 2.1, 2.0, 0.85, 1.3 0.56, 1.7, 0.59, 0.95 0.65, 0.75, 0.68, 0.31	1.1, 0.93, 0.44, 0.45 0.58, 0.88, 0.32, 0.53 0.58, 0.96, 0.32, 0.64 0.21, 0.30, 0.36, 0.25	101/91
France, 1991 (Merlot Noir)	SC	3.0	2.0	10	33	6.0, 8.0, 1.9, 2.0	1.9, 1.5, 1.0, 1.3	101/91
France, 1991 (Gamay Noir)	SC	1.5	1.1	7	0 10 21 45	1.1, 1.2, 2.7, 2.8 1.3, 1.3, 0.7, 0.78 1.2, 1.1, 0.70, 0.79 0.66, 0.27, 0.33, 0.41	0.84, 0.75, 1.0, 1.2 0.33, 0.45, 0.20, 0.41 0.79, 0.83, 0.99, 0.26 0.40, 0.57, 0.56, 0.88	102/91
France, 1991 (Grenache)	WP	1.5	1.9	7	0 10 21 43	0.78, 1.2, 2.0, 0.8 2.2, 1.3, 1.4, 0.52 1.3, 0.41, 0.11, 0.6 0.062, 0.19, 0.097, 0.075	0.64, 0.64, 0.59, 0.64 0.48, 0.53, 1.9, 0.48 0.51, 0.32, 0.19, 0.47 <0.05, 0.2, 0.43, 0.23	103/91
Israel, 1991 (Thompson)	WP	1.4	0.14	3	0 7 14 22	3.2, 1.2, 1.2, 2.0 0.94, 0.21, 0.56, 0.28 1.4, 0.47, 0.27, 0.38 0.66, 0.26, 0.14, 0.14	<0.1 (4) <0.1 (4) <0.1 (4) <0.1 (4)	FP/20/91
Israel, 1991 (Thompson)	WP	2.9	0.29	3	0 7 14 22	4.5, 1.5, 0.92, 1.9 0.96, 0.85, 0.51, 1.4 0.68, 0.69, 1.2, 0.62 1.2, 0.25, 0.32, 0.14	<0.1 (4) <0.1 (4) <0.1 (4) <0.1 (4)	FP/20/91
Italy, 1991 (Dolcetto)	WP	1.5	0.25	7	0 10 20 40	0.82, 0.74, 1.1, 0.79 0.60, 0.40, 0.75, 0.40 0.23, 0.17, 0.16, 0.20 0.12, 0.08, 0.07, 0.15	<0.05 (4) <0.05 (4) <0.05 (4) <0.05 (4)	IT-302-91
Italy, 1991 (Dolcetto)	WP	3.0	0.50	7	0 40	2.6, 5.0, 3.9, 5.9 0.57, 0.50, 0.60, 0.35	0.19, 0.14, 0.17, 0.11 <0.05 (4)	IT-302-91
Italy, 1991 (Merlot)	WP	1.5	0.15	10	0 10 20 40	0.94, 1.3, 0.48, 0.85 0.30, 0.51, 0.30, 0.58 0.17, 0.17, 0.06, 0.10 0.04, 0.03, 0.03, 0.04	<0.05 (4) <0.05 (4) <0.05 (4) <0.05 (4)	IT-301-91
Italy, 1991 (Merlot)	WP	3.0	0.30	10	0 40	2.0, 2.2, 2.6, 2.0 1.2, 0.24, 0.13, 0.13	0.29, 0.40, 0.14, 0.11 <0.05 (4)	IT-301-91
Spain, 1991 (Cabernet Sauvignon)	WP	0.8	0.2	3	0 10 20	6.0, 6.5, 6.0, 7.0 2.2, 1.1, 2.0, 1.4 1.3, 0.6, 2.0, 0.9	0.5, 0.5, 0.6, 0.55 0.55, 0.3, 0.4, 0.4	SP-201-91

Table 6. Folpet residues in strawberries from supervised trials in Brazil, Hungary, Israel, Spain and Uruguay. Underlined residues are from treatments within GAP.

Country, year (Variety)	Application				Day	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		Folpet	Phthalimide	
Brazil, 1991 (AGF-80)	WP	0.14	0.02	4	1	1.4, 1.1, 0.24, 0.33	5.5, 3.0, 1.1, 1.6	BRSTMF
Brazil, 1991 (AGF-80)	WP	0.27	0.04	4	1	1.7, 1.1, 1.8, 1.7	1.7, <0.1, 1.2, 2.6	BRSTMF
Hungary, 1991 (Gorella)	WP	1.0	0.10	2 3	14 0 5 10	0.55, 0.21, 0.25, 0.16 0.98, 0.83, 0.83, 0.96 0.47, 0.78, 0.21, 0.34 0.18, 0.22, 0.22, 0.35, 0.21, 0.18, 0.14	<0.1 (4) <0.1 (4) <0.1 (4) <0.1 (7)	FP/27/91
Israel, 1991 (Dorit)	WP	1.4	0.14	3	0 17 24 31	5.8, 7.1, 5.4, 5.6 4.7, 2.4, 4.8, 4.4 2.7, 3.0, 2.4, 0.5 1.7, 2.6, 2.3, 4.2	0.1, <0.1, 0.1, <0.1 <0.1 (4) <0.1 (4) <0.1 (4)	FP/28/92
Israel, 1991 (Dorit)	WP	2.9	0.29	3	0 17 24 31	21, 8.9, 12, 8.6 5.9, 7.6, 10, 6.8 4.4, 3.3, 3.8, 4.5 4.6, 6.4, 5.1, 4.8	0.1, <0.1, 0.1 (2) <0.1 (4) <0.1 (4) <0.1 (4)	FP/28/92
Spain, 1991 (Pájaro)	WP	1.29	0.15	4	0 12 21	1.7, 1.8, 1.0, 1.2 0.64, 1.8, 0.84, 1.0 0.46, 1.1, 0.51, 0.80	1.9, 2.5, 0.96, 1.5 0.68, 0.34, 0.37, 0.4 0.57, 0.87, 0.49, 0.3	
Uruguay, 1991 (Selva)	WP	0.25	0.03	10	7	0.95, 0.7, <0.1 (2)	<0.2 (2), 17, <0.2	#15

Table 7. Folpet residues in vegetables from supervised trials in Brazil, Denmark, France, Greece, Hungary, Israel, The Netherlands, Spain, UK and Uruguay. Underlined residues are from treatments within GAP.

Crop Country, year (Variety)	Application [1]				Day	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		Folpet	Phthalimide	
Cucurbits - melons								
France, 1991 (Alpha)	SC	1.5	0.43	3	0 1 3 5 7	<0.01 (2) <0.01 <0.01 (4) <0.01 <0.01		FR109-91
Israel, 1991 (Ein - Dor Ananas)	WP	2.0	0.66	6	0 7 14	0.56, 0.20, 1.7, 1.8 0.32, 0.37, 0.66, 0.28 0.30, 0.15, 0.72, 0.18	0.1 (2), 0.36, 0.1 0.24, 0.1 (3) 0.22, 0.1, 0.24, <0.1	FP/19/91
Israel, 1991 (Ein - Dor Ananas)	WP	4.0	1.3	6	0 7 14	2.8, 0.97, 0.75, 0.77 0.78, 0.32, 1.5, 1.8 0.44, 0.60, 0.25, 0.08	0.34, 0.1 (3) 0.30, 0.22, 0.1, 0.23 0.23, 0.26, 0.29, 0.1	FP/19/91
Spain	WP	1.3	1.6	3	0 6 12	pulp <0.02 (4) pulp <0.02 (4) pulp <0.02 (4)		

Crop Country, year (Variety)	Application [1]				Day	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		Folpet	Phthalimide	
Cucurbits - squash								
Greece, 1991 (Jedida)	SC	1.1	0.66	2	0 10 20	0.81, 0.22, 0.40, 0.37 <0.02 (4) <0.02 (4)	0.1, <0.1 (3) <0.1 (4) <0.1 (4)	FP/21/91
Greece, 1991 (Jedida)	SC	2.3	1.3	2	0 10 20	0.56, 0.55, 0.60, 0.73 <0.02 (4) <0.02 (4)	<0.1 (2), 0.1, <0.1 <0.1 (4) <0.1 (4)	FP/21/91
Lettuce								
Brazil, 1991 (Floreta)	WP	0.9	0.15	4	7	0.76, 0.95, 1.4, 0.91	0.91, 0.67, 1.0, 1.3	BRLPTMF
Brazil, 1991 (Floreta)	WP	1.8	0.30	4	7	1.4, 1.9, 2.3, 1.4	0.73, 0.49, 0.71, 1.0	BRLPTMF
France, 1991 (Aprilia)	SC	0.75	0.075	3	1 11 15	4.0, 3.5, 3.8, 3.4 0.1, 0.05, 0.1, <0.01 0.03, 0.15, 0.01, 0.05		FR108/91
Israel, 1991 (Nogah)	WP	2.0	0.50	3	0 11	28, 24, 12, 19 3.3, 2.8, 4.1, 3.3	0.86, 1.0, 0.58, 0.72 0.13, 0.1, 0.13, 0.23	FP/17/91
Israel, 1991 (Nogah)	WP	4.0	1.0	3	0 11	49, 49, 29, 56 11, 10, 6.9, 7.2	1.7, 1.4, 0.94, 1.2 0.41, 0.32, 0.25, 0.29	FP/17/91
Potato								
Denmark, 1991 (Bintje)	WG	2.4	1.0	6	0 7 14	<0.02 (3) <0.02 (3) <0.02 (3)	<0.1 (3) <0.1 (3) <0.1 (3)	FP/22/91
Israel, 1991 (Desire)	WP	2.0	0.50	6	0 14	0.02 (4) <0.02 (4)	<0.1 (4) <0.1 (4)	FP/18/91
Israel, 1991 (Desire)	WP	4.0	1.0	6	0 14	0.13, 0.02, 0.05, 0.02 <0.02 (4)	<0.1 (4) <0.1 (4)	FP/18/91
Netherlands, 1991 (Maritiema)	SC	1.9	0.32	11	0 13	<0.02 (4) <0.02 (4)	<0.1 (4) <0.1 (4)	FP/23/91
Netherlands, 1991 (Maritiema)	SC	3.8	0.64	11	0 13	<0.02 (4) <0.02 (4)	<0.1 (4) <0.1 (4)	FP/23/91
UK, 1991	SC	1.3	0.50	6	0 20	<0.01 (4) <0.01 (4)	<0.01 (4) <0.01 (4)	R52593
UK, 1991	SC	2.5	1.0	6	0 20	<0.01 (4) <0.01 (4)	<0.01 (4) <0.01 (4)	R52593
UK, 1991	SC	1.3	0.50	5	0 47	<0.01 (4) <0.01 (4)	<0.01 (4) <0.01 (4)	R52593
UK, 1991	SC	2.5	1.0	5	0 47	<0.01 (4) <0.01 (4)	<0.01 (4) <0.01 (4)	R52593
Uruguay, 1992 (Kennebec)	WP	0.23	0.03	7	7	<0.1, 0.1, 0.49	<0.1 (2)	#14
TOMATO								
France, 1991 (Ferline)	SC	1.5	0.50	3	0 3 6 10	0.08, 0.4 0.03 (3), 0.07 <0.02, 0.06, 0.13 <0.02, 0.02, 0.05		FR106/91
France, 1991 (Ferline)	SC	3.0	1.0	3	0 3 6 10	0.04 0.14 0.13 0.05		FR106/91

Crop Country, year (Variety)	Application [1]				Day	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		Folpet	Phthalimide	
France, 1991 (Trente)	SC	1.5	0.50	g 1	0 4 6 10	0.10, <0.01, 0.01 (2) 0.15, <0.01, 0.01, 0.08 0.15, 0.03, 0.05, 0.01 0.01, 0.05, 0.01, 0.04	0.05 (3), 0.10 0.10, <0.05, 0.05, 0.08 0.10, 0.05 (2), <0.05 <0.05, 0.10, <0.05, 0.06	FR110-91
France, 1991 (Trente)	SC	3.0	1.0	g 1	0 4 6 10	0.30 0.10 0.45 0.40	0.15 0.10 0.12 0.12	FR110-91
France, 1991 (Trente)	SC	1.5	0.50	g 3	4	1.0, 1.2, 0.25, 0.20	1.2, 0.4, 0.20, 0.15	FR110-91
Hungary, 1991 (Korall)	WP	0.63	0.12	5	0 7 14	0.05, 0.06, 0.06, 0.05 <0.02 (4) <0.02 (4)	<0.1 (4) <0.1 (4) <0.1 (4)	FP/26/91
Israel, 1991 (Var 182)	WP	2.0	1.0	3	0 4 11	0.33, 0.38, 0.71, 0.47 0.28, 0.29, 0.26, 0.10 0.21, 0.18, 0.17, 0.17	0.1 (4) 0.1 (4) 0.1 (4)	FP/16/91
Israel, 1991 (Var 182)	WP	4.0	2.0	3	0 4 11	0.80, 1.3, 0.92, 1.0 0.84, 0.65, 0.53, 0.43 0.23, 0.26, 0.30, 0.23	0.22, 0.25, 0.25, 0.22 0.24, 0.1 (3) 0.1 (4)	FP/16/91

g: glasshouse use (Tomato, column 5).

FATE OF RESIDUES

Stability of pesticide residues in stored analytical samples

Schlesinger (1991a, FP/15/91) tested the storage stability of folpet and phthalimide residues in analytical samples (25 g) separately fortified with each compound at 1 mg/kg and then held in plastic bags in a freezer at -18◦C. The results (Table 8) suggest that 10-20% of the residues may be lost during 6 months freezer storage; analytical variation makes it difficult to be precise.

Table 8. Stability of folpet and phthalimide residues in analytical samples fortified at 1 mg/kg and held in frozen storage at -18◦C (Schlesinger, 1991a, FP/15/91).

Commodity	Storage interval, months	% remaining

		folpet	phthalimide
Lettuce	6	91	76
	7	87, 100, 112	73, 71, 74
Potato	6	92	57
	7	58, 59, 76	53, 49
Tomato	6	68	67
	7	78, 63, 81	61, 63
Melon	5	64	-
	6	68, 58	57
	7	-	67, 75

METHODS OF RESIDUE ANALYSIS

Schlesinger (1991a, FP/15/91) described in detail a method for the analysis of folpet and its metabolite phthalimide in non-oily crops.

Samples were chopped and extracted with ethyl acetate (+ sodium sulphate and phosphoric acid), then cleaned up on a Florisil column for folpet, or by solvent partition (hexane, phosphate buffer) for phthalimide. Gas liquid chromatography on a megabore column was used for the final determination, with a ^{63}Ni electron-capture detector for folpet and a thermionic nitrogen-specific detector for phthalimide.

The method was validated for apples, grapes, strawberries, lettuce, melons, potatoes, squash and tomatoes. Recoveries were determined at levels of 0.02, 0.05, 0.25 mg/kg and higher (folpet) and 0.1, 0.2, 1.0 and 2 mg/kg (phthalimide) with 11 or 12 analyses on each crop. Mean recoveries from each crop were 89-100% for folpet and 75-96% for phthalimide. Individual recoveries, except 2, were in the ranges 60-135% for folpet and 60-130% for phthalimide.

Limits of determination were 0.05 mg/kg for folpet and 0.2 mg/kg for phthalimide. Limits of detection were lower by factors of 2-2.5.

No interference was caused by 25 common pesticides which might occur in crop samples.

This method and variations of it were used for the supervised residue trials. Acetone was used as an extracting solvent. A mixed activated carbon and silica gel column assisted the clean-up of folpet. Florisil was used for phthalimide clean-up.

Folpet is included in the lists of compounds determined by Multi-Residue Methods 1 and 12 published by the Ministry of Welfare, Health and Cultural Affairs, The Netherlands (1988).

NATIONAL MAXIMUM RESIDUE LIMITS

The Meeting was aware of the following national MRLs for folpet.

Country	MRL, mg/kg	Commodities
Canada	15	citrus, cucumbers, garlic, melons, pumpkins, squash
	25	apples, avocado, blackberry, blueberry, boysenberry, cherries, crabapples, cranberries, currants, dewberry, gooseberry, grapes, huckleberry, leeks , lettuce, loganberry, onion, raspberry, strawberries, tomatoes
	30	celery
EC	3	apple, grapes, lettuce, tomato
Hungary	5	fruits, table grapes, vegetables
Netherlands[1]	0.1	other fruits, other vegetables, other food commodities 0* (0.1)
	2	cherries, leafy vegetables group I, leek, legume vegetables, stone fruits, witloof chicory (sprouts)
	3	berries and other small fruits, blackberries, red, white and black currants, pome fruits, strawberry, tomato
USA	15	citrus, melon, squash
	25	apple, grapes, potato, strawberries, tomato
	50	lettuce

[1] residue definition: sum of captan and folpet.

APPRAISAL

Folpet was evaluated first in 1969, and several times since. The 1987 JMPR recommended that a detailed review of all aspects of the use of folpet be carried out at the 1989 Meeting or as soon as possible. At the 23rd (1991) Session of the CCPR it was decided (ALINORM 91/24A, para 95) to maintain CXLs for apple, cherries, cucumbers, grapes, bulb onions and strawberries, regarding them as temporary until 1992 when the results of current and planned supervised trials could be reviewed.

The 25th (1993) Session of the CCPR was informed that the manufacturer had provided information for all commodities with temporary MRLs except cherries and onions (ALINORM 93/24A, para 66).

The Meeting received information on registered uses of folpet and data from supervised trials on fruit and vegetables. MRLs for bulb onions and cherries will not be supported by new supervised field trials. Residue data from supervised trials on the following crops were reviewed:

mandarins (*Spain*), oranges (*Israel, Spain*), apples (*Chile, France, Israel, Portugal*), grapes (*Argentina, Chile, France, Israel, Italy, Spain*), strawberries (*Brazil, Hungary, Israel, Spain, Uruguay*).

melons (*France, Israel*), squash (*Greece*), lettuce (*Brazil, France, Israel*), potatoes (*Denmark, Israel, Netherlands, UK, Uruguay*), tomatoes (*France, Hungary, Israel*).

The Meeting was informed that the proposed GAP for folpet in Israel would probably become official in the near future. Only current official GAP is used in the evaluation of residue data.

There are no current registered uses for folpet on citrus, so the Meeting could not estimate a maximum residue level for citrus fruits. If the proposed use in Israel becomes registered, supervised trials data from Israel and Spain would suggest an MRL of 2 mg/kg.

The registered use of folpet on apples in Portugal requires a spray concentration of 0.13 kg ai/hl and a PHI of 21 days. Trials in Portugal and Israel conformed with this use pattern; the highest residues of folpet were

1.4 and 1.8 mg/kg. The Meeting was also aware of supervised trials on apples in France currently awaiting a final report. Because of the limited number of trials currently available within GAP the Meeting recommended withdrawal of the temporary MRL for apples.

Folpet is registered for use on grapes in Argentina, France, Italy and Spain. Supervised trials data were available from these countries. Trials were also available from Israel and were evaluated against GAP for grapes in Portugal and Spain. Residues arising from use according to GAP commonly fall in the 0.5-1 mg/kg range but residues of 1.3 and 2.0 mg/kg were recorded in a Spanish trial. The Meeting estimated a maximum residue level of 2 mg/kg for folpet in grapes.

Residue trial data on strawberries were provided from Brazil, Hungary, Israel, Spain and Uruguay, but there was no GAP for Hungary, Israel or Uruguay. Most of the residues in the Brazilian trials within GAP were in the 1-2 mg/kg range. The highest folpet residue in the Spanish trial within GAP was 1.1 mg/kg.

A folpet trial on strawberries in Israel (where registration is proposed) was evaluated against Portuguese GAP. Residues were consistently in the 2-5 mg/kg range, and were quite persistent. The highest residues were 4.7 and 4.8 mg/kg. The Hungarian trial was evaluated against Netherlands GAP; the highest folpet residue was 0.78 mg/kg. The Meeting estimated a maximum residue level of 5 mg/kg for folpet in strawberries.

No folpet was detected (<0.01 mg/kg) in melons from a French trial where an exaggerated spray concentration, approximately threefold, had been used. The laboratory had reported some problems with folpet recoveries when the sample was spiked before chopping. The fact that no residues were detected on samples taken the same day as the final application also throws doubt on the validity of the trial results.

No residues were detected (<0.02 mg/kg) in the pulp of melons in the Spanish trial. Data are required on a whole fruit-basis for MRL purposes. Trial data from the melon trials in Israel could not be evaluated because there is, as yet, no registered use in Israel.

The Meeting was unable to estimate a maximum residue level for folpet residues in winter squash because the data were too limited. The Meeting was informed that cucumber trial data would become available in the future from Turkey, Israel and Cyprus.

Folpet residues in lettuce treated according to Brazilian GAP ranged up to 1.4 mg/kg. Trial data on lettuce from Israel could not be evaluated because there is no registered use of folpet on lettuce in Israel, although it is proposed. The Meeting was also aware of supervised trials on lettuce in France currently awaiting a final report. Because of the limited number of trials available at present within GAP the Meeting recommended withdrawal of the temporary MRL for lettuce.

Supervised trials data for folpet on potatoes were available from Denmark, Israel, The Netherlands, the UK and Uruguay. The only country in this list which has registered uses for folpet on potatoes is Uruguay. Folpet residues in the trials from these countries were mostly not detectable (<0.01, <0.02 mg/kg). The pattern of residues expected for potatoes from the foliar use of a non-systemic pesticide is the occasional detection where a tuber has been directly exposed, but with no residues in most tubers. This pattern would not be much affected by the rate of application. The highest residue detected was 0.49 mg/kg in one plot in the Uruguay trial.

The Meeting noted that residues were generally undetectable in potatoes from application rates of 1.3 to 4.0 kg ai/ha in a number of different countries. The Meeting estimated a maximum residue level of 0.02* mg/kg for folpet in potatoes.

The maximum application rate for folpet on tomatoes in France is 1 kg ai/ha, but the rates used in the supervised trials were 1.5 and

3.0 kg ai/ha, so the data could not be used to estimate maximum residue levels. Folpet is not registered for use on tomatoes in Israel (although there is a proposed registration) so the data from trials in Israel could not be used. Folpet residues in tomatoes in Hungary treated according to GAP were not detectable (<0.02 mg/kg) 14 days after the final application. The Meeting considered the data were insufficient to estimate a maximum residue level for tomatoes.

Phthalimide residue data were also provided for most of the supervised trials. Phthalimide is the major primary metabolite of folpet. In many cases phthalimide residues were not detected in the trials, but in some cases they were of the same order as those of folpet, or even exceeded them. Phthalimide levels were generally not well related to the use of folpet and should not be included in the residue definition as an indicator of compliance with GAP.

The stability of folpet and phthalimide residues in stored analytical samples (lettuce, potato, tomato, melon), separately fortified with each compound at 1 mg/kg, was tested at -18°C. About 10-20% of the residues were lost during 6 months freezer storage.

In the analytical methods used for many of the trials, samples were chopped and extracted with ethyl acetate, then cleaned up on a Florisil column for folpet, or by solvent partition (hexane, phosphate buffer) for phthalimide. Gas-liquid chromatography on a megabore column with a ^{63}Ni electron-capture detector for folpet and with a thermionic nitrogen-specific detector for phthalimide was used for the final determination. No interference was caused by 25 common pesticides which might occur in crop samples. The limits of determination were 0.05 mg/kg for folpet and 0.2 mg/kg for phthalimide. Limits of detection were lower by factors of 2-2.5.

The Meeting received information on national MRLs for folpet from Canada, the EEC, Hungary, The Netherlands and the USA.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits.

Definition of the residue: folpet.

Commodity		Recommended MRL, mg/kg		PHI on which based, days
CCN	Name	New[1]	Previous	
FP 0226	Apple	W	10 T	
FS 0013	Cherries	W	15 T	
FC 0001	Citrus fruits	W	10 T	
VC 0424	Cucumber	W	2 T	
FB 0269	Grapes	2	25 T	21
VL 0482	Lettuce, Head	W	15 T	
VC 0046	Melons, except Watermelon	W	2 T	
VA 0385	Onion, Bulb	W	2 T	
FB 0275	Strawberry	5	20 T	7
VR 0589	Potato	0.02*		
VO 0448	Tomato	W	5 T	

[1] W: the previous recommendation is withdrawn.

FURTHER WORK OR INFORMATION

Desirable

1. Full details and results of the French trials on apples and lettuce now awaiting final reports, together with full details of the relevant French GAP.

REFERENCES

1. Barbina, M.T., Mondini, R., Pezzoni, A. and Valentino, A. 1991. Analysis of residues of folpet and fthalimide in samples of grapes. R6625, relates to IT-301-91. R6628, relates to IT-302-91. CRSA, Italy. Unpublished.

2. Benac, G. 1992. Residue trial report, grapes. Report FR 101/91. Institut Technique de la Vigne et du Vin, France. Unpublished.

3. Berger, J.L. 1991. Residue trial report, grapes. Report FR 102/91. Unpublished.

4. Byast, T.H. and Tolhurst, A.C. 1992. Determination of folpet and its metabolite phthalimide residues in potatoes. Study R 52593. Oxford Analytical, UK. Unpublished.

5. Cabello, F. 1991. Residue trial report. Trial SP-201-91. Agricultural Investigation Centre of the Community of Madrid, Spain. Unpublished.

6. Cooper, J.F. 1992. Residus de folpel et de phthalimide dans des echantillons de tomates. Relates to FR110-91. Universite de Montpellier, France. Unpublished.

7. Laurent, J.C. 1991. ITV Orange 84, essai folpel captane 91. Report FR 103/91. Institut Technique de la Vigne et du Vin, France. Unpublished.

8. Luttringer, M. 1992. Folpel residues in tomatoes and melons. Relates to FR106/91 (tomatoes), FR109-91 (cantaloup). INRA, France. Unpublished.

9. Ministry of Welfare, Health and Cultural Affairs. 1988a. Multi-residue method 1, electron captive compounds. Analytical Methods for Residues of Pesticides in Foodstuffs. The Netherlands.

10. Ministry of Welfare, Health and Cultural Affairs. 1988b. Multi-residue method 12, gel permeation clean-up. Analytical Methods for Residues of Pesticides in Foodstuffs. The Netherlands.

11. Munita, J.E. 1992. Determinación de residuo de folpet y captan en manzana Red King Oregon y uva Thompson Seedless. Report 7538/7779. Analab, Ltda, Chile. Unpublished.

12. Navarro, V. and Saes, K. 1991. Determination des residus de folpel et captane dans des echantillons de grapes. Relates to FR 101/91, FR 102/91, FR 103/91. Institut Technique de la Vigne et du Vin, France. Unpublished.

13. Navarro, V. and Saes, K. 1992. Determination des residus de phthalimide et tetrahydrophthalimide dans des echantillons de grapes. Report R604, relates to FR 101/91, FR 102/91, FR 103/91. Institut Technique de la Vigne et du Vin, France. Unpublished.

14. Perrone, D.H. 1992a. Determinación de residuos de captan, folpet, y metabolitos en muestras de papas. Sistemas Analiticos Laboratorios, SRL. Unpublished.

15. Perrone, D. 1992b. Determinación de residuos de captan, folpet y matabolitos en muestras de frutillas. Sistemas Analiticos Laboratorios,

SRL. Unpublished.

16. Schlesinger, H.M. 1991a. A method for the determination of folpan and phthalimide residues in non-oily crops. Project FP/15/91. Analyst Research Laboratories, Israel. Unpublished.

17. Schlesinger, H.M. 1991b. Determination of folpan and phthalimide residues in tomatoes (Israel). Project FP/16/91, relates to trial IS3. Analyst Research Laboratories, Israel. Unpublished.

18. Schlesinger, H.M. 1991c. Determination of folpan and phthalimide residues in lettuce (Israel). Project FP/17/91, relates to trial IS1. Analyst Research Laboratories, Israel. Unpublished.

19. Schlesinger, H.M. 1991d. Determination of folpan and phthalimide residues in potatoes (Israel). Project FP/18/91, relates to trial IS2. Analyst Research Laboratories, Israel. Unpublished.

20. Schlesinger, H.M. 1991e. Determination of folpan and phthalimide residues in melons (Israel). Project FP/19/91, relates to trial IS4. Analyst Research Laboratories, Israel. Unpublished.

21. Schlesinger, H.M. 1991f. Determination of folpan and phthalimide residues in grapes (Israel). Project FP/20/91, relates to trial IS6. Analyst Research Laboratories, Israel. Unpublished.

22. Schlesinger, H.M. 1991g. Determination of folpan and phthalimide residues in squash (Thessalonika, Greece). Project FP/21/91, relates to trial IS4. Analyst Research Laboratories, Israel. Unpublished.

23. Schlesinger, H.M. 1991h. Determination of folpan and phthalimide residues in potatoes (Denmark). Project FP/22/91, relates to trial 38910201. Analyst Research Laboratories, Israel. Unpublished.

24. Schlesinger, H.M. 1991i. Determination of folpan and phthalimide residues in potatoes (The Netherlands). Project FP/23/91, relates to trial LF g1-25/1. Analyst Research Laboratories, Israel. Unpublished.

25. Schlesinger, H.M. 1991j. Determination of folpan and phthalimide residues in apples (Israel). Project FP/24/91, relates to trial IS5. Analyst Research Laboratories, Israel. Unpublished.

26. Schlesinger, H.M. 1991k. Determination of folpan and phthalimide residues in apples (Portugal). Project FP/25/91, relates to trial PR-401-91. Analyst Research Laboratories, Israel. Unpublished.

27. Schlesinger, H.M. 1991l. Determination of folpan and phthalimide residues in tomatoes (Hungary). Project FP/26/91, relates to trial 571/1/91. Analyst Research Laboratories, Israel. Unpublished.

28. Schlesinger, H.M. 1991m. Determination of folpan and phthalimide residues in strawberries (Hungary). Project FP/27/91, relates to trial 571/2/91. Analyst Research Laboratories, Israel. Unpublished.

29. Schlesinger, H.M. 1992a. Determination of folpan and phthalimide residues in strawberries (Israel). Project FP/28/92, relates to trial IS8. Analyst Research Laboratories, Israel. Unpublished.

30. Schlesinger, H.M. 1992b. Determination of folpan and phthalimide residues in oranges (Israel). Project FP/29/92, relates to trial IS7. Analyst Research Laboratories, Israel. Unpublished.

Cross-index of study numbers, report numbers and references.

Reference numbers are indicated by #.

38910201	FP/22/91	#23	571/2/91	FP/27/91	#28
571/1/91	FP/26/91	#27	7538/7779	#11	

FP/15/91 #16
FP/16/91 IS3 #17
FP/17/91 IS1 #18
FP/18/91 IS2 #19
FP/19/91 IS4 #20
FP/20/91 IS6 #21
FP/21/91 IS4 #22
FP/22/91 38910201 #23
FP/23/91 LF g1-25/1 #24
FP/24/91 IS5 #25
FP/25/91 PR-401-91 #26
FP/26/91 571/1/91 #27
FP/27/91 571/2/91 #28
FP/28/92 IS8 #29
FP/29/92 IS7 #30
FR 101/91 #2
FR 101/91 #12
FR 101/91 R604 #13
FR 102/91 #3
FR 102/91 #12
FR 102/91 R604 #13FR 103/91 #12
FR 103/91 R604 #13
FR 103/91 #7
FR106/91 #8
FR110-91 #6
IS1 FP/17/91 #18
IS2 FP/18/91 #19
IS3 FP/16/91 #17
IS4 FP/21/91 #22
IS4 FP/19/91 #20
IS5 FP/24/91 #25
IS6 FP/20/91 #21
IS7 FP/29/92 #30
IS8 FP/28/92 #29
IT-301-91 R6625 #1
IT-302-91 R6628 #1
LF g1-25/1 FP/23/91 #24
PR-401-91 FP/25/91 #26
R 52593 #4
R604 FR 103/91 #13
R604 FR 102/91 #13
R604 FR 101/91 #13
R6625 IT-301-91 #1
R6628 IT-302-91 #1
SP-201-91 #5

HEPTACHLOR (43)

EXPLANATION

Heptachlor has been evaluated several times by the JMPR, first in 1963 and most recently in 1991. The 1987 Meeting noted that all the limits were Extraneous Residue Limits. The 22nd Session of the CCPR (1992) considered withdrawing the ERL for vegetables, but decided to wait for further data. Information on use patterns was supplied to the 1991 JMPR as a brief summary but no registered uses on vegetables were reported. Residue data were also provided from monitoring heptachlor and heptachlor epoxide. The 1991 Meeting recommended that the ERLs for heptachlor in carrots, tomatoes and other vegetables should be converted to temporary limits and required more information on the possible occurrence of residues in food in commerce or at consumption.

Monitoring data were received by the present Meeting from The Netherlands, Sweden and the USA.

USE PATTERN

No information was supplied on the registered or recommended uses of heptachlor on edible crops or animals.

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

Data from monitoring residues of heptachlor in plant crops and animal products were received from the governments of The Netherlands, Sweden and the USA.
All residues of heptachlor in the Tables are the sum of heptachlor and heptachlor epoxide, in conformity with the Codex definition.

In The Netherlands agricultural plant products are routinely analysed for residues of heptachlor and heptachlor epoxide. In the period 1987-1991 about 15,200 samples of fruits, vegetables and cereal grains were analysed. Analyses with the limit of determination of 0.01 mg/kg detected residues in 13 samples of which 3 were apples. In two samples (apple and wheat) residues were higher than the maximum residue limit in The Netherlands.

In the period 1990-1992, samples of fat from the following domestic animals were analysed for residues of heptachlor epoxide: 180 veal calves, 144 dairy cows, 143 broilers, 143 fattened bulls, 324 pigs, 72 sheep, 48 goats and 37 horses, together with 24 samples of egg powder. No residues were found in any of the samples, with a limit of determination of 0.02 mg/kg.

In Sweden domestic and imported vegetables were analysed for residues of heptachlor and heptachlor epoxide in 1990-1992. The limit of determination was 0.01-0.02 mg/kg. No residues were detected in 360 samples of carrots from 10 countries or in 1249 samples of tomatoes from 21 countries. Residues of heptachlor were present at the level of 0.03 mg/kg in one of 301 samples of imported melons from 19 countries, and at 0.04 and 0.05 mg/kg in two of 71 samples of imported squash from 9 countries.

Information was supplied from the USA on residues of heptachlor found in monitoring carried out in 1991 and 1992. Results were reported on citrus fruits, carrots,

pineapples, sugar beet and tomatoes, and on other fruit and vegetables as a group. Data were also available for residues in crude soya bean oil, cereal grains and animal products. Results for fruit, vegetables and cereals are shown in Table 1.

Table 1. Residues of heptachlor from monitoring fruit, vegetables and cereal grains in the USA, 1991-1992.

Commodity	No. of samples	No. of samples with residues[1]	90th percentile, mg/kg	Heptachlor, mg/kg, max. residue
Domestic				
Carrots	227	0		
Citrus fruit	408	0		
Pineapples	37	0		
Sugar beets	36	0		
Tomatoes	227	0		
Other fruits and vegetables	6132	13	0	0.05
Soya bean oil (crude)	8	0		
Whole cereal grain	573	5	0	0.03
Imported				
Carrots	126	0		
Citrus fruit	302	0		
Pineapples	143	0		
Tomatoes	717	0		
Other fruits and vegetables	6459	5		0.07

[1] Limit of determination 0.01 mg/kg.

In monitoring domestic animal products in the USA in 1991-92, 748 samples of milk and 621 samples of eggs were analysed for residues of heptachlor epoxide. No residues were found in any of the samples with the limit of determination at 0.01 mg/kg. In domestic animals heptachlor epoxide was found in one sample of veal at 0.15 mg/kg, in three samples of pigs at the level of 0.03-0.13 mg/kg and in two samples of geese at 0.04-0.06 mg/kg.

Imported meat products analysed in the USA in 1991-92 comprised 3934 samples of cattle fat, 2181 samples of pig fat, 738 samples of fat from sheep, lambs and goats, and 73 samples of fat from poultry. Residues of heptachlor epoxide were not detected in any of the samples.

APPRAISAL

Information on use patterns of heptachlor was supplied to the 1991 JMPR, but only in a summarized form and no registered or recommended uses on vegetables were available. Residue data from monitoring heptachlor and heptachlor epoxide in fruits and vegetables and animal products were also reported. The 1991 Meeting recommended that the existing Extraneous Residue Limits for heptachlor in carrots, tomatoes and other vegetables should be converted to temporary limits until more information was available on the possible occurrence of residues in food in

commerce or at consumption.

Monitoring data were received by the present Meeting from The Netherlands, Sweden and the USA.

Residues in fruit and vegetables occurred only to a very limited extent. In 15,300 samples of fruit, vegetables and cereal grains examined in The Netherlands residues were present in only 13 samples. In Sweden residues occurred in only 3 of about 9000 samples analysed, including many samples of carrots and tomatoes. In the USA residues of heptachlor were present in 18 of 14,800 samples of fruit and vegetables and 5 of 573 samples of cereal grains. The monitoring in the USA as well as in Sweden included the analysis of carrots and tomatoes. No residues were present in any of about 700 samples of carrots and 2200 samples of tomatoes analysed. The limit of determination in the three countries was 0.01-0.02 mg/kg.

In animals heptachlor is metabolised to heptachlor epoxide. This compound was not present in the animal products examined, with a few exceptions. No residues were detected in about 800 samples of domestic animal products examined in The Netherlands. In the USA no residues were found in milk, eggs or imported meat. Residues occurred at a level of 0.03-0.13 mg/kg in only three samples of domestic pigs and at 0.04 and 0.06 mg/kg in two samples of geese.

With this very low incidence of heptachlor in carrots, sugar beets, tomatoes and other vegetables the Meeting was of the opinion that there is no further need for ERLs for heptachlor in vegetables.

RECOMMENDATIONS

On the basis of the residue data received from monitoring in three countries the Meeting concluded that the TERLs and the ERL listed below should be withdrawn.

Definition of the residue: sum of heptachlor and heptachlor epoxide.

Commodity		Recommended MRL (mg/kg)	
CNN	Name	New	previous
VR 0577	Carrots	withdrawn	0.2 E T
VR 0596	Sugar beets	withdrawn	0.05 E
VO 0448	Tomato	withdrawn	0.02 E T
AO1 0002	Vegetables	withdrawn	0.05 E T

MANCOZEB (50)

EXPLANATION

Mancozeb was originally evaluated in 1967, and has been reviewed several times since. MRLs for dithiocarbamate fungicides were consolidated by the CCPR into a combined list in 1977 under the heading DITHIOCARBAMATES (105).

Mancozeb was scheduled for re-evaluation in 1993 in the CCPR periodic review programme.

The Meeting was provided with extensive information on use patterns, supervised residues trials, fate of residues, and miscellaneous studies by the Mancozeb Task Force and basic manufacturers. Information was also supplied by Australia, Canada, Finland, Germany and Spain.

IDENTITY

ISO common name: mancozeb
Chemical name:

IUPAC manganese ethylenebis(dithiocarbamate)(polymeric) complex with zinc salt

CA [[1,2-ethanediylbis[carbamodithioato]](2-)]manganese mixture with [[1,2-ethanediylbis[carbamodithioato]](2-)]zinc

CAS No: 8018-01-7
CIPAC No: CIPAC-34
Synonyms: Dithane M-45^{R}, Penncozeb®, Manzate^{R} 200

Structural formula:

$$[\text{-MnSC(:S)NHCH}_2\text{CH}_2\text{NHC(:S)S-}]_x\ \text{Zn}_y \quad \text{where } x/y = 11$$

A polymer coordination complex of zinc and manganese ethylenebis(dithiocarbamate) containing 20% manganese and 2.5% zinc.

Molecular weight per monomer unit: 271.2

Physical and chemical properties

Pure active ingredient

Vapour pressure:	Negligible
Solubility:	Water, 6 mg/l at 25°C (Schweitzer, 1987). Essentially insoluble in most organic solvents.
Hydrolysis:	Half-lives for aqueous hydrolysis of 10 mg/l suspended in distilled water:

pH 5: 36 hours
pH 7: 55 hours
pH 9: 16 hours

Technical material

Dithane M-45 is a polymeric, non-crystalline solid, a light yellow free-flowing powder with decomposition occurring at 150°C, a slight sulphurous odour, and an active ingredient content of about 80%.

Bulk density: 0.43 (loose), 0.48 (packed).
Stability: Stable in the absence of moisture, heat, flame, oxidising agents and acids. Decomposed by water under acidic conditions. Thermal decomposition may yield carbon disulphide and hydrogen sulphide.

USE PATTERN

Mancozeb is a protective fungicide effective against a wide range of foliar fungal diseases. It is registered for use in many countries on horticultural and agricultural food crops as well as on ornamentals and tobacco, and in forestry.

The registered uses of mancozeb are summarized in Tables 1-11.

Table 1. Citrus fruits.
Table 2. Pome fruits.
Table 3. Stone fruits.
Table 4. Berries and other small fruits.
Table 5. Tropical and subtropical fruits.
Table 6. Bulb vegetables and root and tuber vegetables.
Table 7. Brassica vegetables, leafy vegetables, and stalk and stem vegetables
Table 8. Fruiting vegetables.
Table 9. Legume vegetables.
Table 10. Cereals, tree-nuts and oilseed crops.
Table 11. Miscellaneous crops, including hops, coffee and tea.

Table 1. Registered uses of mancozeb on citrus fruits.

CROP	COUNTRY	APPLICATION			PHI, days
		Max no.	Rate per applicn. kg ai/ha	Spray concn. kg ai/hl	
Citrus fruits	Australia	2	6.0-13	0.16	
Citrus fruits	Brazil	4		0.12	14
Citrus fruits	Chile	2	3.0-7.7	0.14-0.19	21
Citrus fruits	Japan	2	1.9-4.0	0.094-0.19	60
Citrus fruits	Korea	2	5.2	0.15	21
Citrus fruits	Spain	2	13	0.32	15
Citrus fruits	Taiwan	3	3.2	0.16	40

Table 2. Registered uses of mancozeb on pome fruits.

CROP	COUNTRY	APPLICATION			PHI, days
		Max no.	Rate per applicn. kg ai/ha	Spray concn. kg ai/hl	
Apple	Australia	6	2.3-4.8	0.11-0.16	14
Apple	Brazil	10		0.16	7
Apple	Canada	6	4.5	0.53	45
Apple	Chile	6	2.9-5.8	0.14-0.19	21
Apple	France		1.6	0.16	
Apple	Japan	3	6.3-9.4	0.13-0.19	60

CROP	COUNTRY	APPLICATION			PHI, days
		Max no.	Rate per applicn. kg ai/ha	Spray concn. kg ai/hl	
Apple	Korea	2	7.5	0.15	21
Apple	Netherlands			0.15-0.16	
Apple	Portugal	4	1.6	0.16	15
Apple	Spain	3	2.4	0.16	15
Apple	UK			0.15-0.2	30
Apple	USA	4	5.4		not past bloom
Apple	USA	7	2.7		77
Crab-apple	USA	4	5.4		not past bloom
Crab-apple	USA	7	2.7		77
Medlar	Spain	3	2.4	0.16	15
Pear	Australia	8	2.3-4.8	0.11-0.16	14
Pear	Brazil	10		0.16	14
Pear	Canada	3	4.0-6.5	0.6	45
Pear	Chile	6	2.9-5.8	0.14-0.19	15
Pear	Japan	5	5.0-7.5	0.13-0.19	45
Pear	Korea	1	7.5	0.15	14
Pear	Netherlands			0.15-0.16	
Pear	Portugal	4	1.6	0.16	15
Pear	UK			0.15-0.2	30
Pear	USA	4	5.4		not past bloom
Pear	USA	7	2.7		77
Pome fruits	Austria	8	2.4-3.2	0.16	45
Pome fruits	Belgium	8		0.12-0.16	14
Pome fruits	Bulgaria	8	2.4-3.6	0.24	45
Pome fruits	Eire	10	3.6		28
Pome fruits	France	10		0.16	
Pome fruits	Germany	12	1.3-2.4	0.16	28
Pome fruits	Greece	6	4.0	0.2	7
Pome fruits	Hungary	8	2.1-3.2	0.16	45
Pome fruits	Italy	12	3.2	0.16	28
Pome fruits	Netherlands	4	1.2	0.12	56
Pome fruits	Romania	8	2.1-3.6	0.15-0.18	45
Pome fruits	Switzerland	4	1.2	0.12	21
Pome fruits	Turkey	6	4.0	0.2	21
Pome fruits	UK	10	3.6	0.18	28
Quince	USA	4	5.4		not past bloom
Quince	USA	7	2.7		77

Table 3. Registered uses of mancozeb on stone fruits.

CROP	COUNTRY	APPLICATION			PHI, days
		Max no.	Rate per applicn. kg ai/ha	Spray concn. kg ai/hl	
Apricot	Australia	2	2.3-4.8	0.11-0.16	14
Apricot	Chile	5	2.9-3.8	0.14-0.19	14
Cherry	Australia	2	2.3-4.8	0.11-0.16	14
Cherry	France	Po[1]		0.16	
Nectarine	Australia	2	2.3-4.8	0.11-0.16	14
Nectarine	Chile	5	2.9-3.8	0.14-0.19	14
Peach	Australia	2	2.3-4.8	0.11-0.16	14
Peach	Brazil	10		0.16	
Peach	Chile	5	2.9-3.8	0.14-0.19	14
Peach	Spain	2	2.4	0.16	15
Plum	Australia	2	2.3-4.8	0.11-0.16	14
Plum	Brazil	6		0.16	21
Plum	Chile	5	2.9-3.8	0.14-0.19	14
Plum	France	3		0.16	30
Stone fruits	Austria	4	2.1-3.2	0.16	45-60
Stone fruits	Bulgaria	4	2.1-3.2	0.24	45-60
Stone fruits	Germany	12	1.3-2.4	0.16	28
Stone fruits	Hungary	4	2.1-3.2	0.16	45-60
Stone fruits	Portugal	4	1.6	0.16	15
Stone fruits	Romania	4	2.3-3.2	0.16	45-60
Stone fruits	Spain	4	2.4-4.8	0.16-0.32	15
Stone fruits	Switzerland	2	1.6	0.16	21

[1] Po: Post-harvest.

Table 4. Registered uses of mancozeb on berries and other small fruits.

CROP	COUNTRY	APPLICATION			PHI, days [1]
		Max no.[2]	Rate per applicn. kg ai/ha	Spray concn. kg ai/hl	
Black currants	Eire	8	2.4		28
Black currants and gooseberries	Finland			0.16	
Black currants and gooseberries	UK	8	2.4	0.12	28
Cranberry	USA	3	5.4		30
Grapes	Australia	4	1.6-2.4	0.15	14
Grapes	Austria	5	0.80-1.9	0.24	40-60
Grapes	Brazil	8	2.8	0.28	7
Grapes	Bulgaria	5	2.4	0.24	40-60
Grapes	Canada	4	5.4	0.36	30
Grapes	Chile	3	2.2-3.8	0.14-0.19	66
Grapes	Columbia	15	1.7-4.8		30-45
Grapes	France	5+5	2.8 then 1.4		30
Grapes	Germany	8	0.96-4.8	0.16	56
Grapes	Greece	5	2.0	0.2	7
Grapes	Hungary	5	0.96-1.6	0.16	40-60
Grapes	Italy	6	1.6	0.16	28
Grapes	Japan	2fg	1.9-3.1	0.075-0.13	60
Grapes	Korea	?	3.7	0.12	30
Grapes	Philippines	12	0.6-1.5	0.2-0.38	
Grapes	Romania	5	0.96-1.6	0.16	40-60
Grapes	Spain	4	2.4		28
Grapes	Switzerland	4	1.4	0.2	1st postblossom
Grapes	Taiwan	3	2.0	0.13	14
Grapes	Turkey	5	1.6	0.16	21
Grapes	USA	6	2.2-3.6		66
Strawberry	Chile	4	0.9-2.7	0.14-0.19	2
Strawberry	France	5	1.6		
Strawberry	Japan	6g	1.9	0.13	76
Strawberry	Spain	2	1.6	0.16	3
Vine	Portugal	6	1.6-2.8	0.33-0.93	T 45, W 75

[1] T: table grapes; W: wine grapes.
[2] g: use in glasshouse; fg: use in field and glasshouse.

Table 5. Registered uses of mancozeb on tropical and subtropical fruits.

CROP	COUNTRY	APPLICATION			PHI, days
		Max no.	Rate per applicn. kg ai/ha	Spray concn. kg ai/hl	
Avocado	Brazil	10		0.16	21
Banana	Australia	24	1.7-3.6	0.16+oil	7
Banana	Brazil	1	2.0	0.2-4.0	21
Banana	Columbia	20	1.1-1.5		0
Banana	Philippines	5	1.6-2.2	5.3-7.2 a[1]	
Banana	Taiwan	8	1.6	5.3 a	14
Banana	USA	10	2.7		0
Fig	Brazil	6		0.16	21
Mango	Australia	10	1.6-2.4	0.16	14
Mango	Brazil	6		0.16	20
Mango	Malaysia	?		0.16-0.20	14
Mango	Philippines	12	4-7.5	0.20-0.38	
Mango	Taiwan	4	5.0	0.25	30
Olive	Greece	3	4.0	0.2	7
Olive	Spain	2	2.4	0.16	15
Papaya	Philippines	10	2.5-4.7	0.20-0.38	
Papaya	USA	14	2.2		0
Passion fruit	Australia	4	1.4	0.16	14
Persimmon	Japan	2	5.0-7.5	0.13-0.19	45

[1] Aerial application.

Table 6. Registered uses of mancozeb on bulb vegetables and root and tuber

vegetables.

CROP	COUNTRY	APPLICATION			PHI, days
		Max no.	Rate per applicn. kg ai/ha	Spray concn. kg ai/hl	
Beet	Brazil	6	1.6	0.16	7
Beet	Columbia	4	0.8-2.4		7
Beetroot	Australia	4	1.3-1.8	0.11-0.16	14
Carrot	Australia	4	1.3-1.8	0.11-0.16	7
Carrot	Brazil	6	1.6	0.16	7
Carrot	Canada	4	1.8	0.3	7
Carrot	Columbia	4	0.8-2.4		7
Carrot	Portugal	2	1.6	0.16	
Carrot	Switzerland	3	1.0	0.2	21
Carrot	UK		1.4-1.8		7
Celeriac	Hungary	3	0.84-1.6	0.16	20-30
Chinese yam	Japan	4	3.1-4.7	0.13-0.19	21
Garlic	Philippines	15	1.6-3.0	0.2-0.38	7
Garlic	Brazil	4	1.6	0.16	7
Garlic	Chile	6	1.6-2.0	0.14-0.19	7
Garlic	France	8	1.6		21, 30 ?
Garlic	Japan	5	2.5-3.8	0.13-0.15	7
Garlic	Portugal	2	1.6	0.16	35
Garlic	Spain	2	1.6	0.16	35
Ginseng	Canada	6	3.5	0.18	30
Leek	Belgium	10		0.28	28
Leek	Chile	6	1.6-2.0	0.14-0.19	7
Leek	France	10	1.6		60
Leek	Japan	3	1.9-2.8	0.13-0.19	21
Leek	Philippines	15	1.6-3.0	0.2-0.38	7
Lotus (East Indian)	Japan	3fg[1]	1.1	3.8	1
Onion	Australia	5	1.6-2.8	0.11-0.16	7
Onion	Belgium	8	2.4		28
Onion	Brazil	4	1.6	0.16	7
Onion	Canada	5	2.6	0.43	10
Onion	Chile	6	1.6-2.0	0.14-0.19	7
Onion	France	8	1.6		30
Onion	Greece	4	2.0	0.20	3
Onion	Hungary	3	0.84-1.6	0.16	30-45
Onion	Japan	5	1.3-2.8	0.13-0.19	7
Onion	Korea		1.9	0.12	7
Onion	Netherlands	6	2.4	0.4	28
Onion	Philippines	15	1.6-3.0	0.2-0.38	7
Onion	Portugal	2	1.6	0.16	35
Onion	Romania	3	0.9-1.0	0.16	30-45
Onion	Spain	2	1.6	0.16	35
Onion	Sweden	5	0.5-2.0		30
Onion	Switzerland	5	1.0	0.2	21
Onion	Turkey	3	1.6	0.16	28
Onion	UK		1.8-2.7		7
Onion	USA	10	2.7		7
Potato	Australia	6	1.3-1.8	0.11-0.16	
Potato	Austria	5	1.6-2.4	0.27-0.40	30-45
Potato	Bangladesh		2		14
Potato	Belgium	10	1.5-3.2		21
Potato	Brazil	10	2.4	0.24	7
Potato	Bulgaria	5	0.96-1.6	0.16	30-45
Potato	Canada	6	1.8	0.3	1
Potato	Chile	10	0.9-1.9	0.14-0.19	
Potato	Columbia	10	0.8-2.4		15-20
Potato	Denmark	5	0.5-4		14
Potato	Eire	10	1.8		7
Potato	Finland	2	0.5-2.4		21
Potato	France	10	1.6		21
Potato	Germany	1	st[2]		
Potato	Germany	5	1.1		7
Potato	Greece	4	2.0	0.20	3
Potato	Hungary	5	0.84-1.6	0.16	30-45
Potato	Indonesia	12	0.96-1.9		7
Potato	Italy	6	2.4	0.24	28
Potato	Japan	7	1.9-9.4	0.13-0.19	14
Potato	Korea		1.9	0.12	14
Potato	Netherlands	14	2.4-3.2	0.4	14
Potato	Norway	5	0.5-2		14
Potato	Philippines	20	1.1-4.4	0.58	

CROP	COUNTRY	APPLICATION			PHI, days
		Max no.	Rate per applicn. kg ai/ha	Spray concn. kg ai/hl	
Potato	Portugal	4	1.6	0.16	28
Potato	Romania	5	1.6-2.0	0.26-0.33	30-45
Potato	Spain	4	1.6	0.16	28
Potato	Sweden	5	0.5-2.0		30
Potato	Switzerland	8	2.4	0.4	21
Potato	Turkey	4	1.6	0.16	14
Potato	UK	10	1.4		7
Potato	USA	7	1.8		3
Shallot	Belgium	8	2.4		28
Shallot	France	8	2		30
Shallot	Indonesia	5	0.8-1.6		7
Shallot	Philippines	15	1.6-3.0	0.2-0.38	7
Sugar beet	Canada	5	1.8		21
Sugar beet	Chile	5	1.8-2.7	0.14-0.19	14
Sugar beet	France	3	3.2		30
Sugar beet	Japan	4	1.9-2.8	0.13-0.19	45
Sugar beet	Spain	2	1.6		28
Sugar beet	USA	7	1.8		14

[1] Use in field and glasshouse.
[2] Seed treatment

Table 7. Registered uses of mancozeb on brassica vegetables, leafy vegetables and stalk and stem vegetables.

CROP	COUNTRY	APPLICATION	PHI, days

		Max no.[1]	Rate per applicn. kg ai/ha	Spray concn. kg ai/hl	
Artichoke	Spain	2	1.6	0.16	15
Asparagus	Belgium	8	2.8		28
Asparagus	France	8	1.6	0.16	
Asparagus	Germany	4	0.96		early applic
Asparagus	Netherlands	4	2.4	0.4	
Asparagus	Spain	4	1.6	0.16	15
Asparagus	USA	4	1.8		180
Asparagus	USA (CA, AZ)	4	1.8		120
Broccoli	Australia	4	1.3-1.8	0.11-0.16	7
Broccoli	Brazil	8	1.6	0.16	7
Broccoli	Chile	5	0.6-1.9	0.14-0.19	15
Broccoli	Philippines	15	1.2-3.0	0.2-0.38	
Brussels sprouts	Australia	4	1.3-1.8	0.11-0.16	7
Brussels sprouts	Chile	5	0.6-1.9	0.14-0.19	15
Cabbage	Australia	4	1.3-1.8	0.11-0.16	7
Cabbage	Brazil	8	1.6	0.16	7
Cabbage	Chile	5	0.6-1.9	0.14-0.19	15
Cabbage	Columbia	4	0.8-2.4		7
Cabbage	Japan	3	1.9-3.8	0.13-0.19	45
Cabbage	Malaysia			0.16-0.20	21
Cabbage	Philippines	15	1.2-3.0	0.2-0.38	
Cauliflower	Australia	4	1.3-1.8	0.11-0.16	7
Cauliflower	Brazil	8	1.6	0.16	7
Cauliflower	Chile	5	0.6-1.9	0.14-0.19	15
Cauliflower	Malaysia			0.16-0.20	21
Cauliflower	Philippines	15	1.2-3.0	0.2-0.38	
Celery	Australia	5	1.3-1.8	0.11-0.16	7
Celery	Belgium	10		0.16	30-60
Celery	Canada	3	1.8-2.4	0.3	14
Celery	Chile	6	0.6-1.9	0.14-0.19	14
Celery	Columbia	4	0.8-2.4		7
Celery	France	10	1.6		30-60
Celery	Philippines	15	1.2-3.0	0.2-0.38	14
Celery	Portugal	2fg	1.6	0.53	
Celery	Switzerland	5	1.0	0.2	21
Celery	UK		1.8		14
Chard	Australia	4	1.3-1.8	0.11-0.16	14
Chinese cabbage	Indonesia	5	0.8-1.9		7
Chinese cabbage	Japan	3	1.9-2.8	0.13-0.19	30
Cole	Portugal	1fg	1.6	0.16	
Endive	France	5	1.6		root dip
Kale	Brazil	8	1.6	0.16	14
Lettuce	Australia	5	1.3-1.8	0.11-0.16	14
Lettuce	Chile	5	0.6-1.9	0.14-0.19	15
Lettuce	Columbia	4	0.8-2.4		7
Lettuce	France	10	1.4		
Lettuce	Malaysia			0.16-0.20	21
Lettuce	Portugal	4fg	1.6	0.16	21
Lettuce	Spain	4fg	1.6	0.16	15
Lettuce	Switzerland	5fg	0.8	0.16	21
Lettuce	UK	8	3.1		14
Lettuce	UK	2g	3.1		21
Rhubarb	Australia	4	1.3-1.8	0.11-0.16	14
Spinach	Australia	4	1.3-1.8	0.11-0.16	14
Spinach	Columbia	4	0.8-2.4		7
Spinach	Malaysia			0.16-0.20	21

[1] g: use in glasshouse; fg: use in field and glasshouse.

Table 8. Registered uses of mancozeb on fruiting vegetables.

CROP	COUNTRY	APPLICATION	PHI, days

		Max no.[1]	Rate per applicn. kg ai/ha	Spray concn. kg ai/hl	
Cantaloupe	Australia	4	1.3-1.8	0.11-0.16	7
Cantaloupe	Canada	1	2.6	0.43	14
Cantaloupe	Columbia	4	0.8-1.5		15
Cantaloupe	Philippines	12	0.6-2.6	0.2-0.38	
Cantaloupe	USA	8	2.7		5
Cucumber	Australia	4	1.3-1.8	0.11-0.16	7
Cucumber	Brazil	8	1.6	0.16	7
Cucumber	Canada	1	2.6	0.43	14
Cucumber	Chile	6	1.6-2.0	0.14-0.19	5
Cucumber	Columbia	4	0.8-1.5		15
Cucumber	Japan	3g	2.5-3.8	0.13-0.19	1
Cucumber	Korea		2.4	0.12	2
Cucumber	Philippines	12	0.6-2.6	0.2-0.38	
Cucumber	Spain	4	1.6-2.4	0.16	15
Cucumber	USA	8	2.7		5
Cucurbits	Belgium	10fg		0.2	3
Cucurbits	France	10	1.6		3
Cucurbits	Greece	3	2.0	0.2	3
Cucurbits	Malaysia			0.16-0.20	0
Cucurbits	Turkey	3fg	1.6	0.16	14
Eggplant	Brazil	6	2.4	0.24	7
Eggplant	Columbia	4	0.8-2.4		7
Eggplant	Greece	4	2.0	0.2	3
Eggplant	Philippines	10	1.6-3.4	0.2-0.38	7
Fruiting vegetables, edible peel	Austria	4	0.8-1.92	0.16-0.24	4-14
Fruiting vegetables, edible peel	Romania	4	0.9-1.6	0.16	21-35
Melon	Australia	4	1.3-1.8	0.11-0.16	7
Melon	Canada	1	2.6	0.43	14
Melon	Chile	6	1.6-2.0	0.14-0.19	5
Melon	Japan	5g	2.5-5.6	0.13-0.19	7
Melon	Philippines	12	0.6-2.6	0.2-0.38	
Melon	Portugal	4fg	1.6	0.16	3
Melon	Spain	4	1.6-2.4	0.16	15
Melon	Turkey	3	1.6	0.16	7
Melon	USA	8	2.7		5
Peppers	Brazil	6	2.4	0.24	7
Peppers	Columbia	4	0.8-2.4		7
Peppers	Greece	4	2.0	0.2	3
Peppers	Malaysia			0.16-0.20	14
Peppers, chilli	Philippines	10	1.6-3.4	0.2-0.38	7
Peppers	Portugal	4fg	1.6	0.16	3
Peppers	Spain	4	1.6-2.4	0.16	15
Pumpkin	Australia	4	1.3-1.8	0.11-0.16	7
Pumpkin	Brazil	10	1.6	0.16	14
Pumpkin	Canada	1	2.6	0.43	14
Roselle	Indonesia	6	1.2-2.2		
Squash	Australia	4	1.3-1.8	0.11-0.16	7
Squash	Canada	1	2.6	0.43	14
Squash	Chile	6	1.6-2.0	0.14-0.19	5
Squash	Japan	3	1.9-3.0	0.13-0.19	30
Squash	Philippines	12	0.6-2.6	0.2-0.38	
Summer squash	Australia	4	1.3-1.8	0.11-0.16	7
Summer squash	USA	8	2.7		5
Sweet corn	Philippines	20	1.2-3.0	0.2-0.38	7
Sweet corn	USA	15	1.3		7
Tomato	Australia	6	1.4-2.8	0.11-0.16	7
Tomato	Belgium	10		0.16	3
Tomato	Brazil	10	2.4	0.24	7
Tomato	Bulgaria	5	1.6-2.4	0.16	20-30
Tomato	Canada	2	fg 2.6	0.43	7
Tomato	Chile	10	1.4-2.2	0.14-0.19	5
Tomato	Columbia	12	0.8-2.4		0
Tomato	Eire	10	2.3-2.7		5
Tomato	France	10	1.6		15
Tomato	Germany	4	1.2-1.4		7
Tomato	Greece	6	2.0	0.2	3
Tomato	Hungary	4	0.84-1.6	0.16	3
Tomato	Indonesia	10	1.3-1.9		7
Tomato	Italy	6	2.4	0.24	28
Tomato	Japan	5g	2.5-3.8	0.13-0.19	1
Tomato	Malaysia			0.24	14
Tomato	Philippines	10	1-3.4	0.2-0.38	
Tomato	Portugal	4fg	1.6	0.16	3

CROP	COUNTRY	APPLICATION			PHI, days
		Max no.[1]	Rate per applicn. kg ai/ha	Spray concn. kg ai/hl	
Tomato	Spain	4	1.6-2.4	0.16	15
Tomato	Switzerland	6	1.0	0.2	21
Tomato	Taiwan	3	2.0	0.2	7
Tomato	Turkey	6fg	1.6	0.16	14
Tomato	UK		1.4-2.7		5
Tomato	USA	7	1.8-2.7		5
Watermelon	Canada	1	2.6	0.43	14
Watermelon	Chile	6	1.6-2.0	0.14-0.19	5
Watermelon	Columbia	4	0.8-1.5		15
Watermelon	Japan	5	2.5-3.8	0.13-0.19	7
Watermelon	Korea		2.4	0.16	5
Watermelon	Philippines	12	0.6-2.6	0.2-0.38	
Watermelon	Portugal	4fg	1.6	0.16	3
Watermelon	Turkey	3	1.6	0.16	7
Watermelon	USA	8	2.7		5

[1] g: use in glasshouse; fg: use in field and glasshouse.

Table 9. Registered uses of mancozeb on legume vegetables.

CROP	COUNTRY	APPLICATION			PHI, days
		Max no.	Rate per applicn. kg ai/ha	Spray concn. kg ai/hl	
Azuki bean	Japan	3	1.3-1.9	0.13-0.19	14
Beans	Australia	4	1.3-1.8	0.11-0.16	7
Beans	Belgium	2		0.16	28
Beans	Brazil	6	1.6	0.16	14
Beans	Chile	4	1.4-2.2	0.14-0.19	14
Beans	France	2	1.6		21
Beans	Greece	4	2.0	0.2	3
Beans	Malaysia			0.16-0.20	14
Beans	Philippines	12	1-3.4	0.2-0.38	
Beans	Portugal	2	1.6	0.16	
Beans	Spain	2	1.6-2.4	0.16	15
Broad bean	Australia	2	1.2-2.0	0.11-0.16	7
Chickpea	Chile	4	1.4-2.2	0.14-0.19	14
French bean	Brazil	6	1.6	0.16	7
Green bean	Columbia	5	0.8-2.4		15-20
Green pea	Chile	4	1.4-2.2	0.14-0.19	14
Kidney bean	Japan	4	1.3-1.9	0.13-0.19	30
Lentil	Chile	4	1.4-2.2	0.14-0.19	14
Mung bean	Philippines	12	1-3.4	0.2-0.38	
Peas	Brazil	6	1.6	0.16	7
Peas	Columbia	5	0.8-2.4		15-20
Peas	France	2	1.6		
Peas	Malaysia			0.16-0.20	14
Peas	Philippines	12	1-3.4	0.2-0.38	
Peas	Portugal	2	1.6	0.16	
Peas	Spain	2	1.6-2.4	0.16	15
Soya bean	Australia	4	1.8		7
Soya bean	Hungary	3	0.84-1.6	0.15	30-45
Soya bean	Philippines	6	1-3.4	0.2-0.38	
Soya bean	Taiwan	4	2.4	0.2	

Table 10. Registered uses of mancozeb on cereals, tree-nuts and oilseed crops.

CROP	COUNTRY	APPLICATION			PHI, days
		Max no.	Rate per applicn. kg ai/ha	Spray concn. kg ai/hl	
Almond	Australia	2	2.3-4.8	0.11-0.16	14
Barley	Brazil	3	2.0		21
Barley	Canada	st			
Barley	Chile	3	1.6-2.0		26
Barley	Columbia	2	2.0		14-20
Barley	Eire	3	2.0		26
Barley	UK	3	1.6		gs

CROP	COUNTRY	APPLICATION			PHI, days
		Max no.	Rate per applicn. kg ai/ha	Spray concn. kg ai/hl	
Barley	USA	3	1.8		26
Cereals	Chile	st[1]			
Cereals	Spain	2	3.2		28
Cereals	UK		1.6-1.8		26-28
Coconut	Indonesia	6	0.24-1.44		7
Cotton	Philippines	15	1.2-3.0	0.2-0.38	
Cotton	USA	4	1.8		45
Flax	Canada	st			
Maize	Canada	st			
Maize	USA	10	1.3		40
Oats	Canada	st			
Oats	Chile	3	1.6-2.0		26
Oats	Eire	3	2.0		26
Oats	USA	3	1.8		26
Peanut	Australia	4	1.4-1.8		14
Peanut	Columbia	3	1.5		15-20
Peanut	Indonesia	4	0.8-1.6		7
Peanut	Korea		2.4		14
Peanut	Malaysia			0.16-0.20	14
Peanut	Philippines	12	1-3.4	0.2-0.38	14
Peanut	Taiwan	4	2.4	0.20	
Peanut	Turkey	2	1.6	0.16	14
Peanut	USA	8	1.8		14
Rice	Brazil	3	3.6		25
Rice	Bulgaria	3	0.96	0.16	40-50
Rice	Columbia	2	2.0-4.0		14-20
Rice	Philippines	15	0.32-0.9	0.2-0.38	
Rice	Taiwan	4	2.0	0.17	
Rye	Chile	3	1.6-2.0		26
Rye	Eire	3	2.0		26
Rye	USA	3	1.8		26
Sesame	Korea		2.4		7
Sorghum	Columbia	2	1.3-2.4		15-20
Wheat	Belgium	3	1.6		28
Wheat	Brazil	3	2.0		32
Wheat	Canada	2	1.8		40
Wheat	Canada	st			
Wheat	Chile	3	1.6-2.0		26
Wheat	Columbia	2	2.0		14-20
Wheat	Eire	3	2.0		26
Wheat	France	3	3.2		
Wheat	Netherlands	2	1.5	0.25	28
Wheat	Portugal	2	3.2		35
Wheat	Romania	1	1.6-1.8	0.36-0.46	35
Wheat	UK	3	1.6		gs[2]
Wheat	USA	3	1.8		26
Winter oilseed rape	UK	2	1.4		gs

[1] st: seed treatment.
[2] gs: growth stage restriction.

Table 11. Registered uses of mancozeb on miscellaneous crops including hops, coffee and tea.

CROP	COUNTRY	APPLICATION	PHI, days

		Max no.	Rate per applicn. kg ai/ha	Spray concn. kg ai/hl	
Cacao	Indonesia	6	0.8-0.96		7
Cacao	Brazil	4	3.0	0.3	14
Coffee	Brazil	6	4.0	1.0	21
Coffee	Columbia	3	0.8-1.2		0
Coffee	Indonesia	6	0.24-1.44		7
Fennel	USA	8	1.8		14
Hops	Belgium	10		0.16	42
Hops	Germany	12	1.8-6.4	0.16	35
Hops	Hungary	3	2.1-3.2	0.16	30-45
Hops	Turkey	2	1.2	0.12	42
Tea	Indonesia	5	0.34-0.67		7

RESIDUES RESULTING FROM SUPERVISED TRIALS

Residue data from supervised trials on horticultural and agricultural crops are summarized in Tables 12 to 50.

The information supplied was sometimes only in summary form, but most trials were fully or adequately described. Some residues were adjusted for analytical recoveries and some were not; in summary sheets very often no statement was made either way. Analytical recoveries were mostly high (>80%) for both dithiocarbamates and ETU, so adjustment of results should not influence interpretations. US results were adjusted; Australian were not. Attention is drawn to cases where analytical recoveries were less than 70%.

In the French trials of 1990, recoveries from 10 crops and wine (23 tests) ranged from 46 to 114%, with a mean recovery of 86%, at concentrations of 0.8-2.7 mg/kg (Wasser, 1993n). In the French trials of 1991, recoveries from 8 crops and wine (13 tests) ranged from 47 to 88%, with a mean recovery of 68%, at concentrations of 0.14-1.5 mg/kg (Mellet, 1993a).

Dithiocarbamate residues are expressed as mg CS_2/kg throughout the Tables and text. EBDC is used as an abbreviation for ethylenebis(dithiocarbamate)s in the Tables.

Where residues were not detected, data are recorded in the Tables as less than the limit of determination (LOD), e.g. <0.1 mg/kg. Residues have generally been rounded to 2 significant figures or, near the LOD, to 1 significant figure. When residues were detected in control samples they are recorded in the Tables. In the majority of cases no residues were detected in control samples; these are not recorded.

Plot sizes in the Australian trials were usually 8-20 m of 1-2 rows (4 replicates) for row crops and 1 tree (4 replicates) for tree crops. Mancozeb was applied with a hand-held high-volume sprayer or a self-propelled small-plot sprayer. Analytical recoveries exceeded 70% except in the following trials: peaches (AUE-91-027, Table 20) dithiocarbamates 62%; bananas (2495/89, Table 26) ETU 55-111%, dithiocarbamates 58%; watermelon (AUK-92-005, Table 32) dithiocarbamates 60%; beans (3137/88/5, Table 37) ETU 53-57%, dithiocarbamates in straw 67%.

Mancozeb was applied by a tractor-mounted sprayer in the Canadian trials on onions and lettuce. Plot size was the equivalent of 50-120 m of row.

Plot sizes in the Netherlands trials (PH references) were apples 5 trees, barley 25 m^2, beans 20 m^2, onions 3.6-20 m^2, potatoes 20-25 m^2, wheat 25 m^2-1 ha. Mancozeb was applied to crops in these trials with a propane-pressure knapsack (beans, onions, potato, tomatoes, wheat), a knapsack mist blower (apples) and a motorised compressed air sprayer (potato). Recoveries of dithiocarbamates were all satisfactory. ETU recoveries were sometimes low (<70%). Low recoveries in individual tests were reported in the analysis of apples, barley straw, onions, potatoes, tomatoes and wheat.

Dithiocarbamate residues or apparent residues were detected in untreated control samples in US trials on citrus (Table 13), apples (Table 19), cranberries (Table 22), bananas and papayas (Table 27), onions (Table 29), cucurbits (Table 33), tomatoes (Table 35), sugar beet (Table 40), celery (Table 42), cereal grains (Table 44), cereal fodder (Table 49) and sugar beet tops (Table 50). Control samples also occasionally showed low residues of ETU: citrus (Table 13), cranberries (Table 22) and onions (Table 29).

In the extensive series of French trials dithiocarbamate or apparent dithiocarbamate residues were detected in untreated control samples. Instances are recorded in the Tables: pome fruits (Table 15), plums (Table 20), grapes (Table 23), bulb vegetables (Table 28), tomatoes (Table 34), carrots and potatoes (Table 38), asparagus (Table 41), cereal grains (Table 43) and cereal fodder (Table 48).

Contamination could have occurred in some cases from a high-level sample during handling or shipment (Wasser, 1993n). A coextractive from

carrots, tomatoes and asparagus may have contributed to a false colour in the Cullen's reagent in the analysis leading to a dithiocarbamate reading for a control sample (Wasser, 1993d and related references). Mellet (1993d) reported that the extraction-distillation step of potato analysis produces a yellow colour in the sodium hydroxide trap that fades after a few minutes. An excess of that contamination might account for apparent residues in some of the untreated potato samples. Similar explanations were provided for cereal grain and straw, and garlic.

ETU was detected (about 0.01 mg/kg) in samples of orange concentrate made from untreated fruit in Brazilian orange trials (81-0191, Table 12).

There was some detection of dithiocarbamates in control apples from the Belgian trials (Table 14) at 0.01-0.02 mg/kg, which is near the limit of determination.

In the UK apple trials (R71.16, Table 18) treated areas were 2-3 ha in the first four studies, where samples were taken for residue decline measurement. Samples were also taken from 11 commercial orchards with recorded spray programmes (R71.16, Table 18).

Mancozeb was applied by air-blast equipment in the US apple trials (ETU 91-02, Table 19). The plot size was 8 trees.

Cranberries in the US trials (Table 22) were grown on plots of 10-40 m^2, and were hand-sprayed.

In five separate experiments in France in 1976 (Haines, 1978), wine was produced from grapes treated with mancozeb (6-9 times, final application 0.8-1.2 kg ai/ha) and harvested 50-70 days after the final application. Neither dithiocarbamates (<0.05 mg/kg as CS_2) nor ETU (<0.02 mg/kg) were detected in the wine.

In a similar set of experiments with 3 wines in Germany (Haines, 1979) dithiocarbamate residues were not detected (<0.05 mg/kg as CS_2) but ETU was detected in one wine at 0.21 mg/kg and identified by GC-MS. In this case the final mancozeb application had been at 2.2 kg ai/ha 72 days before harvest.

Samples of wine, 1989 vintage ready for commercialisation, were taken from two different French vineyards with accurately recorded pesticide use (R78.85, R78.82, Table 23). Dithiocarbamate residues were not detected (Wasser, 1993m). The results agreed with a previous similar investigation in 1988 reported by Wasser (1993l) on three French vineyards (R78.78, R78.89, R79.1, Table 23).

UK residue data on black currants are summarized in Table 25. The first three trials were supervised trials on 5 m row plots (4 replicates) with application by a motorised knapsack sprayer. The remainder were grower trials on areas of approximately 1 ha. Analytical recoveries of dithiocarbamates were low (61%).

Papayas in the US (Florida) trials were on 1.2 ha plots and were ground-sprayed (Table 27). The trials in Hawaii were on a smaller scale, 300 m^2 plot, and the papayas were hand-sprayed.

The plot size was 2.7-4.4 ha for three of the four onion trials in the USA where mancozeb was applied by aerial and ground equipment (Table 29). The plot sizes in the remaining trials were 5-30 m^2.

Cucurbit vegetables in the US trials (Table 33) were mostly sprayed with ground equipment (some hand-spraying). The plot size was in the 15-45 m^2 range.

Mellet (1993j) reported that an apparent dithiocarbamate residue of 0.23 mg/kg in untreated tomatoes from a Spanish trial (R80.30, Table 34) could be due to interference in the analytical method by a co-extractive forming a yellow colour with Cullen's reagent, or contamination may have occurred during handling or shipping samples.

Plants in the first five tomato trials listed in Table 35 were hand-sprayed on 10 m^2 plots.

Carrot trials in France (R77.33/34, Table 38) in 1990 were carried out on plots of 3.2 $\times$ 10 m.

A series of trials on potatoes in 1975-76 in 9 States of the USA (Table 40) showed that dithiocarbamate residues were rarely detectable in potatoes even when mancozeb was used at exaggerated application rates. Analytical recoveries for ETU were sometimes down to 60%, but ETU was not detectable in these trials. Plot sizes ranged from 15 m^2 for ground-sprayed carrots to 2.2 ha for aerial spraying. Four of the sugar beet trials were on 3-4 ha plots, while two trials were on 40 ha fields.

ETU residues were not detected (<0.02 mg/kg) in cooked and processed products (baked potato skin, baked potato, boiled potato, chips, flakes and French fries) produced from potatoes in trials 75-537-02, 75-538-02 and 75-514-02 (Table 40). ETU residues were not detected (<0.02 mg/kg) in cooked and processed products (chips, flakes and French fries) produced from potatoes in trials 75-459-02, 75-494-02 and 75-443-02 (Table 40).

Mancozeb was aerially and ground-applied to celery in US trials (Table 42), with three trials on 4 ha plots and one (85-0165) on 80 ha.

Detection of apparent dithiocarbamate residues in control wheat straw (Table 48) from the Canadian trials may have been interference in the analytical method by hydrogen sulphide (Frank *et al.*, 1986).

Wheat trials were conducted in 1975 and 1981 in 5 different States of the USA with 8 different wheat varieties (Tables 44, 49). Dithiocarbamate residues were detected in some control grain and straw samples, probably as a result of drift to the control plots. Dithiocarbamate residues were detected in bran, flour and bread prepared from control wheat (81-0167, 81-0168, 81-0428, 81-0429, 81-0430, 81-0426, 81-0427, 81-0212, 81-0214). Details of the milling are recorded in Table 75.

The US barley trials (Tables 44, 49) in Idaho and Washington State were on 5 ha plots. The other two trials were on 20 m^2 plots.

Analytical recoveries were lower than usual for mancozeb in peanuts 69-87% and peanut hay 68-75%, and for ETU in peanuts 53-110% and peanut hay 56-58% in a US trial (74-171-02, Table 46, 50). Mancozeb recoveries were 58-90% from peanuts in trial 74-180-02 (Table 46).

Table 12. Mancozeb residues (as CS_2) in citrus fruits from supervised trials in Australia, Brazil, Japan and Spain. Underlined residues are from treatments according to GAP.

CROP Country, year (Variety)	Application				Day	Residues, mg/kg[1]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
CITRUS "SUMMER" ORANGES, SOUR								
Japan, 1990 (Kawano-amanatsu)	WP	5.0	0.13	2	60 75 90	pu 0.010, pe 1.5 pu 0.011, pe 1.3 pu 0.006, pe 1.0	pu <0.01, pe 0.02 pu <0.01, pe 0.01 pu <0.01, pe <0.01	Hei.-3-3-5
Japan, 1990 (Amanatsu)	WP	5.0	0.13	2	63 75 91	pu <0.004, pe 0.78 pu 0.005, pe 0.58 pu <0.004, pe 0.32	pu <0.01, pe 0.01 pu <0.01, pe 0.01 pu <0.01, pe <0.01	Hei.-3-3-5
LEMONS								
Spain, 1992 (Verna)	WP		0.3	1	0 7 14 21	2.5 0.19 0.10 0.01		MAPA 23.06.93

CROP Country, year (Variety)	Application				Day	Residues, mg/kg[1]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
MANDARINS								
Japan, 1977 (Okitsuwase)	WP	3.8-4.7	0.19	2	30 44 60	pu 0.04, pe 1.6 pu 0.04, pe 0.93 pu 0.06, pe 1.8	pu <0.01, pe 0.07 pu <0.01, pe 0.05 pu <0.01, pe 0.06	53P-7-68
Japan, 1977 (Okitsuwase)	WP	3.8-4.7	0.19	4	30 44 60	pu 0.06, pe 1.6 pu 0.07, pe 1.4 pu 0.07, pe 2.1	pu 0.01, pe 0.08 pu 0.01, pe 0.08 pu 0.01, pe 0.08	53P-7-68
Japan, 1977 (Miyakawa)	WP	9.4	0.19	2	29 46 60	pu 0.12, pe 3.5 pu 0.04, pe 1.5 pu 0.07, pe 1.8	pu <0.01, pe 0.10 pu <0.01, pe 0.06 pu 0.01, pe 0.08	53P-7-68
Japan, 1977 (Miyakawa)	WP	9.4	0.19	4	29 46 60	pu 0.07, pe 3.1 pu 0.12, pe 3.1 pu 0.12, pe 3.7	pu 0.01, pe 0.13 pu 0.01, pe 0.11 pu 0.01, pe 0.12	53P-7-68
Spain, 1989 (Clementine)	WP	17	0.32	1	0 14 22	2.2 1.1 1.2		MAPA 7/5/91
Spain, 1989 (Satsuma)	WP	17	0.32	1	0 14 22	2.7 1.2 1.0		MAPA 7/5/91
Spain, 1989 (Clementine)	SC	15	0.25	1	0 7 14 21 28	1.7 1.8 1.4 1.1 0.80		7404/VI/89
Spain, 1989 (Satsuma)	WP	9.6	0.32	1	0 7 14 22	2.5, w 0.30 2.1, w 0.14 1.7 0.76, w 0.36		R77.11
Spain, 1990 (Clementine)	WP	4.3	0.16	1	0 6 14	2.3, w 0.12 3.9, w 0.34 2.0		R80.5
Spain, 1990 (Clementine)	WP	8.6	0.32	1	0 6 14	2.2, w 0.35 6.8, w 0.05 6.6, w 0.45		R80.5
Spain, 1990 (Satsuma)	WP	5.2	0.16	1	0 6 14	4.4, w 0.34 5.3, w 0.23 2.1		R80.7
Spain, 1990 (Satsuma)	WP	10.4	0.32	1	0 6 14	6.5, w 1.9 9.2, w 1.4 4.7, w 0.15		R80.7
ORANGES								
Australia, 1992 (Valencia)	WG		0.15	2	0 7 14 21 28	0.3 0.4 0.2 0.4 0.5		AUE-92-001
	WG		0.30	2	0 7 14 21 28	1.8 1.6 1.0 1.7 1.6		
Brazil, 1989 (Natal)	WP	1.2		1	0 7 14 21 56	0.31, j <0.03 0.36, j <0.03 0.25, j <0.03 0.76, j <0.03 0.19, j <0.03	<0.01, j <0.01 0.01, j <0.01 <0.01, j <0.01 <0.01, j <0.01 <0.01, j <0.01	89-0191
Brazil, 1989 (Natal)	WP	2.4		1	0 7 14 21 56	0.54, j 0.05 1.7, j 0.04 0.53, j <0.03 0.59, j <0.03 0.23, j <0.03	0.01, j <0.01 0.04, j 0.02 <0.01, j <0.01 <0.01, j <0.01 <0.01, j <0.01	89-0191
Spain, 1989 (Havelina)	SC	15	0.25	1	0 7 14 21 28	0.52 0.38 0.24 0.26 0.19		7404/VI/89
Spain, 1989 (Havelina)	SC	15	0.25	1	0 7 14 21 28	1.4 0.80 0.68 0.67 0.47		7404/VI/89
Spain, 1989 (Newhall)	WP	9.6	0.32	1	0 7 14	2.2, w 0.06 2.3, w 0.09 0.93, w 0.10		R77.12

CROP Country, year (Variety)	Application				Day	Residues, mg/kg[1]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
					22	0.66, w 0.09		
Spain, 1990 (Navel)	WP	0.88 0.88 0.88	0.044 0.044 0.044	1 1 1	24 16 10	0.12 0.90 1.3		R80.4
Spain, 1990 (Navel)	WP	17	0.32	1	0 14 22	1.2 0.85 0.49		MAPA 7/5/91
Spain, 1990 (Valencia)	WP	17	0.32	1	0 14 22	0.78 0.64 0.53		MAPA 7/5/91
Spain, 1991 (Valencia)	WP	16	0.32	1	0 7 14 21 28 56	1.4 0.96 0.80 0.84 0.66 0.69		MAPA 7/5/91
Spain, 1991 (Valencia)	WP	13	0.32	1	0 14	1.7 1.3		MAPA 7/5/91

[1] pu: pulp; pe: peel; w: washed fruit; j: juice.

Table 13. Mancozeb residues (as CS_2) in citrus fruits from supervised trials in the USA.

CROP State, year (Variety)	Application				Day	Residues, mg/kg [1]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
GRAPEFRUIT								
TX, 1986 (Ruby Red)	WP	9.0	0.78	4	0 7 14 28	7.3 6.2 4.6 1.4	0.31 0.42 0.20 0.12	34A-88-13
	WP	18	1.6	4	0 7 14 28	12 10 6.2 2.2	0.40 0.15 0.30 0.21	
CA, 1986 (Ruby White)	WP	11	0.24	4	0 7 15 27	3.4 2.3 1.3 0.98	0.03 0.04 0.05 0.04	34A-88-13
	WP	22	0.48	4	0 7 15 27	7.8 6.7 5.1 3.3	0.07 0.07 0.06 0.11	
CA, 1986 (Ruby Red)	WP	11	0.24	4	0 7 15 27	7.3 6.2 5.0 3.2	0.04 0.05 0.06 0.06	34A-88-13
	WP	22	0.48	4	0 7 15 27	16 14 11 5.6	0.20 0.13 0.20 0.16	
LEMONS								
CA, 1986 (Eureka)	WP	5.6	0.12	4	0 0 0 7 14 28	7.9 pe 17 pu 0.46 5.6 3.3 2.3	0.24 pe 0.25 pu 0.054 0.13 0.16 0.12	86-0148
	WP	11.2	0.24	4	0 7 14 28	20 17 11 6.7 c pe 0.14	0.44 0.39 0.42 0.26 c 0.02	
CA, 1986 (Eureka)	WP	5.6	0.12	4	0 0 0 7 14 28	10 pe 26 pu 1.2 6.9 5.9 3.5	0.27 pe 0.34 pu 0.041 0.23 0.25 0.19	86-0149
	WP	11.2	0.24	4	0 7 14	20 12 9.2	0.64 0.28 0.33	

CROP State, year (Variety)	Application				Day	Residues, mg/kg [1]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
					28	6.9 c pe 0.07	0.27	
FL, 1987 (Bearss)	WP	9.0	0.10	4	0 0 0 6 12 20 27	3.4 pe 6.2 pu 0.34 2.7 1.5 1.2 1.4	0.032 pe 0.065 pu 0.01 0.079 0.044 0.061 0.062	87-0017
	WP	18	0.20	4	0 6 12 20 27	13 6.6 3.5 2.6 1.6 c pe 0.15	0.19 0.37 0.25 0.13 0.12 c pe 0.01	
FL, 1987 (Meyer)	WP	18	0.57	5	0 0 0 6 11 19	27 pe 120 pu 0.67 21 18 9.3	0.39 pe 1.4 pu 0.029 0.50 0.38 0.17 c 0.01	87-0024
FL, 1987 (Bearss)	WP	9.0	0.19	5	0 0 0 6 12 20 27	5.7 pe 7.7 pu 0.26 3.9 1.8 1.3 0.82	0.052 pe 0.14 pu 0.02 0.052 0.044 0.063 0.041	87-0018
	WP	18	0.38	5	0 6 12 20 27	14 7.5 3.5 3.0 2.3 c pe 0.30	0.23 0.12 0.091 0.10 0.086	
LIMES								
FL, 1987 (Persian)	WP	9.0	0.10	4	0 0 0 6 12 20 27	4.8 pe 28 pu 0.15 2.6 2.0 1.0 0.49	0.21 pe 0.26 pu 0.01 0.051 0.12 0.086 0.080	87-0020
	WP	18	0.20	4	0 6 12 20 27	12 8.2 5.6 2.2 1.6 c pe 0.05	0.43 0.21 0.24 0.19 0.21 c 0.02	
FL, 1987 (Persian)	WP	9.0	0.19	4	0 0 0 6 12 20 27	4.0 pe 17 pu 0.67 3.3 2.3 1.5 1.1	0.15 pe 0.24 pu 0.01 0.062 0.052 0.058 0.076	87-0019
	WP	18	0.38	4	0 6 12 20 27	10.3 8.3 7.0 4.9 2.4 c 0.27 c pe 0.69 c pu 0.07	0.13 0.30 0.15 0.15 0.22 c 0.025	
ORANGES								
FL, 1986 (Valencia)	WP	9.0	0.1	4	0 7 15 28	3.1 1.1 0.88 0.38	0.038 0.01 0.01 <0.01	86-0134
	WP	18	0.2	4	0 7 15 28	5.4 2.4 1.2 0.93 c 0.04	0.074 0.043 0.026 0.034	
USA (TX), 1986 (Valencia)	WP	9.0	0.38	4	0 7 14 28	6.2 3.4 2.9 1.2	0.056 0.16 0.19 0.21	86-0495
		18	0.77	4	0 7 14 28	10 6.2 5.6 2.9	0.25 0.28 0.32 0.22	
CA, 1986	W?	11	0.24	4	0 8	7.8 4.2	0.050 0.065	86-0599

CROP State, year (Variety)	Application				Day	Residues, mg/kg [1]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
(Navel)					16	3.4	0.073	
					26	1.6	0.02	
		22	0.48	4	0	14	0.18	
					8	7.0	0.15	
					16	6.2	0.18	
					26	3.2	0.057	
FL, 1987 (Valencia)	WP	9.0	0.29	4	0	15	0.30	87-0025
					0	pe 35	pe 1.1	
					0	pu 0.30	pu 0.025	
					6	13	0.24	
					11	11	0.25	
					19	7.7	0.17	
					26	5.3	0.11	
	WP	18	0.58	4	0	29	0.63	
					6	23	0.56	
					11	18	0.34	
					19	15	0.24	
					26	10	0.19	
						c pe 0.16		
FL, 1987 (Valencia)	WP	9.0	0.10	4	0	3.8	0.13	87-0040
					0	pe 20	pe 0.31	
					0	pu 0.29	pu 0.018	
					6	1.7	0.057	
	WP	18	0.20	4	0	9.3	0.14	
					6	2.7	0.049	
							c 0.01	

[1] pe: peel; pu: pulp; c: control sample.

Table 14. Mancozeb residues (as CS_2) in pome fruits from supervised trials in Australia, Austria, Belgium, Brazil, Hungary, Japan and The Netherlands. Underlined residues are from treatments according to GAP.

CROP Country, year (Variety)	Application				Day	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
Apples								
Australia, 1991 (Hi-Early Red Delicious)	WG		0.15	14	0	3.8		AUE-90-026
					7	3.7		
					14	<u>2.2</u>		
					22	<u>1.8</u>		
					28	<u>2.1</u>		
	WG		0.30	14	0	5.9		
					7	2.9		
					14	3.3		
					22	2.5		
					28	2.4		
Austria, 1983 (Golden Delicious)	WG	1.7	0.14	10	0	7.2		R72.21
					7	5.3		
					14	5.0		
					21	3.1		
					28	<u>1.4</u>		
Belgium, 1991 (Jonagold)	WP	2.4	0.24	8	0	5.9		R&H/BA 7.138/1991
					54	1.8		
					75	0.53		
Belgium, 1991 (Jonagold)	WP	2.4	0.80	8	0	2.7		R&H/BA 7.138/1991
					54	0.55		
					75	0.15		
Brazil, 1989	WP		0.13	2	1	3.1		FPA-89-007
					4	2.0		
					7	<u>1.5</u>		
					14	<u>0.39</u>		
					22	<u>0.28</u>		
Brazil, 1989	WP		0.26	2	1	3.9		FPA-89-007
					4	2.0		

CROP Country, year (Variety)	Application				Day	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
					7	1.7		
					14	0.78		
					22	0.67		
Hungary, 1986	SC	1.4	0.14	6	0	7.6[1]		R65.29
					1	6.4[1]		
					3	5.2[1]		
					7	3.6[1]		
					11	3.2[1]		
					18	1.9[1]		
					25	0.71[1]		
					32	0.39[1]		
Hungary, 1989	WG	2.3	0.23	1	1	1.5		R72.20
					3	1.3		
					5	0.56		
					7	0.58		
					9	1.6		
					29	0.27		
Japan, 1986 (Tsugaru)	WP	7.5	0.15	3	30	0.22	<0.01	Saku62P-2-54
					45	0.16	<0.01	
Japan, 1986 (Starking)	WP	7.5	0.15	3	30	0.58	<0.01	Saku62P-2-54
					45	0.30	<0.01	
					60	0.29	<0.01	
Netherlands, 1984 (Golden Delicious)	WP		0.12-0.16	10	49	0.21, <0.01	<0.002 (2)	PH8410
	SC		0.12-0.16	10	49	0.08, 0.02	0.004, <0.002	
	SC		0.11-0.14	10	49	0.14, <0.01	<0.002 (2)	
Netherlands, 1984 (Golden Delicious)	WP		0.12-0.16	9	58	0.08, <0.01	<0.002 (2)	PH8411
	SC		0.12-0.16	9	58	0.04, 0.12	<0.002 (2)	
	SC		0.11-0.14	9	58	<0.01 (2)	0.004, <0.002	
Netherlands, 1985 (Golden Delicious)	WP	1.2-1.6	0.12-0.16	9	81	<0.01 (2)	0.031, <0.002	PH8510
	SC	1.2-1.6	0.12-0.16	9	81	<0.01 (2)	0.029, 0.040	
	SC	1.2-1.6	0.11-0.14	9	81	<0.01 (2)	<0.002, 0.007	
Netherlands, 1985 (Golden Delicious)	WP	1.2-1.6	0.12-0.16	10	85	<0.01 (2)	0.016, 0.019	PH8512
	SC	1.2-1.6	0.12-0.16	10	85	<0.01 (2)	<0.002, 0.040	
	SC	1.2-1.6	0.11-0.14	10	85	<0.01 (2)	0.020, 0.027	
Netherlands, 1986 (Golden Delicious)	WP	1.2-1.6	0.12-0.16	7	88	<0.01, 0.03	<0.002 (2)	PH8610
	SC	1.2-1.6	0.12-0.16	7	88	<0.01, 0.02	<0.002 (2)	
	SC	1.2-1.6	0.12-0.16	7	88	<0.01 (2)	<0.002 (2)	
	SC	1.2-1.6	0.07-0.10	7	88	0.06, <0.01	<0.002 (2)	
	WP	1.2-1.6	0.12-0.16	7	88	<0.01	<0.002 (2)	
	SC	1.2-1.6	0.11-0.14	7	88	<0.01 (2)	<0.002 (2)	
Netherlands, 1987 (Golden Delicious)	WP	1.2-1.6	0.12-0.16	8	79	0.14, 0.10	0.003, <0.002	PH8711
	SC	1.2-1.6	0.12-0.16	8	79	0.18, 0.08	0.002 (2)	
	SC	1.2-1.6	0.12-0.16	8	79	0.04, 0.06	<0.002, 0.002	
	WG	1.2-1.6	0.12-0.16	8	79	0.17, 0.14	<0.002, 0.006	
	WP	1.2-1.6	0.12-0.16	8	79	0.10, 0.06	<0.002 (2)	
	SC	1.2-1.6	0.11-0.14	8	79	0.08, 0.04	0.002, 0.004	
Netherlands, 1987 (Golden Delicious)	WP	1.2-1.6	0.12-0.16	10	81	0.04, 0.06	0.003, 0.005	PH8712
	SC	1.2-1.6	0.12-0.16	10	81	0.04, 0.08	0.002, 0.004	
	SC	1.2-1.6	0.12-0.16	10	81	0.03, 0.03	0.002, 0.006	
	WG	1.2-1.6	0.12-0.16	10	81	0.06, 0.08	<0.002, 0.005	
	WP	1.2-1.6	0.12-0.16	10	81	0.08, 0.10	<0.002, 0.005	
	SC	1.2-1.6	0.11-0.14	10	81	<0.02 (2)	<0.002 (2)	
Netherlands, 1988 (Golden Delicious)	WP	1.2-1.6	0.12-0.16	9	71	0.14	0.003, 0.002	PH8845
	SC	1.2-1.6	0.12-0.16	9	71	0.13, <0.05	0.004, 0.003	
	WG	1.2-1.6	0.12-0.16	9	71	0.14, <0.05	<0.001, 0.003	
	SC	1.2-1.6	0.11-0.14	9	71	0.11, 0.14	0.002, 0.004	
Netherlands, 1988 (Golden Delicious)	WP	1.2-1.6	0.12-0.16	9	>63	0.32, 0.18	0.005 (2)	PH8847
	SC	1.2-1.6	0.12-0.16	9	>63	0.45, 0.48	0.006, 0.011	
	WG	1.2-1.6	0.12-0.16	9	>63	0.34, 0.43	0.004 (2)	

CROP Country, year (Variety)	Application				Day	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
	SC	1.2-1.6	0.11-0.14	9	>63	0.63, 0.33	0.011, 0.004	
Netherlands, 1989 (Golden Delicious)	WP	1.2-1.6	0.12-0.16	8	75	0.37, 0.46	0.006, <0.002	PH8959
Netherlands, 1990 (Jonagold)	WG SC	1.2-1.6 1.2-1.6	0.12-0.16 0.12-0.16	9 9	72 72	0.12, 0.10 0.09, 0.17	<0.002 (2) <0.002 (2)	PH9042
Netherlands, 1990 (Golden Delicious)	WG SC	1.2-1.6 1.2-1.6	0.12-0.16 0.12-0.16	8 8	65 65	<0.05, 0.15 <0.05, 0.14	<0.002 (2) <0.002 (2)	PH9044
PEARS								
Australia, 1992 (Beurre Bosc)	WG		0.15	6	0 8 14 21 28 42	1.3 1.0 <u>0.5</u> <u>0.6</u> <u>0.6</u> <u>0.3</u>		AUE-91-026
	WG		0.30	6	0 8 14 21 28 42	2.4 2.0 2.0 1.0 0.9 0.6		
Brazil, 1990	WP		0.16	3	0 14 21 35	2.8 <u>2.2</u> <u>2.0</u> <u>1.1</u>		094/90
Brazil, 1990	WP		0.32	3	0 14 21 35	3.6 2.5 2.2 2.0		094/90
Japan, 1986 (Kosui)	WP	6.0	0.15	3	30 45 60	0.38 <u>0.14</u> <u>0.10</u>	0.0004 0.005 0.005	Saku61P-6-136
Japan, 1986 (Hosui)	WP	6.0	0.15	5	30 45 60	0.47 0.18 0.10	0.016 0.007 0.008	Saku61P-6-136

[1] fruit without stalk.

Table 15. Mancozeb residues (as CS_2) in pome fruits from supervised trials in France. Underlined residues are from treatments according to GAP.

CROP Year (Variety)	Application [1]				Day	EBDC residues, mg/kgas CS_2	Ref.
	Form	kg ai/ha	kg ai/hl	No.			
APPLES							
1989 (Golden Delicious)	WP	1.4	0.28	8	122	0.45[2]	R73.21
1989 (Golden Delicious)	WP	1.4 +0.7 +1.4	0.9 +0.5 +0.9	1 +3 +3	135	0.05[2]	R73.20
1989 (Golden Delicious)	WP	2.1 +1.1 +2.1	0.21 +0.1 +0.21	1 +7 +8	104	0.2[2]	R73.18
1989 (Golden Delicious)	WP	2.1 +2.8 +1.1 +1.4	0.21 +0.28 +0.10 +0.14	1 +1 +2 +4	132	<u>0.25</u>[2]	R73.17
1989 (Golden Delicious)	WP	1.8	0.12	7	116	<u>0.1</u>	R73.13
1989 (Golden Delicious)	WP	1.8	0.12	6	110	<u>0.25</u>	R73.12
1990 (Golden Delicious)	WP	1.6	0.13	10	90	<u>0.07</u>	R79.4

CROP Year (Variety)	Application [1]				Day	EBDC residues, mg/kgas CS_2	Ref.
	Form	kg ai/ha	kg ai/hl	No.			
1990 (Golden Delicious)	WP	2.3 +2.0 +2.1 +2.3 +2.8 2.9		1 +1 +1 +1 +1 +1	56	1.1	R78.50
1990 (Bertane)	WP	2.0t[1] +1.0 +1.2 +1.0 +1.2 +1.0	0.66t +0.33 +0.4 +0.33 +0.4 +0.33	1t +3 +2 +1 +2 +1	123	<0.05	R78.67
1990 (Golden Delicious)	WP	2.4 +2.0	0.64 +0.53	1 +3	162	<0.05	R78.70
1990 (Golden Delicious)	WP	1.7	0.17	2	147	<0.05	R79.56
1990 (Golden Delicious)	WP	1.8 +1.4	0.15 +0.12	3 +1	126	0.1	R79.59
1990 (Golden Delicious)	WP	1.6 +2.0	0.16 +0.2	2 +1	107	0.3	R79.60
1990 (Granny Smith)	WP	2.0t +1.0		1t +16	11	<0.05	R78.69
1990 (Golden Delicious)	WP	1.9	0.16	7	89	0.28	R78.13
1990 (Starkrimson)	WP	2.0 +1.6 +0.8 +1.6 +2.0	0.4 +0.32 +0.16 +0.32 +0.4	2 +4 +3 +4 +1	86	<0.05	R78.14
1990 (Granny Smith)	WP	2.4 +0.8	0.16 +0.05	4 +1	6	1.8	R78.15
1990 (Welspur, Melrose, Granny Smith)	WP	1.4 +1.1	0.23 +0.21	10 +11	83	0.88 0.49 1.1 [3] c 0.08 [4]	R78.34
1991 (Golden Delicious)	WP	2.0-2.6	0.2-0.26	20	44	2.7 [2]	R80.32
1991 (Granny Smith)	WP	2		4	86	<0.1 [2]	R80.33
PEARS							
1990 (Beurre-Hardy)	SC	1.4 +1.1	0.23 +0.21	3 +7	83 7	0.34 0.72 c 0.08	R78.36
1990y(Doyenné de Comice)	SC	1.4 +1.1	0.23 +0.21	3 +7	83 7	0.24 0.22 c 0.15	R78.38
1990 (Williams)	SC	1.4 +1.1	0.23 +0.21	3 +7	83 7	0.44 0.84 c 0.05	R78.42
1990 (Passe Crassone)	SC	1.4 +1.1	0.23 +0.21	3 +7	83 7	0.28 0.40 c 0.14	R78.40

[1] t: thiram
[2] whole fruit without stalk
[3] 3 trials with 3 apple varieties.
[4] c: control sample.

Table 16. Mancozeb residues (as CS_2) in pome fruits from supervised trials in Germany. Underlined residues are from treatments according to GAP.

CROP Year (Variety)	Application				Day	Residues, mg/kg [1]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
Apples								
1982 (Roter Boskoop)	WP	2.4	0.48	10	0 7 14	4.8 2.3 2.6	<0.02	R68.7
1982 (Jonathon)	WP	2.4	0.16	12	0 7 14	2.2 2.2 1.9	<0.02	R68.7
1982 (Cox's Orange)	WP	2.4	0.16	12	0 7 14	8.7 1.8 4.0	<0.02	R68.7

CROP Year (Variety)	Application				Day	Residues, mg/kg [1]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
1983 (Gravenstein)	WP	3.4	0.16	10	21 28	0.26 0.34	 <0.02	R68.4
1983 (Cox's Orange)	WP	2.4	0.16	10	21 28	0.68 0.59	s <0.02 <0.02, s <0.02	R65.13
1983 (Boskoop)	WP	2.4	0.5	10	21 28	4.0 2.4	s 0.1 <0.02, s 0.1	R65.13
1986 (Idared)	WG	2.3	0.15	15	0 7 14 21 28 35	0.76 0.79 0.97 0.87 0.63 0.36	 <0.02 <0.02	R65.28
1986 (Cox's Orange)	WG	2.3	0.15	12	0 14 21 28 35	3.0 1.8 1.7 2.6 1.1	 <0.02 	R65.28
1986 (Idared)	WG	2.3	0.45	12	0 7 14 21 28 35	2.9 1.7 1.3 1.4 1.1 1.1	 <0.02 	R65.28
1986 (Cox's Orange)	SC	2.4	0.16	12	0 14 21 28 35	2.1 2.3 3.7 4.1 2.8	 <0.02 	R65.27
1986 (Idared)	SC	2.4	0.48	12	0 7 14 21 28 35	3.9 3.9 2.8 2.5 2.9 2.8	 <0.02 	R65.27
1986 (Cox's Orange)	WP	2.4	0.16	10	0 14 21 28 35	4.2 3.7 2.2 3.0 2.2	 <0.02 	R65.26
1986 (Idared)	WP	2.4	0.16	14	0 7 14 21 28 35	0.70 1.8 1.3 0.78 0.92 0.33	 <0.02 <0.02	R65.26
1986 (Idared)	WP	2.4	0.48	12	0 7 14 21 28 35	2.9 3.0 2.2 2.2 1.5 1.5	 <0.02 	R65.26
1991 (Gloster)	WG	2.3	0.75	8	0 28 35 41 41	2.4 1.1 0.56 0.56 w 0.49		R80.38
1991 (Cox's Orange)	WG	1.5	0.15	8	0 42	1.4 0.09		R80.38
1991 (Jonagold)	WP	1.6	0.64	8	0 28 35 42 49 56	8.9 0.54 0.37 0.35 0.26 0.28		R80.39
1991 (Golden)	WG	2.4	0.48	8	0 28 35 42	4.0 0.72 0.53 0.74		R80.39
1991 (Gloster)	WG	2.4	0.16	8	0 28 35 41	4.2 1.2 1.0 0.67		R80.39
1991 (Cox's Orange)	WG	1.6	0.16	8	0 42 49 56	1.0 0.14 0.07 0.06		R80.39
PEAR								

CROP Year (Variety)	Application				Day	Residues, mg/kg [1]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
1982 (Conference)	WP	2.4	0.48	10	0 7 14	5.6 3.5 2.3	 0.04	R68.7
1982 (Charneu)	WP	2.4	0.16	12	0 7 14	8.3 6.6 4.0	 <0.02	R68.7
1982 (Charneu)	WP	0.8	0.05	14	0 3 7	1.3 1.4 <0.05	 <0.02	R68.7
1983 (Charneu)	WP	2.4	0.16	10	21 28	2.0 1.5	cm <0.02 <0.02, cm <0.02	R65.13
1983 (Conference)	WP	2.4	0.5	10	21 28	1.9 1.1	cm <0.02 <0.02, cm 0.02	R65.13
1983 (Williams)	WP	2.4	0.16	10	21 28	0.93 0.64	 0.09	R68.4
1983	WP	2.4	0.48	10	21 28	1.9 1.1	 <0.2	R65.13

[1] s: sauce. w: washed fruit. cm: compote.

Table 17. Mancozeb residues (as CS_2) in pome fruits from supervised trials in Italy. Analyses were on whole fruit without stalk. Underlined residues are from treatments according to GAP.

CROP Year (Variety)	Application				Day	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
Apples								
1989 (Double Red)	WP	7.4	0.2	1	10	1.6	<0.01	R72.5/6/7
1989 (Double Red)	WP	2.1 +3.2 +4.2	0.16	1 +4 +7	10[1] 97[1]	0.73 0.77	<0.01 <0.01	R72.5/6/7
1989 (Double Red)	WG	2.1 +3.2 +4.2	0.16	1 +4 +7	97	0.15	<0.01	R72.5/6/7
1989 (Double Red)	SC	2.1 +3.2 +4.2	0.16	1 +4 +7	97	0.77	<0.01	R72.5/6/7
1989 (Double Red)	WP	2.1 +3.2 +4.2 +5.6	0.16	1 +4 +7 +5	29[1] 29[1] 29[1]	0.67 0.48 0.88	<0.01 <0.01 <0.01	R72.5/6/7
1989 (Double Red)	WG	2.1 +3.2 +4.2 +5.6	0.16	1 +4 +7 +5	29	0.76	<0.01	R72.5/6/7
1990 (Rome Beauty)	WP	2.6	0.16	10 15 16 17	132 56 42 28	<0.1 0.72 1.3 1.7	<0.01 <0.01 0.01 0.02	R75.5
1990 (Golden)	WP	1.6-2.0	0.12- 0.16	14	28 42	0.64 0.47	<0.01 <0.01	R75.5
1990 (Golden)	WP	1.6-2.0	0.12- 0.16	8 11	104 55	<0.1 0.14	<0.01 <0.01	R75.5
1990 (Morgenduft)	WP	1.6	0.16	8	116	<0.1	<0.01	R75.5
1990 (Morgenduft)	WP	1.6 +2.4	0.16	8 +5	52	0.38	<0.01	R75.5
1990 (Morgenduft)	WP	1.6 +2.4	0.16	8 +6	37	0.92	<0.01	R75.5
1990 (Morgenduft)	WP	1.6 +2.4	0.16	8 +7	19 28	1.4 1.3	 <0.01	R75.5
1990 (Jonathan)	WP	1.9 +2.4	0.16	3 +4	89	0.32	<0.01	R75.5
1990 (Jonathan)	WP	1.9 +2.4	0.16	3 +6	62	0.37	0.01	R75.5

CROP Year (Variety)	Application				Day	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
1990 (Jonathan)	WP	1.9 +2.4	0.16	3 +7	42	0.38	0.01	R75.5
1990 (Jonathan)	WP	1.9 +2.4	0.16	3 +8	28	0.82	0.01	R75.5
1990 (Jonathan)	WP	1.9 +2.4	0.16	3 +9	14	1.6	0.01	R75.5
1990 (Super Stark)	WP	1.6	0.16	3	155	<0.1	<0.01	R75.5
1990 (Super Stark)	WP	1.6 +1.9	0.16	3 +3	123	<0.1	<0.01	R75.5
1990 (Super Stark)	WP	1.6 +1.9 +2.2	0.16	3 +3 +4	55	0.26	0.03	R75.5
1990 (Super Stark)	WP	1.6 +1.9 +2.2	0.16	3 +3 +5	28	0.40	<0.01	R75.5
1990 (Hi Early Starking)	WP	2.4	0.16	15 18 20 21	98 56 42 28	0.31 0.52 0.91 1.4		R75.5
1990 (Golden Granny)	WP	2.4	0.16	7	114	<0.1		R75.5
1990 (Badami)	WP	2.4	0.16	13	56	0.20		R75.5
1990 (Neijpling Early)	WP	2.4	0.16	14	42	0.86		R75.5
1990 (Acrynae)	WP	2.4	0.16	15	28	1.5		R75.5
1990 (Cooper)	WP	2.0	0.16	10	92	<0.1		R75.5
1990 (Golden)	WP	2.0	0.16	13 14 15	56 42 28	0.60 0.61 0.79		R75.5
1990 (Cooper 7)	WP	2.4	0.16	13	111	0.59		R75.5
1990 (Perleberg)	WP	2.4	0.16	19	56	1.2		R75.5
1990 (Starkrimson)	WP	2.4	0.16	21 22	42 28	1.2 1.2		R75.5
1990 (Golden)	WP	2.9	0.16	12 16 18	90 56 28	0.36 0.68 1.1		R75.5
1990 (Golden Stark)	WP	2.4	0.24	10 15 17 18	123 56 42 28	0.15 0.37 0.74 1.9		R75.5
1990 (Golden)	WP	2.4	0.8	9 13 13 14	113 56 42 28	<0.1 0.42 0.57 0.91		R75.5
1990 (Double Red)	WP	3.2	0.16	14	92	0.19		R75.5
1990 (Low Red)	WP	3.2	0.16	17 20 22	56 48 28	0.36 0.63 0.92		R75.5
PEARS								
1990 (William)	WP	2.6	0.16	9 10 11 12	71 56 42 28	0.18 0.30 0.41 0.76	<0.01 <0.01 <0.01 <0.01	R75.5
1990 (William)	WP	2.4	0.16	7 13 14 15	114 56 42 28	<0.1 0.13 0.74 1.7		R75.5

[1] trials with different formulations.

Table 18. Mancozeb residues (as CS_2) in apples from supervised trials in the UK. Underlined residues are from treatments according to GAP.

Year (Variety)	Application				Day	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
1989 (Bramley)	WP	3.6	0.36	3	0 7 14 28 46 83	6.7[1] 2.8[1] 1.2[1] 0.58[1] 0.38 0.14	<0.006[1]	R71.16
1989 (Cox)	WP	2.4 +1.7 +3.6	0.48 +0.34 +0.90	2 +1 +1	0 7 14 28 46 83	5.4[1] 1.6[1] 1.6[1] 0.39[1] 0.24 0.78	<0.006[1]	R71.16
1989 (Bramley)	WP	4.0 +3.4	0.8 +0.7	2 +2	0 7 14 28 46 84	4.0[1] 2.8[1] 3.5[1] 0.30[1] 0.60 <0.05	<0.006[1]	R71.16
1989 (Bramley)	WP	3.4-3.8	0.9-1.1	5	0 7 14 28 46 88	2.2[1] 4.6[1] 2.5[1] 2.3[1] 2.6 1.1	0.16[1]	R71.16
1989 (Bramley)	WG	1.6	0.32	3	31 69	0.13[2] 0.13[2]		R71.16
1989 (Bramley)	WG	3.4 +1.6 +3.0	0.68 +0.32 +0.60	2 +1 +1	19 57	1.4[2] 0.14[2]		R71.16
1989 (Idared) (Golden) (Cox) (Red Delicious)	WG WG WG WG	3.4+1.6 3.4+1.6 3.4+1.6 3.4+1.6	0.7+0.3 0.7+0.3 0.7+0.3 0.7+0.3	2+1 2+1 2+1 2+1	69 69 69 69	 0.12[2] 0.11[2] 0.13[2] 0.12[2]		R71.16
1989 (Bramley)	WG	1.6	0.32	2	57	0.17[2]		R71.16
1989 (Bramley)	WG	3.4	1.7	2	26 57	1.5[2] 0.36[2]		R71.16
1989 (Cox)	WG	2.4 +1.7 +3.4	0.48 +0.34 +0.68	2 +1 +1	28	0.35[2]		R71.16
1989 (Cox)	WG	1.6 +2.5	0.32 +0.50	2 +1	42	0.10[2]		R71.16
1989 (Bramley)	WG WG	1.7 +2.6 1.7 +2.6		1 +1 1 +2	44 96 34 87	<0.05[2] <0.05[2] 0.39[2] <0.05[2]		R71.16
1989 (Bramley)	WG	1.6 +2.1	0.53 +0.70	2 +1	29 71	0.77[2] 0.49[2]		R71.16
1989	WG	1.6 +2.5	0.38 +0.56	1 +3	3 25 70	4.5[2] 1.1[2] 0.31[2]		R71.16

[1] nut-sized immature fruit. [2] fruit without stalk.

Table 19. Mancozeb residues (as CS_2) in pome fruits from supervised trials in the USA (Loftus, 1991. ETU 91-02). Underlined residues are from treatments according to GAP.

CROP State, year (Variety)	Application	Day [1]	Residues, mg/kg [2]	Ref.

	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
APPLES								
MI, 1990 (Paula Red)	WP	5.4		4	fb 96	0.01	<0.005	ETU 91-02
MI, 1990 (Paula Red)	WP	2.7		4	fb 96	<0.01	<0.005	ETU 91-02
MI, 1990 (Paula Red)	WP	5.4		7	70	<0.01	<0.005	ETU 91-02
MI, 1990 (Paula Red)	WP	2.7		7	70	<0.01	<0.005	ETU 91-02
MI, 1990 (Paula Red)	WP	5.4		12	0 10 21 38	3.2 0.78 0.25 0.08	0.031 <0.005 0.011 <0.005 c 0.006	ETU 91-02
NY, 1990 (Twenty Ounce)	WP	5.4		4	fb 126	<0.01	<0.005	ETU 91-02
NY, 1990 (Twenty Ounce)	WP	2.7		4	fb 126	<0.01	<0.005	ETU 91-02
NY, 1990 (Twenty Ounce)	WP	5.4		7	77	0.05	<0.005	ETU 91-02
NY, 1990 (Twenty Ounce)	WP	2.7		7	77	<0.01	<0.005	ETU 91-02
NY, 1990 (Twenty Ounce)	WP	5.4		12	42	0.49	0.016	ETU 91-02
OH, 1990 (MacIntosh)	WP	10.8		2	fb 120	0.02	<0.005	ETU 91-02
OH, 1990 (MacIntosh)	WP	5.4		2	fb 120	<0.01	<0.005	ETU 91-02
OH, 1990 (MacIntosh)	WP	10.8 +5.4		2 +3	75	0.16	<0.005	ETU 91-02
OH, 1990 (MacIntosh)	WP	5.4 +2.7		2 +3	75	0.07	<0.005	ETU 91-02
OH, 1990 (MacIntosh)	WP	10.8 +5.4		2 +8	42	0.22	0.017	ETU 91-02
PA, 1990 (Empire)	WP	5.4		4	fb 119	<0.01	<0.005	ETU 91-02
PA, 1990 (Empire)	WP	2.7		4	fb 119	0.01	<0.005	ETU 91-02
PA, 1990 (Empire)	WP	5.4		7	74	0.02	<0.005	ETU 91-02
PA, 1990 (Empire)	WP	2.7		7	74	0.08	<0.005	ETU 91-02
PA, 1990 (Empire)	WP	5.4		12	42	0.32	0.022	ETU 91-02
VA, 1990 (Red Delicious)	WP	7.2		3	fb 110	0.08	0.005	ETU 91-02
VA, 1990 (Red Delicious)	WP	7.2		3	fb 110	0.13	<0.005	ETU 91-02
VA, 1990 (Red Delicious)	WP	7.2 +5.4		3 +3	70	0.34	<0.005	ETU 91-02
VA, 1990 (Red Delicious)	WP	3.6 +2.7		3 +3	70	0.31 c 0.03	<0.005	ETU 91-02
VA, 1990 (Red Delicious)	WP	7.2 +5.4		3 +8	42	1.8	0.033	ETU 91-02
WA, 1990 (Red Delicious)	WP	5.4		4	fb 158	<0.01	<0.005	ETU 91-02
WA, 1990 (Red Delicious)	WP	2.7		4	fb 158	<0.01	<0.005	ETU 91-02
WA, 1990 (Red Delicious)	WP	5.4		7	104	0.16	<0.005	ETU 91-02
WA, 1990 (Red Delicious)	WP	2.7		7	104	0.17	<0.005	ETU 91-02
WA, 1990 (Red Delicious)	WP	5.4		12	21 42	0.73 1.3	0.034 0.031	ETU 91-02
PEARS								
CA, 1985 (Bartlett)	WP	1.8	0.19	6	7 14 22	5.5 3.3 2.4	0.01 <0.01 <0.01	85-0223

CROP State, year (Variety)	Application				Day [1]	Residues, mg/kg [2]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
CA, 1985 (Bartlett)	WP	1.8	0.19	6	8 15 22	5.5 3.4 1.9	0.02 0.01 <0.01	85-0224
PA, 1985 (Bartlett)	WP	7.2	0.19	6	7 14 21	2.9 2.4 1.5	0.046 0.054 0.048	85-0315

[1] fb: final application at full bloom.
[2] c: control sample.

Table 20. Mancozeb residues (as CS_2) in stone fruits from supervised trials in Australia, Brazil and France. Underlined residues are from treatments according to GAP.

CROP Country, year (Variety)	Application				Day	Residues, mg/kg[1], EBDC as CS_2	Ref.
	Form	kg ai/ha	kg ai/hl	No.			
APRICOT							
Australia, 1992 (Moorpark)	WG		0.15	6	0 7 14 21	6.3 4.0 2.2 1.3	AUI-91-034
	WG		0.30	6	0 7 14 21	20 11 5.0 2.9	
Australia, 1992 (Moorpark)	WP		0.16	6	0 7 14 21	7.9 3.3 2.1 1.2	AUI-91-034
	WP		0.32	6	0 7 14 21	16 11 4.5 3.3	
PEACH							
Australia, 1991 (Red Haven)	WG		0.15	4	43 51 57	2.0 1.0 1.6	AUE-91-027
	WG		0.30	4	43 51 57	3.7 3.0 2.5	
Brazil, 1989	WP		0.16	4	0 14 21 35	1.1 0.95 0.39 0.1	072/90
Brazil, 1989	WP		0.32	4	0 14 21 35	2.2 1.1 0.56 0.45	072/90
PLUMS							
Brazil, 1990	WP		0.16	4	0 14 21 28	2.7 1.4 0.45 0.28	071/90
Brazil, 1990	WP		0.32	4	0 14 21 28	3.4 2.5 0.84 0.36	071/90
France, 1990 (Ente 707)	WP	1.9	0.15	4	62	0.14 c 0.08	R78.54/5
France, 1990 (Ente 707)	WP	1.6	0.16	3	54	0.16	R78.56
France, 1990 (Mirabellier)	WP	1.6 1.6 1.6 1.6		1 4 5 6	88 67 48 34	0.48[2] 0.33[2] 0.49[2] 0.55[2]	R78.59

[1] c: control sample
[2] fruit without stone

Table 21. Mancozeb residues (as CS_2) in berry fruits from supervised trials in Australia, Brazil, Hungary, Japan, Portugal and Spain. Underlined residues are from treatments according to GAP.

CROP Country, year (Variety)	Application				Day	Residues, mg/kg [2]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
GRAPES								
Australia, 1990 (Rhine Reisling)	WP		0.16	3 4	7[1] 0 7 14 28	29 50 36 29 13		867/90
	WP		0.32	3 4	7[1] 0 7 14 28	49 83 59 38 38		
Australia, 1990 (Rhine Reisling)	WG		0.15	3 4	7[1] 0 7 14 28	22 42 29 16 12		867/90
	WG		0.30	3 4	7[1] 0 7 14 28	30 57 40 31 20		
Australia, 1990 (Rhine Reisling)	WG		0.15	2 3	19[1] 0 7 14 28 42 56	13 39 29 25 14 5.6 4.5	0.33 0.78 0.69 0.32 0.28 0.14 0.10	868/90 868/90/5
	WG		0.30	2 3	19 0 7 14 28 42 56	23 55 36 40 26 6.7 5.6	0.43 0.81 0.67 0.58 0.36 0.22 0.15	
Brazil, 1990	WP	2.8		2	0 7 14 21	2.0 1.7 0.56 <0.03		030/90
Brazil, 1990	WP	5.6		2	0 7 14 21	3.9 3.6 1.1 0.17		030/90
Hungary, 1986	SC	1.4	0.13	7	0 2 3 6 10 14 20 30 37	0.41 0.59 0.58 0.52 0.53 0.63 0.67 0.49 0.34		R65.33
Japan, 1989 (Delaware)	WP	1.9	0.075	2	46 60	0.59 0.04	0.02 <0.01	Saku1P-6-139
Japan, 1989 (Delaware)	WP	1.9	0.075	2	42 60	0.81 0.12	0.04 <0.01	Saku1P-6-139
Japan, 1989 (Kyoh_)	WP	1.9	0.075	2	45 60	0.56 0.15	0.03 <0.01	Saku1P-6-139
Japan, 1989 (Kyoh_)	WP	1.9	0.075	2	45 60	0.09 0.04	<0.01 <0.01	Saku1P-6-139
Portugal, 1991 (Cardinal)	WP	0.93 +1.6	0.38 +0.66	3 +3	43	0.91[2]		R80.27
STRAWBERRIES								
Japan, 1983 (Reik_)	WP	1.9	0.13	3 6	97 76	0.04 0.05		?
Japan, 1983 (Reik_)	WP	1.9	0.13	3 6	97 76	0.05 0.06		?

CROP Country, year (Variety)	Application				Day	Residues, mg/kg [2]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
Spain, 1985 (Cruz)	WP	3.2	0.16	1	0 4 7 14 21	13 6.0 4.3 3.2 2.0		R66.22/23
Spain, 1985 (Cruz)	WP	3.2	0.16	1	0 3 7 14 21	3.5 2.2 2.0 1.8 1.5		R66.22/23
Spain, 1986 (Douglas)	WP	4.8	0.24	3	0 3 7 14 21	5.0 3.7 2.8 1.8 0.4		R66.22/23

[1] sampled one hour before the final application
[2] whole cluster

Table 22. Mancozeb residues (as CS_2) in cranberries from supervised trials in the USA. Underlined residues are from treatments according to GAP.

State, year (Variety)	Application				Day	Residues, mg/kg [1]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
WA, 1985 (McFarlin)	WP	4.5	0.60	3	15 30 44	0.55 0.24 0.073	0.01 <0.01 <0.01	85-0294
WA, 1985 (McFarlin)	WP	5.4	0.72	3	15 30 44	0.50 0.29 0.11	<0.01 <0.01 <0.01	85-0295
OR, 1985 (McFarlin)	WP	5.4	1.9	4	15 30 45	13 3.1 2.5 c 0.03	0.054 0.025 0.025 c 0.01	85-0341
NJ, 1985 (Franklin)	WP	5.4	0.19	3	2 17 31	6.4 2.4 1.5	0.02 0.02 0.01	85-0456
NJ, 1985 (Early Black)	WP	5.4	0.19	3	15 29 47	0.31 0.15 0.059	0.02 0.02 0.02	85-0457
NJ, 1985 (Early Black)	WP	5.4	0.19	3	29 46	0.29 0.12	0.01 <0.01	85-0458
MA, 1988 (Crowley)	WP	5.4		4	30	2.7	0.058	88-0282

[1] c: control sample.

Table 23. Mancozeb residues (as CS_2) in grapes from supervised trials in France. Underlined residues are from treatments according to GAP.

Year (Variety)	Application				Day	Residues, mg/kg [1]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
1975 (Chardonnay + Pinot Meunier)	WP	2.8 +0.7 +0.6 +0.8		6 +1 +1 +1	56 71	w <0.1 w <0.1	w <0.02 w <0.02	R60.9
1976 (Pinot Meunier)	WP	3.0 +2.4 +1.2		4 +1 +1	74	w <0.1	w <0.02	R60.9
1976 (Pinot Meunier + Chardonnay)	WP	2.8 +1.0		5 +1	54 64	w <0.1 w <0.1	w <0.02 w <0.02	R60.9
1976 (Pinot Noir + Pinot Meunier)	WP	2.8 +1.0		3 +3	60 66	w <0.1 w <0.1	w <0.02 w <0.02	R60.9
1976 (Chardonnay + Pinot Meunier)	WP	2.1 +2.8 +1.0		2 +2 +2	53 62	w <0.1 w <0.1	w <0.02 w <0.02	R60.9
1988 (Carbernet + Merlot)	WP	1.2 +1.4 +0.4		1 +8 +5	44	w <0.05		R78.78
1988 (Carbernet + Merlot)	WP	1.4 +0.4		8 +2	38	w <0.05		R78.82

Year (Variety)	Application				Day	Residues, mg/kg [1]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
1988 (Carbernet + Merlot)	WP	2.5 +4.6 +2.1 +1.4 +2.3 +0.4 +1.4 +2.3		1 +1 +1 +3 +1 +1 +2 +1	39	w <0.05		R78.89
1988 (Carbernet + Merlot)	SC	0.95 +1.4 +3.0 +1.6		1 +4 +1 +3	48	w <0.05		R79.1
1989 (Carbernet + Merlot)	WP	2.5 +1.4 +0.4		2 +4 +2	46	w <0.05		R78.85
1990 (Pinot Noir)	WP	1.4 +0.4	0.35 +0.1	4 +4	31	1.0 w 0.23		R78.44
1990 (Pinot)	WP	1.4	0.42	2	118	<0.05[2] w 0.1		R78.57
1990 (Carignan)	WP	1.0 +0.91 +1.0 +0.32 +0.28 +0.32	0.87 +0.76 +0.87 +0.26 +0.23 +0.26	1 +1 +2 +2 +1 +3	43	0.66[2] w <0.05		R78.62/63
1990 (Gamay)	WP	1.4 +0.4	0.56 +0.15	6 +1	37	1.1[2] w <0.05		R79.5
1990 (Carignan)	WP	1.6	1.1	6	47 54	0.21[2] 0.4 j <0.05		R79.26
1990 (Carignan)	WP	1.4	0.93	5	52	0.7[2] w <0.05		R79.30
1990 (Cabernet Sauvignon)	WP	1.8 +1.2 +0.4	1.2 +0.77 +0.26	2 +3 +2	69	0.13[2]		R78.46
1990 (Pinot Noir)	WP	2.8	2.8	8	61	1.8[2]		R78.69
1990 (Carignan)	WP	2.8	1.1	8	32	3.2[2] c 0.32		R78.64
1990 (Pinot Meunier)	WP	2.8	2.8	6	68	0.44[2]		R78.65
1990 (Meunier)	WP	2.8	1.9	8	71	2.0[2] c 0.12		R78.66
1990 (Auxerrois)	WP	2.8	1.3	4	117	0.48[2]		R78.68
1990 (Merlot)	WP	2.4 +1.4 +1.6 +0.36	0.96 +0.56 +0.62 +0.14	2 +5 +1 +2	23	2.0[2] w 0.09		R78.71
1990 (Ugni blanc)	WP	2.8	0.56	10	38	2.0[2] c 0.08		R79.8
1990 (Malbec)	WP	2.8 +1.4 +0.4		2 +2 +2	81	0.87[2]		R79.13
1990 (Syrah)	WP	2.8	1.4	8	24	4.1[2] c 0.23		R79.16
1990 (Cabernet Sauvignon)	WP	2.8	0.56	10	13	1.5[2] c 1.8		R79.29
1990	WP	3.2 +1.0		4 +3	93	0.35[2]		R79.50/51
1991 (Grenache)	WP	0.7 +2 +1.8 +2.4 +0.4	0.46 +1.3 +1.2 +1.6 +0.26	1 +1 +1 +1 +2	28	0.28[2]		R80.13
1991 (Merlot)	SC	1.4 +1.8 +1.2 +1.5 +1.7	0.9 +1.2 +0.76 +0.98 +1.1	1 +1 +1 +2 +2	46	1.2[2]		R80.24
1991 (Carignan)	WP	1.2 +1.6	0.48 +0.64	4 +1	40	1.3[2]		R80.34
1991 (Sauvignon)	WG	2.8 +1.4 +1.6	1.9 +0.93 +1.1	3 +3 +2	30	w <0.1		R79.65

Year (Variety)	Application				Day	Residues, mg/kg [1]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
		+2.8 +1.4 +0.4	+1.9 +0.93 +0.26	+3 +3 +2				
1991 (Merlot Noir)	WG	2.8 +1.4 +1.6 +2.8 +1.4 +0.4	1.9 +0.93 +1.1 +1.9 +0.93 +0.26	3 +3 +2 +3 +3 +2	39	w <0.1		R79.73

[1] j: juice. w: wine. c: control sample.
[2] whole cluster

Table 24. Mancozeb residues (as CS_2) in grapes from supervised trials in Italy. Underlined residues are from treatments according to GAP.

Year (Variety)	Application				Day	Residues, mg/kg [1]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
1989 (Barbera)	WP	1.3	0.16	6	28	2.1	<0.01	R72.1
1989 (Barbera)	WG	1.3	0.16	6	28	2.1	<0.01	R72.1
1989 (Barbera)	WP	1.3	0.16	6	28	2.1	<0.01	R72.1
1989 (Barbera)	SC	1.3	0.16	6	28	2.8	<0.01	R72.1
1989 (Garganega)	WP	1.4 +0.4	0.1 +0.03	4 +4	39	0.2 m 0.1 w <0.1	<0.01 m <0.01 w <0.01	R72.1
1989 (Garganega)	WP	1.4	0.1	4	90	0.13 m <0.1 w <0.01	<0.01 m <0.01 w <0.01	R72.1
1989 (Merlot)	WP	0.38 +0.50 +0.56 +0.44 +0.56 +0.64	0.018 to 0.032	1 +3 +3 +1 +3 +1	37	0.16 w <0.1	<0.01 w <0.01	R72.1
1989 (Refosco)	WP	0.8 +1.0 +1.2 +0.38	0.1 +0.1 +0.12 +0.038	2 +2 +1 +5	31	0.27 w <0.1	0.01 w <0.01	R72.1
1989 (Trebbiano)	WP	1.5 +0.48 +1.5	0.037 to 0.15	4 +4 +1	28	0.2	<0.01	R72.1
1989 (Trebbiano)	WP	1.0 +1.25 +1.5 +0.48	0.032 to 0.15	1 +1 +2 +7	43	0.25	<0.01	R72.1
1989 (Sangiovese)	WP	1	0.1	3	84	<0.1	<0.01	R72.1
1990 (Pinot Nero)	WP	1.1 +0.9 +1.1 +1.3 +0.36	0.088 +0.075 +0.075 +0.087 +0.024	1 +1 +1 +1 +4	35	0.56 m 0.20 w <0.1	w <0.01	R75.1
1990 (Pinot Nero)	WP	1.05 +0.90 +1.1 +1.3	0.088 +0.075 +0.075 +0.087	1 +1 +1 +1	95	0.60 m 0.30 w <0.1	w <0.01	R75.1
1990 (Müller Thurgau)	WP	0.65 +0.80 +0.22 +0.25	0.048 to 0.170	1 +1 +2 +1	35	<0.1 m <0.1 w <0.1	m <0.01 w <0.01	R75.1
1990 (Chardonnay)	WP	0.65 +0.80		1 +1	84	<0.1 m <0.1 w <0.1	m <0.01 w <0.01	R75.1
1990 (Sangiovese)	WP	0.28	0.09	8	35	0.27 m <0.1 w <0.1	m <0.01 w <0.01	R75.1
1990 (Sangiovese)	WP	0.28		4	83	0.17 m <0.1 w <0.1	m <0.01 w <0.01	R75.1
1990 (Trebbiano)	WP	2.1 +1.5 +0.36	0.024 to 0.16	1 +3 +7	38	0.2 m <0.1 w <0.1	m <0.01 w <0.01	R75.1

Year (Variety)	Application				Day	Residues, mg/kg [1]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
1990 (Trebbiano)	WP	2.1 +1.5		1 +3	121	<0.1 m <0.1 w <0.1	m <0.01 w <0.01	R75.1
1990 (Italia)	WP	1.0	0.1	6	29	0.3	<0.01	R75.1
1990 (Sangiovese)	WP	2.0 +0.56	0.1 +0.028	2 +5	30	0.4		R75.1
1990 (Sangiovese)	WP	3.2	0.16	2	111	<0.25		R75.1
1990 (Sangiovese)	WP	1.9		5	37	0.52		R75.1
1990 (Sangiovese)	WP	1.9		3	98	<0.25		R75.1
1990 (Sangiovese)	WP	0.8	0.1	5	37	0.29		R75.1
1990 (Sangiovese)	WP	0.26	0.032	5	37	<0.25		R75.1
1990	SC		0.16 0.12	1 1	79 79	0.08 0.13		4579-6/2

[1] m: must; w: wine.

Table 25. Mancozeb residues (as CS_2) in black currants from supervised trials in the UK. Underlined residues are from treatments according to GAP.

Year (Variety)	Application				Day	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		Dithiocarbamates as CS_2	ETU	
1990 (Ben Lomond)	WG	2.3	0.11	5	0 6 15 21 31	7.0 2.9 2.7 2.0 2.0	 0.016	R80.35
1990 (Baldwin)	WG	2.3	0.10	5	0 6 15 21 31	11 5.3 4.4 3.5 3.0	 0.071	R80.35
1990 (Baldwin)	WG	2.3	0.11	5	0 7 19 21 31	13 6.7 3.5 3.4 3.0	 0.032	R80.35
1990 (Baldwin)	WG	2.3	0.05	6	2 27	8.0 5.1	 0.18	R80.35
1990 (Baldwin)	WG	2.3	0.3	7	0 26	14 4.3	 0.084	R80.35
1990 (Baldwin)	WG	2.3	0.05	8	0 24	17 5.4	 0.012	R80.35
1991 (Ben Lomond)	WG	2.3	0.11	5	0 21	5.2 2.6		R80.36
1991 (Ben Lomond)	WG	2.3	0.11	5	0 27	4.2 1.4		R80.36
1991 (Ben Lomond)	WG	2.3	0.11	5	0 20	2.9 3.0		R80.36

Table 26. Mancozeb residues (as CS_2) in tropical and subtropical fruits from supervised trials in Australia, Brazil, Honduras and Japan. Underlined residues are from treatments according to GAP.

CROP Country, year (Variety)	Application [1]	Day	Residues, mg/kg [2]	Ref.

	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
AVOCADO								
Brazil, 1982	WP		0.18 0.36	3 3	21 21	0.60 0.80		SR 179/82
Bananas								
Australia, 1989 (Cavendish)	SC	1.0		7	1 7 14 28	pu <0.1, pe 1.3, f 0.6 pu <0.1, pe 1.7, f 0.8 pu <0.1, pe 1.3, f 0.6 pu <0.1, pe 0.8, f 0.4	pu <0.1, pe <0.1, f <0.1 pu <0.1, pe <0.1, f <0.1 pu <0.1, pe <0.1, f <0.1 pu <0.1, pe <0.1, f <0.1	2495/89/5 2495/89
Australia, 1989 (Cavendish)	SC	1.5		7	1 7 14 28	pu <0.1, pe 1.9, f 0.9 pu <0.1, pe 1.3, f 0.6 pu <0.1, pe 1.5, f 0.7 pu <0.1, pe 0.3, f 0.2	pu <0.1, pe <0.1, f <0.1 pu <0.1, pe <0.1, f <0.1 pu <0.1, pe <0.1, f <0.1 pu <0.1, pe <0.1, f <0.1	2495/89/5 2495/89
Australia, 1989 (Cavendish)	SC	1.8		7	1 7 14 28	pu <0.1, pe 3.3, f 1.4 pu <0.1, pe 2.5, f 1.1 pu <0.1, pe 2.1, f 1.0 pu <0.1, pe 0.6, f 0.4	pu <0.1, pe <0.1, f <0.1 pu <0.1, pe <0.1, f <0.1 pu <0.1, pe <0.1, f <0.1 pu <0.1, pe <0.1, f <0.1	2495/89/5 2495/89
Australia, 1989 (Cavendish)	WP	1.8		7	1 7 14 28	pu <0.1, pe 2.7, f 1.2 pu <0.1, pe 1.9, f 0.9 pu <0.1, pe 1.2, f 0.6 pu <0.1, pe 1.0, f 0.5	pu <0.1, pe <0.1, f <0.1 pu <0.1, pe <0.1, f <0.1 pu <0.1, pe <0.1, f <0.1 pu <0.1, pe <0.1, f <0.1	2495/89/5 2495/89
Brazil, 1986 (Nanacao)	WP	3.5 7.0		4 4	21 21	0.23 0.75	<0.01 0.05	86-0091
Honduras, 1988 (Grand Nain)	SC	2.1	0.29	a 45	9	0.11 pe 0.51 pu <0.005	<0.01 pe <0.01 pu 0.01	88-0040
FIGS								
Brazil, 1982	WP		0.16	3	7 21	1.1 0.62		181/82
Brazil, 1982	WP		0.32	3	7 21	2.8 1.6		181/82
MANGO								
Australia, 1990 (Kensington)	WP		0.16	9	1 7 14 28	1.7 1.5 0.9 0.5		90/3058
	WP		0.32	9	1 7 14 28	1.9 1.8 1.5 0.7		
Brazil, 1986 (Imperial)	WP	2.0 4.0		2 2	20 20	0.33 0.62	<0.01 0.01	86-0047
PASSION FRUIT								
Australia, 1991 (Barlow's E23)	WG		0.15	1	0 7 14 21	0.8 1.7 1.9 0.5		AUH-91-012
	WG		0.30	1	0 7 14 21	1.6 3.7 1.1 0.7		
PERSIMMON, JAPANESE								
Japan, 1989 (Hiratanenashi)	WP	7.5	0.19	6	20 30 45	0.54 0.43 0.11	0.02 0.02 0.01	Saku1P-7-186
Japan, 1989 (Fuyuu)	WP	7.5	0.19	6	21 30 45	0.40 0.22 0.15	0.06 0.05 0.02	Saku1P-7-186

[1] a: aerial application.

[2] pu: pulp; pe: peel; f: residues calculated on whole fruit basis from residues in pulp and peel and measured weights of peel and pulp.

Table 27. Mancozeb residues (as CS_2) in tropical fruits from supervised trials in the USA. Underlined residues are from treatments according to GAP.

CROP State, year (Variety)	Application				Day	Residues, mg/kg [1]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		Dithiocarbamates as CS_2	ETU	
BANANAS								
HI, 1988 (Valarie)	WG	3.6	0.64	8	0	0.20 pe 5.0 pu 0.21 c 0.20 c pe 1.5 c pu 0.12	<0.01 pe <0.01 pu <0.01	88-0029
HI, 1988 (Williams)	WG	3.6	0.64	11	0	0.48 pe 5.2 pu 0.55 c 0.23 c pe 3.3 c pu 0.27	<0.01 pe <0.01 pu <0.01	88-0030
Papayas								
FL, 1985 (Florida Type)	WP	2.2	0.14	10	0 7 14 21	2.5 0.98 0.49 0.40	<0.01 <0.01 0.01 0.02	85-0206
FL, 1985 (Florida Type)	WP	2.2	0.14	14	0 7 15 21	2.3 1.7 0.81 0.43	0.025 0.054 0.02 0.02	85-0594
HI, 1985 (Kapoho)	WP	3.4	0.36	12	7	3.1 c 0.12	0.074	85-0625
HI, 1985 (Kapoho)	WP	3.4	0.36	12	13	2.2 c 0.17	0.059	85-0632
HI, 1985 (Kapoho)	WP	3.4	0.36	12	21	1.1	0.031	85-0638
HI, 1988 (Kapoho Solo)	WP	2.2		13	0 0 0 0	6.6 pu 2.7 w 3.2 w pu 1.1	0.46 pu 0.17 w 0.47 w pu 0.16	88-0266

[1] pe: peel; pu: pulp; w: washed fruit; c: control sample.

Table 28. Mancozeb residues (as CS_2) in bulb vegetables from supervised trials in Australia, Brazil, Finland, France, Japan and The Netherlands. Underlined residues are from treatments according to GAP.

CROP Country, year (Variety)	Application	Day	Residues, mg/kg [1]	Ref.

	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
GARLIC								
Brazil, 1990	WP	1.6		4	0 7 14 21	<0.03 <0.03 <0.03 <0.03		070/90
Brazil, 1990	WP	3.2		4	0 7 14 21	<0.03 <0.03 <0.03 <0.03		070/9
France, 1990 (Blanc de Lomange)	WP	2.0		6	29	0.05 c 0.1		R77.32
France, 1989 (Blanc de Lomange)	WP	2.5	0.5	7	22	<0.05		R73.25
France, 1989	WP	1.5		2	34	<0.05		R75.6
France, 1989	WP	1.5		3	19	<0.05		R75.6
France, 1989	WP	2.0		7	21	<0.05		R75.6
France, 1989		1.5		2	34	<0.05		Malet, 1990
France, 1989		1.5		3	19	<0.05		Malet, 1990
France, 1989	WP	2.0		7	21	<0.05		Malet, 1990
Japan, 1990 (Kanchi-white)	WP	3.8	0.19	5	3 7 14	0.01 0.02 <0.005	<0.01 <0.01 <0.01	Hei.-3-1-27
Japan, 1990 (Fukuchi-white)	WP	3.8	0.19	5	3 7 14	<0.005 <0.005 <0.005	<0.01 <0.01 <0.01	Hei.-3-1-27
LEEK (including CHINESE LEEK)								
France, 1991 (Nebraska)	WP	8.0	1.6	4 7 9	68 30 8	<0.1 0.16 0.20		R79.63
France, 1990 (Nebraska)	WP	2.0	0.4	4 7	60 30	<0.05 0.08		R77.49
France, 1990	WP	2.0		3	51	<0.02		R75.8
France, 1990	WP	2.0	0.66	15 18	59 30	0.15 0.23		RF 0062-3
France, 1991 (Carentan)	WG	2.0	0.4	7 9 11	60 35 15	0.30 <0.1 0.21 c 0.21		R79.42
Japan, 1988 (Ichimonji-kuronobori)	WP	1.9	0.13	3	14 21 30	0.34 0.10 0.04	0.06 0.02 0.01	P-3-69
Japan, 1988 (Bohzu-shirazu)	WP	1.9	0.13	3	14 21 30	0.34 0.12 <0.01	0.02 <0.01 <0.01	P-3-69
Japan, 1990 (Jakko-natsu)	WP	1.9	0.13	3	14 21 30	0.17 0.03 0.01	0.01 <0.01 <0.01	?2?12?27?
Japan. 1990 (Kujoh)	WP	1.9	0.13	3	14 21 30	0.22 0.03 <0.01	<0.01 <0.01 <0.01	?2?12?27?
ONION								
Australia, 1991 (Golden Brown)	WG WG	2.3 4.5		8 8	0 3 7 14 21 0 3 7 14 21	0.8 2.0 1.7 0.7 0.9 3.0 2.1 1.3 2.5 1.0		AUK-91-009
Brazil, 1984 (Bala Pirie)	WP WP	1.6 3.2		6 6	7 7	0.06 0.05		84-0245
Canada, 1985 (Rocket)	WP	1.6	0.29	3	0 7 14	0.44 0.12 0.14		#74
Finland, 1979	WP	0.64		3	14 28	<0.1 <0.1		R67.4
Japan, 1981	WP	1.9	0.19	5	3	0.08	<0.01	58P-2-52

CROP Country, year (Variety)	Application				Day	Residues, mg/kg [1]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
(Sapporo-k_)					7 14 20	0.07 0.08 0.14 c 0.12	<0.01 <0.01 <0.01	
Japan, 1982 (Momiji)	WP	2.8	0.19	5	4 7 14 21	0.10 0.10 0.12 0.14 c 0.12	<0.01 <0.01 <0.01 <0.01	58P-2-52
Netherlands, 1984 (Jumbo)	WP SC SC	2.4 2.4 2.2	1.0 1.0 0.86	8 8 8	29 29 29	0.01 (2) 0.01 (2) 0.01, 1.6	0.002 (2) 0.13, 0.03 0.002, 0.004	PH8426
Netherlands, 1985 (Balstora)	WP SC SC	2.4 2.2 2.2	1.2 1.1 1.1	7 7 7	31 31 31	<0.01 (2) <0.01 (2) <0.01 (2)	0.005, 0.002 0.004, 0.005 0.005, <0.002	PH8523
Netherlands, 1985 (Jumbo)	WP SC SC	2.4 2.2 2.2	1.2 1.1 1.1	7 7 7	26 26 26	<0.01 (2) <0.01 (2) <0.01 (2)	0.002, <0.002 0.003, <0.002 0.004, <0.002	PH8524
Netherlands, 1986 (Balstora)	WP SC SC SC WP SC	2.4 2.2 2.2 1.5 2.4 2.2	1.2 1.1 1.1 0.73 1.2 1.1	7 7 7 7 7 7	42 42 42 42 42 42	<0.01 <0.01 <0.01 0.03 <0.01 <0.01	<0.002 <0.002 <0.002 <0.002 <0.002 <0.002	PH8623
Netherlands, 1990 (Marbella)	WG	2.4	0.80	5	9	0.07, 0.04	<0.002 (2)	PH9038
Netherlands, 1990 (Hysam)	WG	2.4	0.12-0.16	6	28	0.09, 0.14	<0.002 (2)	PH9041

[1] c: control sample.

Table 29. Mancozeb residues (as CS_2) in bulb onions from supervised trials in the USA. Underlined residues are from treatments according to GAP. All WP.

State, year (Variety)	Application [1]			Day	Residues, mg/kg [2]		Ref.
	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
FL, 1985 (429)	2.7		10	4 7 14	0.08 0.04 <0.03	<0.01 <0.01 <0.01	85-0130
TX, 1985 (Uno Grande)	2.2	2.4	9 ga	4 8 11 16	<0.03 <0.03 <0.03 <0.03	<0.01 <0.01 <0.01 <0.01	85-0176
CA, 1985 (Austbrn.100)	1.8		6 ga	3 7 14	0.14 0.10 0.06	<0.01 <0.01 <0.01	85-0274
CA, 1985 (Austbrn.100)	1.8		6 ga	c 3 7 14	c 0.04 0.08 0.04 <0.03	c <0.01 <0.01 <0.01 <0.01	85-0275
CA, 1985 (Austbrn.100)	1.8		6 ga	c 3 7 14	c 0.04 0.26 0.17 0.11	c <0.01 <0.01 <0.01 <0.01	85-0276
OH, 1985 (Spartan)	2.7	0.58	10	3 7 10 14	0.05 0.03 0.05 <0.03	0.02 <0.01 <0.01 <0.01	85-0403
OH, 1985 (Spartan Bann)	2.7	0.26	f 1	135	<0.03	<0.01	85-0404
MI, 1985 (Spartan Bann)	2.7	0.58	6	3 7 10 14	0.05 <0.03 <0.03 <0.03	0.01 <0.01 <0.01 <0.01	85-0504
MI, 1985 (Spartan Bann)	2.7	0.82	f 1	110	<0.03	<0.01	85-0512
NY, 1985 (Down.Y.Globe)	2.7	0.29	f 1	119	<0.03	<0.01	85-0652
NY, 1985 (Down.Y.Globe)	2.7	0.29	f 1	119	<0.03	<0.01	85-0653
CA, 1987 (BRB)	2.7	2.9	10	0 7 14	0.50 0.03 <0.03	0.19 0.02 <0.01	88-0041

[1] ga: ground and aerial application. f: furrow drench application at sowing.

[2] c: control sample.

Table 30. Mancozeb residues (as CS_2) in brassica vegetables from supervised trials in Brazil, Germany and Japan. Underlined residues are from treatments according to GAP. All WP.

CROP Country, year (Variety)				Day	Residues, mg/kg [1]		Ref.
	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
BROCCOLI							
Brazil, 1989 (Ramoso)	1.2		5	1 4 7 13	0.84 0.12 0.1 0.06		100/90
Brazil, 1989 (Ramoso)	2.5		5	1 4 7 13	1.1 0.73 0.39 0.28		100/90
CABBAGE							
Brazil, 1988 (Repolho Louco)	0.66		9	1 7 14	0.17 0.1 <0.03		101/90
Brazil, 1988 (Repolho Louco)	1.3		9	1 7 14	0.34 0.22 0.06		101/90
Japan, 1979 (Masuda-kohai-chusei-risoh)	2.8-3.8	0.19	3	21 30 45	0.08 0.06 0.06		55P-3-55
Japan, 1979 (Yahiko)	2.8-3.8	0.19	3	21 30 45	0.09 0.06 0.05		55P-3-55
CAULIFLOWER							
Brazil, 1989		0.16	7	0 7 14 21	0.22 0.17 0.11 0.06		730/89
Brazil, 1989		0.32	7	0 7 14 21	0.34 0.28 0.11 0.10		730/89
Germany, 1972	0.8	0.16	2	47	0.58		R67.27
Spain, 1992	3.8	0.19	1	0 3 14 21	0.43 0.18 0.04 0.06		MAPA 24.06.93
Spain, 1992	4.8	0.24	1	0 3 14 21	0.52 0.29 0.09 0.06		MAPA 24.06.93
CHINESE CABBAGE							
Japan, 1991 (Akogare)	1.9	0.13	3	14 21 30	0.34 0.34 0.06	0.02 <0.01 <0.01	Hei-4-3-11
Japan, 1991 (Ryokei)	1.9	0.13	3	14 21 30	0.25 0.05 0.01, <0.01 c 0.10	0.01 <0.01 <0.01	Hei-4-3-11
Spain, 1992 (Kasumi)	4.8	0.24	1	0 3 7 14 21	5.7 7.2 3.3 2.5 0.17		MAPA 25.06.93
KALE							
Germany, 1972	0.8	0.16	2	47	<0.3		R67.27

[1] c: control sample

Table 31. Mancozeb residues (as CS_2) in cucurbits from supervised trials in Australia, Brazil, France, Germany and Japan. Underlined residues are from treatments according to GAP. All WP.

CROP Country, year (Variety)		Day	Residues, mg/kg [2]	Ref.

	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
GHERKIN							
Germany, 1974 (Delikatess)	1.6	0.16	5	0 2 3 4 7	pe 0.60, pu <0.3 pe 0.56, pu <0.3 pe 0.96, pu <0.3 pe 0.57, pu <0.3 pe 0.45, pu <0.3		R69.13
MELON (Cantaloupe)							
France, 1989	1.6		3	3	<0.1		R75.6
France, 1989	0.91		4	3	<0.1		R75.6
France, 1989	1.4		4	3	<0.1		R75.6
France, 1989	1.5		3	3	<0.05		R75.6
France, 1990	1.6		3	3	0.11		R75.6
France, 1990	1.6		5	3	0.06		R75.6
France, 1990	1.5		5	3	<0.02		R75.6
MELON							
France, 1989	1.5		3	3	<0.05		Malet, 1990
France, 1989	1.6		3	3	<0.05		Malet, 1990
France, 1989	0.91		4	3	<0.1		Malet, 1990
France, 1989	1.4		4	3	<0.1		Malet, 1990
France, 1989	1.6		3	3	<0.1		Malet, 1990
France, 1990	1.5		5	3	<0.02		Malet, 1990
France, 1990	1.6		5	3	0.04		Malet, 1990
France, 1990	1.5		5	3	<0.02		Malet, 1990
France, 1990	1.6		5	3	0.08		Malet, 1990
France, 1990	1.6		3	3	0.11		Malet, 1990
Germany, 1972 (Diamex)	1.6	0.5	4	9	pu <0.3		R67.27
Japan, 1987	5.6	0.19	5i	1 3 7	pu 0.11 pu 0.16 pu 0.08	pu <0.01 pu <0.01 pu 0.01	Saku62P-9-238
Japan, 1987	3.8	0.19	5i	1 3 7	pu 0.24 pu 0.14 pu 0.28	pu 0.02 pu 0.04 pu 0.04	Saku62P-9-238
PUMPKIN							
Australia, 1992 (Jarrahdale)	1.8		5	0 7 14 21 28	<0.1 <0.1 <0.1 <0.1 <0.1		AUK-92-004
	3.5		5	0 7 14 21 28	<0.1 <0.1 <0.1 <0.1 <0.1		
Brazil, 1990	1.6		2	0 14 21 28	0.22 0.11 0.06 0.04		102/90
Brazil, 1990	3.2		2	0 14 21 28	0.56 0.22 0.17 0.11		102/90
SQUASH							
France, 1990	1.5		4	6	<0.02		R75.6
France, 1990	1.5		5	3	<0.02		R75.6
France, 1990	1.6		5	3	0.05		R75.6
France, 1990	1.6		4	6	<0.02		R75.6
France, 1990	2.0		2	2 6	<0.002 <0.002		R75.6
France, 1990	2.0		1	3 10	<0.002 <0.002		R75.6
Japan, 1989	1.9-2.5	0.13	3	21 30	0.17 0.06	0.02 0.02	Hei-1-10-27

CROP Country, year (Variety)				Day	Residues, mg/kg [2]		Ref.
	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
(Miyako)				45	0.03	0.02	
Japan, 1989 (Ebisu)	1.9	0.13	3	21 30 45	0.03 0.01 0.02	<0.01 <0.01 0.01	Hei-1-10-27
SUMMER SQUASH							
Australia, 1992 (Black Regal)	1.8 3.5		5 5	0 6 13 20 27 0 6 13 20 27	<0.1 <0.1 <0.1 <0.1 <0.1 0.2 0.3 <0.1 <0.1 <0.1		AUK-92-006
France, 1990	1.6		5	3	0.05		Malet, 1990
France, 1990	1.5		4	6	<0.02		Malet, 1990
France, 1990	1.6		4	6	<0.02		Malet, 1990
France, 1990	1.5		5	3	<0.02		Malet, 1990
WATERMELON							
Australia, 1992 (War Paint)	1.8 3.5		5 5	0 7 14 21 28 0 7 14 21 28	<0.1 <0.1 <0.1 <0.1 <0.1 <0.1 <0.1 <0.1 <0.1 <0.1		AUK-92-005
Japan, 1984 (Fujik_)	3.6	0.19	7i	7 14	pu 0.017 pu <0.006	pu <0.01 pu <0.01	Saku59P-8-212
Japan, 1984 (Akadoma)	3.6	0.19	7i	1 7 14	pu 0.011 pu <0.006 pu <0.006	pu 0.02 pu 0.01 pu 0.01	Saku59P-8-212

[1] i: indoors.
[2] pe: peel; pu: pulp.

Table 32. Mancozeb residues (as CS_2) in cucumbers from supervised trials. Underlined residues are from treatments according to GAP. All WP.

Country, year (Variety)				Day	Residues, mg/kg		Ref.
	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
Australia, 1992 (Marketer)	1.8 3.5		4 4	0 7 14 21 28 0 7 14 21 28	0.1 <0.1 <0.1 <0.1 <0.1 0.1 0.1 <0.1 <0.1 <0.1		AUK-92-003
Brazil, 1988 (Capira)		0.15	3	7 14 21	<0.03 <0.03 <0.03		FPA-88-023
Brazil, 1988 (Capira)		0.30	3	7 14 21	<0.03 <0.03 <0.03		FPA-88-023
France, 1988	1.4		6	7	<0.01		R75.6
France, 1988	1.4		3	3	<0.1		R75.6
France, 1988	0.91		3	3	<0.1		R75.6
France, 1988	1.4		6	7	<0.01		Malet, 1990
France, 1989	1.6		3	3	<0.1		R75.6
France, 1989	1.6		5	3	<0.1		R75.6

Country, year (Variety)				Day	Residues, mg/kg		Ref.
	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
France, 1989	0.91		3	3	<0.1		R75.6
France, 1989	0.91		3	3	<0.1		Malet, 1990
France, 1989	1.4		3	3	<0.1		Malet, 1990
France, 1989	1.6		5	3	<0.1		Malet, 1990
France, 1989	1.4		3	3	<0.1		Malet, 1990
France, 1989	0.91		3	3	<0.1		Malet, 1990
France, 1989	1.6		3	3	<0.1		Malet, 1990
France, 1990	1.6		3	2	0.03		R75.6
France, 1990	1.6		3	2	0.03		Malet, 1990
Germany, 1974 (Pepiner)	3.2	0.32	5i	0 2 3 4 7	1.4 0.84 <0.3 0.64 <0.3		R67.8
Germany, 1974 (Femdom)	3.2	0.32	5i	0 2 3 4 7	0.50 0.60 0.65 0.40 <0.3		R67.8
Japan, 1983 (Hokkoyku-2 goh)	3.8	0.19	3	1 3 7	0.18 0.25 0.05	0.01 0.01 0.01	59P-1-32
Japan, 1983 (Hokkoyku-2 goh)	2.5	0.13	3i	1 3 7	0.12 0.19 0.07	<0.01 <0.01 0.01	59P-1-32
Japan, 1983 (Kash_-fushinari 2 goh kairy_)	3.8	0.19	3i	1 3 7	0.19 0.12 0.02	<0.01 <0.01 <0.01	59P-1-32
Spain, 1989	2.4		1	0 2 7	0.61 0.15 0.07		MAPA 24.06.93

[1] i: indoors.

Table 33. Mancozeb residues (as CS_2) in cucurbits from supervised trials in the USA. Underlined residues are from treatments according to GAP. All WP.

CROP State, year (Variety)				Day	Residues, mg/kg [1]		Ref.
	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
CANTALOUPE							
FL, 1985 (Tania)	2.7	0.29	12	3 5 10	2.4 1.8 1.1	0.029 0.051 0.058	85-0161
CA, 1985 (Top Mark)	2.7	0.82	5	3 5 10	0.76 0.43 0.16	0.01 <0.01 <0.01	85-0280
CUCUMBER							
FL, 1985 (Slicer)	2.7	0.29	8	4 5 10	0.14 0.09 <0.03	0.01 <0.01 <0.01	85-0126
FL, 1985 (Model)	2.7	0.29	12	3 5 10	1.1 0.65 0.25	0.024 0.02 <0.01	85-0163
OH, 1985 (Market More)	2.7	0.58	8	3 5 7 10	0.25 0.19 0.13 0.10	0.01 <0.01 <0.01 <0.01	85-0325
OH, 1985 (Carolina)	2.7	0.48	6	3 5 7 10	0.20 0.12 0.07 0.06	0.01 <0.01 <0.01 <0.01	85-0339
GA, 1986 (Poinsett)	1.8	0.58	7	0 10	0.54 0.28	0.035 0.02	86-0560
GA, 1986 (Poinsett)	1.8	0.58	7	0 10	0.83 0.27 c 0.62	0.02 0.011	86-0645

CROP State, year (Variety)				Day	Residues, mg/kg [1]		Ref.
	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
TX, 1987 (P76)	2.7	1.4	9	5	0.05	0.043	87-0482
CA, 1987	2.7	1.4	3	5	0.06	0.041	88-0045
SUMMER SQUASH							
FL, 1985 (Prolific St)	2.7	0.29	8	4 5 10	0.40 0.23 0.03	0.01 <0.01 <0.01	85-0127
FL, 1985 (Senator)	2.7	0.29	8	4 5 10	0.32 0.25 0.14	<0.01 <0.01 <0.01	85-0128
VA, 1985 (Crookneck)	2.8	0.60	7	3 5 10	0.16 0.08 0.04	0.02 0.01 <0.01	85-0310
VA, 1985 (Senator)	2.8	0.60	7	3 5 10	0.10 0.07 0.05	<0.01 <0.01 <0.01	85-0311
OH, 1985	2.7	0.58	7	3 5 10	0.12 0.10 0.05	<0.01 <0.01 <0.01	85-0312
NJ, 1985 (Black Beauty)	2.7	0.32	5	2 4 10	1.0 0.83 0.65	0.02 0.02 0.01	85-0428
IN, 1985 (Yellow St Nk)	2.7	0.29	7	3 5 10	0.28 0.21 0.17	<0.01 <0.01 <0.01	85-0484
WATERMELON							
FL, 1985 (Sugar Baby)	2.7	0.29	12	3 5 10	0.81 0.38 0.20	<0.01 <0.01 <0.01	85-0162
WINTER SQUASH							
FL, 1985 (Tatabutu)	2.7	0.29	8	4 5 10	0.27 0.20 0.05	<0.01 <0.01 <0.01	85-0129
OH, 1985 (Acorn)	2.7	0.58	7	3 5 10	0.13 0.08 0.05	0.02 0.02 0.028	85-0460
VA, 1985 (Waltham)	2.8	0.60	7	3 5 10	0.26 0.10 0.08	0.025 0.035 0.025	85-0479
VA, 1985 (Tay Belle)	2.8	0.60	7	3 5 10	0.56 0.38 0.18 c 0.24	0.038 0.033 0.031	85-0480
OH, 1985 (Acorn)	2.7	0.29	7	3 5 10	0.57 0.38 0.18 c 0.36	0.030 0.027 0.02	85-0485

[1] c: control sample.

Table 34. Mancozeb residues (as CS_2) in fruiting vegetables other than cucurbits from supervised trials in Brazil, France, Germany, Italy, Japan, The Netherlands, Portugal and Spain. Underlined residues are from treatments according to GAP.

CROP Country, year (Variety)	Application [1]	Day	Residues, mg/kg [2]	Ref.

	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
EGG PLANT								
Brazil, 1984 (Pira F 100)	WP	2.4 4.8		8 8	7 7	0.26 0.28 c 0.02		84-0105
PEPPERS								
Brazil, 1989	WP		0.24	4	0 7 14 21	2.5 0.56 0.08 <0.02		745/89
Brazil, 1989	WP		0.48	4	0 7 14 21	5.3 0.90 0.78 0.03		745/89
Spain, 1986	WP	2.9	0.16	3	0 4 7 14 21	1.8 1.6 1.0 0.6 0.3		R66.22/23
Spain, 1987 (Cristal)	WP	3.4	0.16	1	0 3 7 14 21	2.2 1.8 1.1 0.49 0.17		MAPA 24.06.93
Spain, 1988 (Magister)	WP	3.8	0.16	1	0 3 7	0.34 0.30 0.19		MAPA 24.06.93
TOMATOES								
Brazil, 1988 (Rio Grande)	WG	2.4		15	1 3 7 14	0.12 0.06 0.03 0.07		Du Pont FPA 88-027 A
Brazil, 1988 (Rio Grande)	WG	4.8		15	1 3 7 14	0.20 0.21 0.08 0.03		Du Pont FPA 88-027 A
France, 1990 (Merveille des Marchés)	WP	1.6	0.32	9	0 2 4 7 10	2.2 0.84 0.72 0.77 0.85 c 0.20		R77.35
France, 1990 (Merveille des Marchés)	WP	0.33	0.06	9	0 2 4 7 10	1 0.7 0.28 0.64 2.9		R77.36
France, 1990 (Merveille des Marchés)	WP	1.2	0.23	9	0 2 4 7 10	1.9 0.81 0.3 1 0.36		R77.37
France, 1990 (Merveille des Marchés)	WP	1.2	0.23	8	0 2 4 7	w 0.1 w 0.11 w 0.25 w 0.25		R77.40
France, 1990 (Merveille des Marchés)	WP	0.33	0.06	8	0 2 4 7	w 0.11 w <0.05 w 0.07 w 0.15		R77.39
France, 1990 (Merveille des Marchés)	WP	1.6	0.32	8	0 2 4 7	w 0.4 w <0.05 w 0.67 w 0.28 c 0.08		R77.38
France, 1990 (Merveille des Marchés)	WP	0.33	0.06	8	0 2 4 7	1.3 0.61 0.43 0.6		R77.42
France, 1990 (Merveille des Marchés)	WP	1.6	0.32	8	0 2 4 7	4.1 1.4 1.1 0.55 c 0.05		R77.41
France, 1990 (Merveille des Marchés)	WP	1.2	0.23	8	0 2 4 7	3.2 1.9 1.3 0.54		R77.43
France, 1990 (Merveille des	WP	1.2	0.23	9	0 2	w 0.4 w 0.11		R77.46

CROP Country, year (Variety)	Application [1]				Day	Residues, mg/kg [2]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
Marchés)					4 7 10	w 0.16 w 0.22 w 0.13		
France, 1990 (Merveille des Marchés)	WP	0.33	0.06	9	0 2 4 7 10	w 0.34 w <0.05 w 0.16 w 0.06 w 0.13		R77.45
France, 1990 (Merveille des Marchés)	WP	1.6	0.32	9	0 2 4 7 10	w 0.11 w 0.31 w 0.26 w 0.37 w 0.4 c 0.13		R77.44
France, 1990 (Ferline)	WP	1.6	0.31	6	3	1.4 c 0.06		R78.53
France, 1990	WP	1.6	0.31	6	3	1.5 c 0.15		R80.8
France, 1991	WG	1.6	0.32	8	0 3 4 7	2.6 1.3 0.94 1.6		R79.43
France, 1991 (Roma)	WG	1.6	0.32	8	7 7 14 14	0.81 w 0.12 0.57 w <0.1		R79.44
Germany, 1974 (Namaza Lizzy)	WP	1.6	0.16	6	0 2 3 4 7	4.3 4.2 1.8 3.8 2.1		R67.28
Germany, 1974 (Rot-käppchen)	WP	3.2	0.16	8	0 2 3 4 7	0.82 0.85 0.52 <0.3 <0.3		R69.14
Germany, 1974 (Rubin)	WP	3.2	0.16	8	0 2 3 4 7	0.95 0.68 0.60 <0.3 <0.3		R69.14
Germany, 1974 (Rheinlands-Ruhm)	WP	0.96	0.16	6	2 3 4 7	1.2 1.3 1.7 1.3		R69.14
Germany, 1975 (MM Nota)	WP	2.4	0.5	8	0 2 3 4 7	1.3 0.84 1.0 0.91 0.63	0.015 0.005 0.005 0.003	R69.14
Germany, 1975 (1080)	WP	0.96	0.16	6	0 2 3 4 7	0.33 <0.2 <0.2 <0.2 <0.2	<0.003 0.003 0.003 0.003 0.003	R69.14
Germany, 1975 (Rot-käppchen)	WP	3.2	0.16	8	0 2 3 4	0.65 0.92 <0.3 <0.3		R69.14
Germany, 1975 (Rheinlands-Ruhm)	WP	1.6	0.16	8 i	0 1 3 4 7 9	1.5 0.95 3.7 1.7 0.89 0.79	0.008 0.008 0.015 0.007 0.007 <0.007	R69.14
Italy, 1986 (HY23)	WP	2.2	0.3	7 6 4	21 28 42	<0.1 <0.1 <0.1	<0.02 <0.02 <0.02	R65.43
Italy, 1986 (HY23)	WP	4.3	0.6	7 6 4	21 28 42	<0.1 <0.1 <0.1	<0.02 <0.02 <0.02	R65.43
Italy, 1986 (OC1023)	WP	2.8	0.3	7 6 4	21 28 42	<0.1 <0.1 <0.1	<0.02 <0.02 <0.02	R65.43
Italy, 1986 (OC102)	WP	4.3	0.6	7 6 4	21 28 42	0.1 <0.1 <0.1	<0.02 <0.02 <0.02	R65.43

CROP Country, year (Variety)	Application [1]				Day	Residues, mg/kg [2]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
Italy, 1986 (UC105)	WP	2.1	0.3	7 6 4	21 28 42	<0.1 <0.1 <0.1	<0.02 <0.02 <0.02	R65.43
Italy, 1986 (OC1053)	WP	4.1	0.6	7 6 4	21 28 42	0.1 <0.1 <0.1	<0.02 <0.02 <0.02	R65.43
Italy, 1987 (Rio Grande)	WP	1.4	0.24	8 7 5	21 28 42	<0.1 <0.1 <0.1	<0.02 <0.02 <0.02	R66.19
Italy, 1987 (Rio Grande)	WP	2.9	0.48	8 7 5	21 28 42	0.29 0.12 <0.1	<0.02 <0.02 <0.02	R66.19
Italy, 1987 (Improy)	WP	1.4	0.24	8 7 5	21 28 42	0.21 <0.1 <0.1	<0.02 <0.02 <0.02	R66.20
Italy, 1987 (Improy)	WP	2.9	0.48	8 7 5	21 28 42	0.1 <0.1 <0.1	<0.02 <0.02 <0.02	R66.20
Japan, 1985 (Zuiken)	WP	1.9	0.094	5i	1 3 7	0.19 0.17 0.27	0.01 0.02 <0.01	60P-5-57
Japan, 1985 (Kyoryoku- kairyo-shuko)	WP	1.9	0.094	5i	1 3 7	0.30 0.31 0.33	0.02 0.02 <0.01	60P-5-57
Netherlands, 1984 (Abunda)	WP SC SC		0.16 0.15 0.14	6 6 6	15 15 15	0.07, <0.01 <0.01, 0.04 <0.01, 0.20	<0.002, 0.002 <0.002 (2) <0.002 (2)	PH8405
Netherlands, 1984 (Abunda)	WP SC SC WP SC SC		0.16 0.15 0.14 0.16 0.15 0.14	3 3 3 8 8 8	15 15 15 4 4 4	0.02, <0.01 <0.01 (2) <0.01 (2) 0.16, 0.01 <0.01, 0.02 0.20, 0.04	<0.002, 0.002 <0.002 (2) 0.01, <0.002 0.046, 0.002 <0.002	PH8406
Portugal, 1990 (Petopride)	WP	1.6	0.45	4 2 2	54, 55 83 84	0.49, w 0.57 0.30, w 0.20 0.13		R79.53
Portugal, 1990 (Rio Fuego)	WP	1.6 m 1.6 1.6	0.45 0.45 0.45	4 2 2	82 82 104	0.46, w 0.32 0.16, w 0.29 0.57, w 0.23		R79.54
Spain, 1986 (Rubí)	WP	3.8	0.19	2	0 3 7 14 21	1.1 0.72 0.52 0.43 0.34		MAPA 24.06.93
Spain, 1986 (Rubí)	WP	3.2	0.16	2	0 3 7 14 21	1.1 0.72 0.46 0.26 0.18		MAPA 24.06.93
Spain, 1987 (Quarenteno)	WP	4.8	0.16	1	0 3 7 14 21	2.2 1.9 0.37 0.31 0.07		MAPA 24.06.93
Spain, 1988	WP	0.24		1	0 2 7 10 15	1.0 0.58 0.57 0.37 0.20		MAPA 24.06.93
Spain, 1990 (Rio Fuego)	WG	1.4	0.22	3	24	0.34		R79.20
Spain, 1990 (Centurion)	WP	0.44 0.4+1.44 0.4+1.44 0.4+1.44 0.4+1.44	0.04	1 1+6 1+5 1+4 1+2	98 9 21 38 65	0.16 0.68 0.3 0.24 0.27		R79.19
Spain, 1990 (Ovad Red)	WP	0.64+0.8 0.64+0.80. 64+0.8 0.64+0.80. 64+0.8	0.13+0.16 0.13+0.16 0.13+0.16 0.13+0.16 0.13+0.16	1+1 1+2 1+3 1+4 1+5	91 73 51 37 1	<0.05 <0.05 <0.05 0.06 0.08		R80.10
Spain, 1990 (Quarenteno)	WP	4.8	0.19	1	0 3 7 14	0.61 0.53 0.19 0.22		MAPA 24.06.93

CROP Country, year (Variety)	Application [1]				Day	Residues, mg/kg [2]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
					21	0.15		
Spain, 1991 (Cristina)	WP	0.2	0.02	1	0 3 7 15 c	2.1 1.4 0.94 0.45 0.23		R80.30
	WP	0.2	0.02	2	0 3 8 15	1.9 1.9 0.84 0.78		

[1] i: indoors; m: metiram also early in spray programme.
[2] w: washed fruit; c: control sample.

Table 35. Mancozeb residues (as CS_2) in fruiting vegetables from supervised trials in the USA. Underlined residues are from treatments according to GAP.

CROP State, year (Variety)	Application [1]				Day	Residues, mg/kg [2]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
SWEET CORN								
PA, 1987 (Penn Fresh)	WP	1.3	0.29	7	7	e 0.21 c&k <0.03 h 1.3	e <0.01 c&k <0.01 h 0.01	34-89-04
	WP	6.7	1.4	7	7	e 0.90 c&k 0.03 h 6.7	e 0.02 c&k 0.02 h 0.18	
OR, 1987 (Gold Jubilee)	WP	1.3	0.36	15	7 7 7	c&k <0.03 e&h 1.3 h 2.9	c&k <0.01 e&h 0.01 h 0.02	87-0384
TOMATOES								
CA, 1971	WP	2.7		6	0 3 7 14	8.7 2.8 4.1 2.5		3-71-51
CA, 1971	WP	5.4		6	0 3 7 14	4.7 3.5 1.8 1.8		3-71-52
DL, 1971 (C-28)	WP	2.7		8	0 3 7 14	0.92 0.72 0.61 0.44	0.05 0.04 0.03 0.03	3-71-61
OH, 1971 (C-28)	WP	2.7		10	0 3 7 14	0.69 0.68 0.53 0.34	0.04 0.04 0.02 0.02	3-71-59
FL, 1972 (Homestead 24)	WP	1.3		13 14	0 0 3 7 14	0.56 0.50 0.47 0.41 0.21	0.02 0.02 0.02 0.03 0.02	3-72-01
FL, 1972 (Homestead 24)	WP	1.3	0.19	13 14	0 0 3 7 14	0.35 0.39 0.30 0.34 0.19	0.03 0.05 0.02 0.01 <0.01	23-72-7
CA, 1985 (785)	WP	2.7	0.58	8	2 5 12	0.59 0.42 0.12	0.01 <0.01 0.03	85-0555
CA, 1985	WP	2.7	0.58	7	3	1.8	0.01	85-0368
CA, 1985	WP	2.7	0.58	6	2 5	1.3 0.81	0.01 <0.01	85-0369
CA, 1985	WP	2.7	0.58	8	2 5	5.1 3.0	0.046 0.031	85-0554
CA, 1985 (785)	WP	2.7	0.58	8	2 5	0.59 0.42	0.01 <0.01	85-0555
CA, 1985 (C16)	WP	2.7	1.4	a 6	2 5 9	0.45 0.17 0.09	<0.01 0.01 0.01	85-0346

CROP State, year (Variety)	Application [1]				Day	Residues, mg/kg [2]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
CA, 1985 (C16)	WP	2.7	1.4	a 6	2 5	0.44 0.14	<0.01 <0.01	85-0347
CA, 1985	WP	2.7	1.4	a 6	2 5 9	0.32 0.18 0.06	0.02 0.02 <0.01	85-0348
CA, 1985 (C16)	WP	2.7	1.4	a 6	2 5 9	0.47 0.23 0.06	0.02 0.01 0.02	85-0349
NJ, 1986 (US28)	WP WG	2.7 2.7	0.34 0.34	7 7	8 8	0.84 0.84 c 0.3	0.054 0.054	86-0596
CA, 1987 (Harris 3075)	WP	2.7	1.4	4	0 5 10	4.6 2.7 1.8	0.10 0.047 0.047	88-0058

[1] a: aerial application.
[2] c&k: cob and kernel; e&h: ear and husk; h: husk; c: control sample.

Table 36. Mancozeb residues (as CS_2) in leafy vegetables from supervised trials in Brazil, Canada and Spain. Underlined residues are from treatments according to GAP. All WP.

CROP Country, year (Variety)	Application			Day	Residues, mg/kg, EBDC as CS_2	Ref.
	kg ai/ha	kg ai/hl	No.			
ENDIVE						
Canada, 1981 (Green Curled)	1.6	0.29	3	0 1 3 7 10 14	22 19 14 7.2 4.4 0.84	#71
KALE						
Brazil, 1989		0.16	4	0 7 14 21	11 1.8 0.95 0.03	731/89
Brazil, 1989		0.32	4	0 7 14 21	13 4.6 1.0 0.13	731/89
LETTUCE						
Canada, 1981 (leaf lettuce, Grand Rapids)	1.6	0.29	3	0 1 3 7 10 14	15 16 13 4.0 2.0 0.47	#71
Canada, 1981 (cos lettuce, Paris Island Cos)	1.6	0.29	3	0 1 3 7 10 14	14 11 7.2 1.4 1.4 0.15	#71
Canada, 1983 (Ithaca)	1.6	0.29	3	0 1 3 10	4.2 3.3 1.9 0.31	#72

CROP Country, year (Variety)	Application			Day	Residues, mg/kg, EBDC as CS_2	Ref.
	kg ai/ha	kg ai/hl	No.			
Canada, 1984 (Ithaca)	1.6	0.29	3	0 1 3 7 10 14	2.9 3.7 1.9 0.99 0.66 0.15	#73
Spain, 1985 (Batavia)	1.9	0.16	3	0 3 7 14 21	8.8 6.3 4.7 3.0 1.4	R66.22/23
Spain, 1985 (Inverne)	3.0	0.16	3	0 3 7 14 21	11 9.7 6.8 3.5 1.9	R66.22/23
Spain, 1985 (Batavia)	2.7	0.16	3	0 3 7 15 22	11 9.2 5.8 3.0 2.6	R66.22/23
Spain, 1986 (Verdia)	4.8	0.24	2	0 3 7 14 21	27 22 15 6.0 3.7	R66.23
Spain, 1987 (Romana)	4.5	0.16	1	0 3 7 14 21 28	17 14 11 10 6.1 2.5	MAPA 25.06.93
Spain, 1989 (Samy)	3.3	0.19	1	0 7 22	5.6 3.9 0.79	MAPA 25.06.93

Table 37. Mancozeb residues (as CS_2) in legume vegetables from supervised trials in Australia, Brazil, France, Japan. The Netherlands and Spain. Underlined residues are from treatments according to GAP.

CROP Country, year (Variety)	Application	Day	Residues, mg/kg [1]	Ref.

	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
AZUKI BEANS (RED BEANS)								
Japan, ? (Takara-azuki)	WP	1.9	0.19	3	14 21 30	s 0.04 s 0.02 s 0.02		Saku54P-10-110
Japan, ? (Ketobuki-azuki)	WP	1.9	0.19	3	14 21 30	s 0.02 s 0.01 s 0.01		Saku54P-10-110
BEANS								
Australia, 1988 (Fiord)	WP	2.0 4.0		4 4	64 64	<0.3 <0.3	0.2 0.2	3137/88/5
Brazil, 1986 (Carioquinha)	WP	1.6 3.2		2 2	14 14	db <0.03 db <0.03	db <0.01 db <0.01	AR 34A-89-24
France, 1973	WP	4.0		1	88	db <0.3		R67.12
France, 1973	WP	2.4		6	26	db <0.3		R67.12
France, 1973	WP	2.4		3	67	db <0.3		R67.12
France, 1990 (Mange tout)	WG	1.6	0.4	1	3 7 10	3.0 2.3 1.6		R73.11
Netherlands, 1989 (Victor)	WP SC WG WP	3.2 3.2 3.2 3.2	0.53 0.53 0.53 0.53	5 5 5 5	45 45 45 45	0.40, 0.43 1.1, 0.38 0.36, 0.47 0.53, 0.48	0.048, <0.01 0.039, 0.023 0.028, 0.047 0.034, 0.036	PH8969
Netherlands, 1989 (Victor)	WP SC WG WP	3.2 3.2 3.2 3.2	0.53 0.53 0.53 0.53	4 4 4 4	15 15 15 15	0.51, 1.1 1.7, 2.8 1.5, 2.7 1.0, 1.9	0.051, 0.036 0.057, <0.01 0.055, 0.11 0.061, <0.01	PH8970
Netherlands, 1990 (Victor)	WG SC	3.5 3.2	0.60 0.53	5 5	23 23	0.11, 0.16 0.10, 0.32	0.013, 0.007 0.005 (2)	PH9031
Netherlands, 1990 (Alfred)	WG SC	3.5 3.5	0.60 0.60	5 5	61 61	0.05, 0.08 0.08, 0.11	0.009, 0.008 0.005 (2)	PH9032
Spain, 1992 (Eagle)	WP	2.2	0.24	1	0 3 7	0.16 0.11 0.06		MAPA 25.06.93
FRENCH BEANS								
Brazil, 1990 (Manteiga)	WP	1.6		5	1 3 6 15 22	0.56 <0.03 <0.03 <0.03 <0.03		FPA-89-032 Du Pont
Brazil, 1990 (Manteiga)	WP	3.2		5	1 3 6 15 22	0.84 <0.03 <0.03 <0.03 <0.03		FPA-89-032 Du Pont
KIDNEY BEANS								
Japan, 1990 (Honkintoki)	WP	1.3	0.13	4	20 30 45	db 0.01 db <0.004 db <0.004	db 0.04 db 0.04 db 0.01	Hei.2-12-7
Japan, 1990 (Shin-edogawa)	WP	1.3	0.13	4	21 30 45	db 0.02 db <0.004 db <0.004	db <0.01 db 0.01 db 0.01	Hei.2-12-7
PEAS								

CROP Country, year (Variety)	Application				Day	Residues, mg/kg [1]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
Brazil, 1988 (Mikado)	WG		0.15	6	1 7 14	0.11 0.03 0.08		FPA 88-020 Du Pont
Brazil, 1988 (Mikado)	WG		0.30	6	1 7 14	0.53 0.06 0.21		FPA 88-020 Du Pont
France, 1990 (Belinda)	WP	1.8	0.45	2	36 36	g 0.17 p 0.47		R80.6
France, 1991 (Ascona)	SC	2.0	0.66	2 3	41 41	g 0.1 g <0.1		R79.32
France, 1991 (Ascona)	SC	2.0	0.66	2 3	42 42	g <0.1 g <0.1		R79.31

[1] db: dry beans; g: grain or seeds; p: pods; s: immature seeds.

Table 38. Mancozeb residues (as CS_2) in root and tuber vegetables from supervised trials in Australia, Brazil, Finland, France, Germany, Italy, Japan, The Netherlands and the UK. Underlined residues are from treatments according to GAP.

CROP Country, year (Variety)	Application [1]				Day	Residues, mg/kg [2]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
BEET								
Brazil, 1989	WP		0.16	4	0 7 14 21	0.12 0.11 0.10 0.08		715/89
Brazil, 1989	WP		0.32	4	0 7 14 21	0.16 0.15 0.11 0.10		715/89
CARROT								
Australia, 1991 (Majestic Red)	WG	1.7		3	0 3 7 14 21	0.3 0.05 <0.05 <0.05 <0.05		AUK-91-008
	WG	3.3		3	0 3 7 14 21	0.55 0.25 0.45 0.05 <0.05		
Brazil, 1990	WP	0.32	0.16	4	0 7 14 21	2.5 0.78 0.67 0.36		288/90
Brazil, 1990	WP	0.64	0.32	4	0 7 14 21	3.3 2.1 0.78 0.42		288/90
France, 1989	WG	1.6	0.5	8	14 26	<0.05 0.05		R72.10
France, 1990 (Tantale)	WP	1.5	0.3	7	15 30	0.11, c 0.29 0.19, c 0.35		R77.33
France, 1990 (Tantale)	WP	1.6	0.32	7	15 30	0.19, c 0.29 0.13, c 0.35		R77.34
France, 1990 (Touchon)	WP	1.6	0.53	14-15	19 30	0.09 0.19		R77.50
France, 1991 (Rouge		1.6	0.32	11-13	15	<0.1		R79.41

CROP Country, year (Variety)	Application [1]				Day	Residues, mg/kg [2]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
Touchon)					30	<0.1		
France, 1991 (Lindoro)	WP	1.6	0.32	7	15 30	<0.1 <0.1		R79.61
France, 1991 (Lindoro)	WP	1.5	0.3	7	15 30	<0.1 <0.1		R79.62
Germany, 1972	WP	0.8	0.16	1	56	<0.3		R67.27
LOTUS (EAST INDIAN)								
Japan, 1989 (Bicch_)	WP	1.1	3.8	3	1 3 7 14	<0.02 <0.02 <0.02 <0.02	<0.01 <0.01 <0.01 <0.01	Hei.-1-10-11
Japan, 1989 (Bicch_)	WP	1.1	3.8	3	1 3 7 14	<0.02 <0.02 <0.02 <0.02	<0.01 <0.01 <0.01 <0.01	Hei.-1-10-11
POTATO								
Australia, 1990 (Norchip)	WG	1.7		8	0 6 14 21	<0.05 <0.05 <0.05 <0.05	<0.05 <0.05 <0.05 <0.05	AUE-90-02
Australia, 1990 (Norchip)	WG	3.3		8	0 6 14 21	<0.05 <0.05 <0.05 <0.05	<0.05 <0.05 <0.05 <0.05	AUE-90-02
Australia, 1990 (Norchip)	WP	1.8		8	0 6 14 21	<0.05 <0.05 <0.05 <0.05		AUE-90-02
Australia, 1990 (Norchip)	WP	3.7		8	0 6 14 21	<0.05 <0.05 <0.05 <0.05		AUE-90-02
Brazil, 1988 (Radosa)	WG	2.25		4	1 7 14	<0.03 <0.03 <0.03		Du Pont FPA 88-024
Brazil, 1988 (Radosa)	WG	4.5		4	1 7 14	<0.03 <0.03 <0.03		Du Pont FPA 88-024
Finland, 1985 (Bintje)	WP	1.6		4	21		<0.009	R.65.2
France, 1990 (Bintje)	WG	1.6 1.3	0.4 0.32	8 4	25 27	0.05 <0.05		R79.27/28
France, 1990 (Bintje)	WG	1.6 1.3	0.4 0.32	8 4	25 27	0.06 <0.05 c 0.12		R79.27
France, 1990 (Bintje)	WP	1.6	0.4	6	10	<0.05 c 0.07		R78.4
France, 1990 (Bintje)	WP	1.6	0.29	10 20	23 19	0.09 0.34		R78.5
France, 1990 (Bintje)	WP	st+1.6	0.4	7	54	<0.05		R78.6
France, 1990 (Bintje)	WP	1.6	0.32	13 1	58 58	<0.05 0.32		R78.10
France, 1990 (Bintje)	WP	1.6	0.32	9	35	0.06 c 0.07		R78.22
France, 1990 (Bintje)	WP	1.6	0.32	14	36	<0.05		R78.23
France, 1990 (Bintje)	WP	1.6 st+1.6		8 8	107 107	<0.05 0.05		R78.25
France, 1991 (Kaptah)	WP	4.8	1.6	10	17	0.15		R78.60
France, 1991 (Stella)	WP	4.8	1.6	7	10	<0.1 c 0.1		R78.61
France, 1991 (Kaptah Vandel)	WP	6.4	1.6	14	13	0.16 c 0.16		R79.55
France, 1991 (Bintje)	WP	1.6 1.6	0.25 0.25	7 9	46 32	<0.1 <0.1 c <0.1 (2), c 0.1		R79.58
Italy, 1986 (Spunta)	WP	2.3	0.24	8 7	21 28	<0.1 <0.1	<0.02 <0.02	R65.39

CROP Country, year (Variety)	Application [1]				Day	Residues, mg/kg [2]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
				5	41	<0.1	<0.02	
Italy, 1986 (Spunta)	WP	4.6	0.48	8 7 5	21 28 41	<0.1 <0.1 <0.1	<0.02 <0.02 <0.02	R65.39
Italy, 1986 (Arsy)	WP	2.4	0.24	8 7 5	21 28 41	<0.1 <0.1 <0.1	<0.02 <0.02 <0.02	R65.39
Italy, 1986 (Arsy)	WP	4.8	0.48	8 7 5	21 28 41	<0.1 <0.1 <0.1	<0.02 <0.02 <0.02	R65.39
Italy, 1986 (Primura)	WP	2.6	0.24	8 7 5	21 28 41	<0.1 <0.1 <0.1	<0.02 <0.02 <0.02	R65.39
Italy, 1986 (Primura)	WP	5.1	0.48	8 7 5	21 28 41	<0.1 <0.1 <0.1	<0.02 <0.02 <0.02	R65.39
Italy, 1987 (Favorita)	WP	1.8	0.24	8 7 5	21 28 42	<0.1 <0.1 <0.1	<0.02 <0.02 <0.02	R66.21
Italy, 1987 (Favorita)	WP	3.6	0.48	8 7 5	21 28 42	<0.1 <0.1 <0.1	<0.02 <0.02 <0.02	R66.21
Italy, 1987 (Primora)	WP	1.4	0.24	8 7 5	21 28 42	<0.1 <0.1 <0.1	<0.02 <0.02 <0.02	R66.18
Italy, 1987 (Primora)	WP	2.9	0.48	8 7 5	21 28 42	<0.1 <0.1 <0.1	<0.02 <0.02 <0.02	R66.18
Japan, 1977 (Danshaku)	WP	2.8-3.8	0.19	4 6	14 21 14 21	0.01 0.02 0.01 0.03	<0.01 <0.01 <0.01 <0.01	53P-7-65-66
Japan, 1977 (Nohrin 1 goh)	WP	4.7	0.19	4 7	15 22 15 22	0.01 0.01 0.01 <0.01	<0.01 <0.01 <0.01 <0.01	53P-7-65-66
Netherlands, 1984 (Bintje)	WP SC SC	1.6-3.2 1.6-3.2 1.4-2.9	0.27-0.53 0.27-0.53 0.24-0.48	5 5 5	14 14 14	<0.01 (2) <0.01 (2) <0.01 (2)	0.002, 0.007 0.009, 0.003 0.008, 0.007	PH8419
Netherlands, 1984 (Bintje)	WP SC SC	1.6-3.2 1.6-3.2 1.4-2.9	0.27-0.53 0.27-0.53 0.24-0.48	5 5 5	11 11 11	<0.01 (2) <0.01 (2) <0.01 (2)	0.01, 0.003 0.006, 0.002 0.003, 0.009	PH8420
Netherlands, 1984 (Bintje)	WP SC SC	1.6-3.2 1.6-3.2 1.4-2.9	0.27-0.53 0.27-0.53 0.24-0.48	10 10 10	7 7 7	<0.01 (2) <0.01 (2) <0.01 (2)	0.003, 0.008 0.005, 0.008 0.002, 0.007	PH8421
Netherlands, 1985 (Bintje)	WP SC SC	1.6-3.2 1.6-3.2 1.4-2.9	0.27-0.53 0.27-0.53 0.24-0.48	9 9 9	9 9 9	<0.01 (2) <0.01 (2) <0.01 (2)	0.015, 0.006 0.004, 0.006 0.010, 0.005	PH8518
Netherlands, 1985 (Bintje)	WP SC SC	1.6-3.2 1.6-3.2 1.4-2.9	0.27-0.53 0.24-0.49 0.24-0.48	8 8 8	17 17 17	<0.01 (2) <0.01 (2) <0.01 (2)	0.011, 0.017 0.009, 0.011 0.009, 0.006	PH8520
Netherlands, 1986 (Bintje)	WP SC SC SC WP SC	1.6-3.2 1.5-2.9 1.5-2.9 1.0-1.9 1.6-3.2 1.5-2.9	0.27-0.53 0.24-0.49 0.24-0.49 0.16-0.32 0.27-0.53 0.24-0.49	9 9 9 9 9 9	20 20 20 20 20 20	<0.01 (2) <0.01, 0.04 <0.01 (2) 0.08, <0.01 <0.01, 0.04 <0.01 (2)	<0.002 (2) 0.002, 0.006 <0.002, 0.008 <0.002 (2) 0.005, <0.002 0.006, 0.007	PH8620
Netherlands, 1987 (Bintje)	SC SC WG SC	1.5-2.9 1.5-2.9 1.6-3.2 1.5-2.9	0.24-0.49 0.24-0.49 0.27-0.54 0.24-0.49	8 8 8 8	12 12 12 12	<0.02 (2) <0.02 (2) <0.02 (2) <0.02 (2)	<0.002, 0.009 0.003, 0.004 0.004, 0.003 0.002, 0.009	PH8719
Netherlands, 1988 (Bintje)	SC WP WG	1.6-3.2 1.6-3.2 1.6-3.2	0.27-0.54 0.27-0.54 0.27-0.54	9 9 9	6 6 6	<0.05, 0.06 <0.05, 0.14 0.10, <0.05	0.009, 0.008 0.011, 0.006 0.014, 0.018	PH8824
Netherlands, 1988 (Bintje)	SC WP WG	1.6-3.2 1.6-3.2 1.6-3.2	0.27-0.54 0.27-0.54 0.27-0.54	8 8 8	31 31 31	<0.05 (2) <0.05 (2) <0.05, 0.10	0.001, 0.010 0.002, 0.014 0.001, 0.016	PH8826
Netherlands, 1988 (Bintje)	WP SC WG	1.6-3.2 1.5-2.9 1.6-3.2	0.27-0.54 0.24-0.49 0.27-0.54	7 7 7	22 22 22	<0.05 (2) <0.05 (2) 0.21, <0.05	0.004, 0.009 0.007 (2) 0.008 (2)	PH8827
Netherlands, 1988 (Bintje)	WP SC WG	1.6-3.2 1.5-2.9 1.6-3.2	0.27-0.54 0.24-0.49 0.27-0.54	10 10 10	18 18 18	<0.05 (2) <0.05 (2) <0.05 (2)	0.007, 0.004 0.009, 0.004 0.005, 0.006	PH8829

CROP Country, year (Variety)	Application [1]				Day	Residues, mg/kg [2]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
Netherlands, 1989 (Bintje) 	WP SC SC	1.6-3.2 1.6-3.2 1.6-3.2	0.27-0.54 0.27-0.54 0.27-0.54	7 7 7	12 12 12	0.14, 0.11 <0.05, 0.07 0.06, <0.05	0.005, 0.010 0.011, 0.008 0.008, 0.010	PH8938
Netherlands, 1989 (Bintje) 	WP SC SC	1.6-3.2 1.6-3.2 1.6-3.2	0.27-0.54 0.27-0.54 0.27-0.54	7 7 7	10 10 10	<0.05 (2) <0.05, 0.06 0.13, 0.07	0.013, 0.031 0.010, 0.007 0.010, 0.023	PH8939
Netherlands, 1990 (Bintje) 	SC WG	1.6-3.2 1.6-3.2	0.27-0.54 0.27-0.54	8 8	22 22	0.03 (2) 0.03, 0.04	0.015, 0.011 0.015, 0.013	PH9055
Netherlands, 1990 (Bintje) 	SC WG	1.6-3.2 1.6-3.2	0.40-0.80 0.40-0.80	9 9	37 37	0.03, 0.04 0.03 (2)	0.008, 0.025 0.005, 0.014	PH9057
Netherlands, 1990 (Bintje) 	WP SC	1.6-3.2 1.6-3.2	0.27-0.54 0.27-0.54	8 8	21 21	0.03, 0.04 0.04 (2)	0.010, 0.004 <0.002, 0.007	PH9059
Netherlands, 1990 (Bintje) 	WP SC	1.6-3.2 1.6-3.2	0.27-0.54 0.27-0.54	7 7	11 11	0.05, 0.03 0.04, 0.03	0.018, 0.013 0.011, 0.032	PH9060
UK, 1991 (Movis Piper)	WG	1.3	0.52	5	20	<0.01		OA/0011
UK, 1991 (Movis Piper)	WG	1.3	0.52	5	26	0.01		OA/011
UK, 1991 (King Edward)	WG	1.3	9.52	4	47	<0.01		OA/011
SUGAR BEET								
France, 1983 (Major)	SC	3.2	0.8	2	51	<0.3		R65.34
France, 1983 (Massabel)	SC	3.2	0.8	2	51	<0.3		R65.34
Italy, 1989 (Kaweduka) 	WP	2.0 4.0	0.7 1.4	3 3	28 28	<0.1 0.17		R72.4
Italy, 1989 (Maribo Monou) 	WP	2.0 4.0	0.7 1.4	3 3	28 28	0.1 0.1	<0.01 <0.01	R72.4
Italy, 1989 (Monohil) 	WP	2.0 4.0	0.4 0.8	3 3	28 28	<0.1 0.2	<0.01 <0.01	R72.4
Japan, 1991 (Mono_su- s) 	WP	2.8	0.19	5	14 21 30	<0.005 <0.005 <0.005	<0.01 <0.01 <0.01	3P-7-246
YAM, CHINESE								
Japan, 1983 	WP	4.7	0.19	4 6	7 14 21 7 14 21	<0.004 <0.004 <0.004 <0.004 <0.004 <0.004		58-11-9

[1] st: seed treatment.
[2] c: control sample.

Table 39. Mancozeb residues (as CS_2) in potatoes from supervised trials in Germany. Underlined residues are from treatments according to GAP.

Year (Variety)	Application [1]				Day	Residues, mg/kg [2]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
1971 (Bintje)	WP	3.4	0.34	st+5	11	<0.3		R67.24
1974 (Saskia) 	WP	1.4	0.24	3	0 3 5 7 10	pu <0.3, pe <0.3 pu <0.3, pe <0.3 pu <0.3, pe <0.3 pu <0.3, pe <0.3 pu <0.3, pe <0.3		R69.12
1974 (Saskia) 	WP	1.8	0.3	3	0 3 6 8 14	<0.3 <0.3 <0.3 <0.3 <0.3		R69.12
1980 (Amigo) 	WP	4.3 +1.2 +1.4	1.1 +0.3 +0.35	1 +1 +2	7 32 56	0.05 <0.02 <0.02	<0.02 <0.02 <0.02	R65.3

Year (Variety)	Application [1]				Day	Residues, mg/kg [2]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
1980 (Grandi-Folia)	WP	1.2 +1.4	0.3 +0.36	1 +2	11 36	0.02 0.02	<0.02 <0.02	R65.3
1980 (Hansa)	WP	1.4	0.36	st+4	7 14 22	0.03 <0.02 <0.02	<0.02 <0.02 <0.02	R65.3
1981 (Amigo)	WP	4.0		st+3	0 7 14	<0.02 <0.02 <0.02	<0.02 <0.02 <0.02	R69.21
1981 (Steffi)	WP	1.4	0.24	st+4	0 7 14	<0.02 <0.02 <0.02	<0.02 <0.02 <0.02	R69.21
1988 (Nicola)	WP	1.4	0.48	6	0 3 5 7	<0.05 <0.05 <0.05 <0.05	 <0.02	R73.4
1988 (Rosi)	WP	1.4	0.48	6	0 3 5 7	<0.05 <0.05 <0.05 <0.05	 <0.02	R73.4
1988 (Quarta)	WP	1.4	0.48	6	7	<0.05	<0.02	R73.4
1988 (Secura)	WP	1.4	0.48	6	7	0.13	<0.02	R73.4
1988 (Hansa)	WP	1.4	0.28	6	7	<0.05	<0.02	R73.4
1988 (Hansa)	WP	1.4	0.28	6	7	<0.05	<0.02	R73.4
1988 (Hansa)	WG	1.4	0.28	6	7	<0.05	<0.02	R73.5
1988 (Hansa)	WG	1.4	0.28	6	7	<0.05	<0.02	R73.5
1988 (Rosi)	WG	1.4	0.48	6	7	<0.05	<0.02	R73.5
1988 (Nicola)	WG	1.4	0.48	6	0 3 5 7	<0.05 <0.05 <0.05 <0.05		R73.5
1988 (Quarta)	WG	1.4	0.48	6	7	0.05	0.02	R73.5
1988 (Secura)	WG	1.4	0.48	6	7	0.26	0.02	R73.5
1990 (Kapta-vandel)	WP	1.6	0.4	12	6	<0.05		R78.26
1990 (Manon)	WP	1.6	0.4	8	11	0.09		R78.27
1990 (Bintje)	WP	1.6	0.53	7	33	0.47		R78.28
1990 (Bintje)	WP	1.6	0.53	6	26	0.21		R78.29
1990 (Bintje)	WP	1.6	0.4	6	10	<0.05		R78.4
1990 (Bintje)	WP	1.6	0.29	10 20	23 19	0.09 0.34		R78.5
1990 (Bintje)	WP	1.6	0.4	st+7	54	<0.05		R78.6
1990 (Bintje)	WP	1.6	0.32	13	58	<0.05		R78.10
1990 (Bintje)	WP	1.6	0.32	9	35	0.07		R78.22
1990 (Bintje)	WP	1.6	0.32	14	36	<0.05		R78.23
1990 (Bintje)	WP	1.6		8	27	<0.05		R78.25

[1] st: seed treatment
[2] pu: pulp; pe: peel.

Table 40. Mancozeb residues (as CS_2) in root and tuber vegetables from supervised trials in the USA. Underlined residues are from treatments according to GAP.

CROP Year (Variety)	Application [1]	Day	Residues, mg/kg [2]	Ref.

	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
CARROT								
CA, 1985 (Emperator)	WP	1.8	0.32	5	3 7 14	<0.03 0.04 0.05	<0.01 <0.01 <0.01	85-0221
CA, 1985 (Emperator)	WP	1.8	0.32	5	3 7 14	<0.03 <0.03 0.04	<0.01 <0.01 <0.01	85-0222
CA, 1985 (Emperator)	WP	1.8	0.32	a 5	3 6 14	<0.03 0.06 <0.03	<0.01 <0.01 <0.01	85-0258
OH, 1985 (Scar. Nantes)	WP	1.8	0.38	5	3 7 10 14	0.08 0.03 <0.03 <0.03	<0.01 <0.01 <0.01 <0.01	85-0279
TX, 1985 (Danver 126)	WP	1.6	3.8	a 6	3 7 10	0.16 0.10 0.04	<0.01 <0.01 <0.01	85-0303
MI, 1985 (Trophy)	WP	1.8	0.38	5	2 6 9 13	0.24 0.10 0.07 0.03 c 0.29	<0.01 <0.01 <0.01 <0.01	85-0506
GINSENG								
WI, 1986	SC	1.8	0.19	33	15	0.16	0.028	86-0321
	SC	3.6	0.38	24	351	0.031	0.02	
WI, 1986	SC	1.8	0.19	24	351	<0.03	<0.01	86-0354
	SC	3.6	0.38	24	351	0.035	<0.01	
WI, 1986	SC	1.8	0.19	13	351	<0.03	<0.01	86-0322
	SC	3.6	0.38	13	351	<0.03	<0.01	
WI, 1987	WP	1.8	0.19	4	14	0.24	0.01	87-0215
	WP	1.8	0.19	5	0	1.1	0.02	
POTATO								
ID, 1975 (Rus Burbank)	WP	1.8	1.9	a 7	50	<0.03	<0.02	75-537-02
ME, 1975 (Rus Burbank)	WP	0.26-0.80 +1.2		ga 6 +6	23	<0.03	<0.02	75-538-02
NY, 1975 (Katahdin)	WP	0.8 +1.2 +1.6		1 +1 +6	27	<0.03	<0.02	75-514-02
OH, 1975 (Norchip)	WP	1.8		ga 9	1	0.1	<0.02	75-459-02
OR, 1975 (Rus Burbank)	WP	1.8	1.9	a 5	1	<0.03	<0.02	75-555-02
PA, 1975 (Katahdin)	WP	1.8	6.4	a 14	1	<0.03	<0.02	75-494-02
WI, 1975	WP	2.2		ga 13	1	<0.03	<0.02	75-443-02
FL, 1976 (Sebago)	WP	1.8		ga 13	0 4 8 12	0.05 <0.03 0.05 <0.03	<0.02 <0.02 <0.02 <0.02	76-0083
FL, 1976 (Red La Soto)	WP	1.8		ga 13	0	<0.03	<0.02	76-0084
FL, 1976 (Norchip)	WP	1.5	0.22	14	0	<0.03	<0.02	76-0155
ID, 1976 (Russet)	WP	1.8	1.9	a 4	2	<0.03	<0.02	76-0408
ID, 1976 (Russet)	WP	1.8	1.9	a 5	9	<0.03	<0.02	76-0409
ID, 1976 (Russet)	WP	1.8	0.74	5	13	<0.03	<0.02	76-0435
ME, 1976 (Superior)	WP	1.1 +1.7	4.0 +6.0	a 2 a 6	1	<0.03	<0.02	76-0699
ME, 1976 (Katahdin)	WP	1.0 +1.7	0.27 +0.40	7 +3	12	<0.03	<0.02	76-0700
ME, 1976 (Chippwa)	WP	1.1	0.4	4	56	<0.03	<0.02	76-0701
ME, 1976 (Superior)	WP	1.1 +1.7	0.34 +0.51	6 +4	12	<0.03	<0.02	76-0703
NY, 1976 (162)	WP	1.8	6.4	a 9	0		<0.02	76-0614
NY, 1976 (Kennebec)	WP	1.8	6.4	a 8	6	<0.03	<0.02	76-0647
OH, 1976 (Norchip)	WP	2.2	4.8	a 8	0 4	0.05 <0.03	<0.02 <0.02	76-0329

CROP Year (Variety)	Application [1]				Day	Residues, mg/kg [2]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
					6 15	<0.03 <0.03	<0.02 <0.02	
OH, 1976 (Superior)	WP	2.2	4.8	a 9	0	<0.03	<0.02	76-0394
OH, 1976 (Superior)	WP	2.2	4.8	a 9	0	<0.03	<0.02	76-0395
PA, 1976 (Katahdin)	WP	1.8	2.1	a 6	0	<0.03	<0.02	76-0407
PA, 1976 (Kathdan)	WP	1.8	0.55	6	0	<0.03	<0.02	76-0421
PA, 1976 (Man Ken Kath)	WP	1.8	6.4	a 10	0	<0.03	<0.02	76-0434
NY, 1976 (162)	WP	1.8	6.4	a 9	0	<0.03	<0.02	76-0613
NY, 1976 (162)	WP	1.8	6.4	a 9	0	<0.03		76-0614
PA, 1976 (Kathadin)	WP	1.8		11	0 3 7 14	<0.03 <0.03 <0.03 <0.03	<0.02 <0.02 <0.02 <0.02	76-0629
WA, 1976	WP	1.8	0.32	7	0	<0.03	<0.02	76-0652
WI, 1976 (Superior)	WP	2.8	6.0	a 13	0 4 7 14	<0.03 <0.03 <0.03 <0.03	<0.02 <0.02 <0.02 <0.02	76-0617
WI, 1976 (Superior)	WP	2.8	6.0	a 14	3	<0.03	<0.02	76-0649
WI, 1976 (Burbank)	WP	1.7	4.5	a 14	2	<0.03	<0.02	76-0650
WI, 1976 (Burbank)	WP	1.7	0.40	13	5	<0.03	<0.02	76-0651
CA, 1987 (Russet Burbank)	WP	2.7	1.4	a 5	0 5 15	<0.03 <0.03 <0.03	<0.01 0.02 0.025	88-0059
SUGAR BEET								
CA, 1985	WP	1.8		a 5	6 14 21	0.04 0.05 <0.03	<0.01 <0.01 <0.01	85-0264
CA, 1985	WP	1.8	0.32	ga 5	6 13 20	0.78 0.39 0.21	0.01 <0.01 <0.01	85-0292
TX, 1985 (Monohy D2)	WP	1.8	3.8	a 6	7 10 14 21 28	0.17 0.12 0.06 0.03 <0.03 c 0.07	<0.01 0.01 <0.01 <0.01 <0.01	85-0329
ID, 1985 (WS-78)	WP	1.8	1.0	8	7 15 21	0.15 0.18 0.07	0.025 0.017 <0.01	85-0363
ID, 1985 (WS-78)	WP	1.8	1.0	8	7 15 21	0.13 0.18 0.10	0.042 <0.01 <0.01	85-0365
MN, 1985 (KW-3394)	WP	1.8	0.33	7	7 14 21	0.13 0.04 0.03	0.02 0.02 0.01	85-0499
ND, 1985 (Monofort)	WP	1.8	3.8	a 5	7 14 21	0.13 0.08 0.05 c 0.03	0.02 0.02 0.02	85-0500
ND, 1985 (Beta 1230)	WP	1.8	3.8	a 5	7 14 21	0.09 0.06 <0.03	0.01 <0.01 <0.01	85-0501
MN, 1985 (KW-3394)	WP	1.8	0.33	7	14	0.12 c 0.10	0.02	85-0515

[1] a: aerial application; ga: ground and aerial application.
[2] c: control sample.

Table 41. Mancozeb residues (as CS_2) in stalk and stem vegetables from supervised trials in Australia, France and The Netherlands. Underlined residues are from treatments according to GAP.

CROP Country, year (Variety)	Application	Day	Residues, mg/kg[1]	Ref.

	Form	kg ai/ha	kg ai/hl	No.[2]		EBDC as CS_2	ETU	
ASPARAGUS								
France, 1990 (Aneto)	WP	2.1	0.42	8	175	0.16, c 0.22		R77.30
France, 1990 (Aneto)	WP	2.1		8	161	0.18, c 0.16		R77.31
France, 1990 (Aneto)	WP	1.5	0.5	7	151	0.36, c 0.21		R77.47
France, 1990 (Juno, Oesto, Cibo)	WP	2.1	1.4	4	233	0.49, c 0.23		R77.29
France, 1991 (Desto)	WP	2.1	0.7	7	142	<0.05, c <0.05		R78.11
France, 1991 (Larac)	WP	2.1	0.42	9	191	<0.05, c <0.05		R78.12
CHARD (SILVER BEET)								
Australia, 1992 (Ford Hook Giant)	WP	1.8		3	0 7 14 21 28	8.3 0.6 0.2 0.3 0.2		AUK-92-007
	WP	3.5		3	0 7 14 21 28	14 1.5 <0.1 0.6 0.3		
WITLOOF								
France, 1984	SC	12 24		1i 1i	24 24	<0.3 <0.3	b 0.28 b 0.25	R65.15
France, 1984	SC	12		1i	20	<0.3	b <0.005	R65.16
France, 1985	SC	6 12		1i 1i	21 21	<0.3 <0.3	<0.01 <0.01	R65.17
France, 1990	WP	1.5[3]		1	208	<0.02		R73.22
France, 1990	SC		0.3[3]	1	24	<0.02		R72.11
France, 1990	WP	150		1	21	0.11		R80.9
Netherlands, 1989	SC		0.65[3]	1	21	<0.05	<0.02	R72.22

[1] c: control sample; b: boiled.
[2] i: indoors.
[3] application to roots.

Table 42. Mancozeb residues (as CS_2) in stalk and stem vegetables from supervised trials in the USA. Underlined residues are from treatments according to GAP.

CROP State, year (Variety)	Application [1]				Day	Residues, mg/kg [2]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
ASPARAGUS								
WA, 1985 (WSU1)	WP	1.3	0.29	4	231	<0.03	<0.01	85-0134
MI,1985	WP	1.8	0.80	5	252	<0.03	<0.01	85-0136
WA, 1985	WP	1.8	1.9	a 1	321	0.05	<0.01	85-0278
CA, 1986 (Colossal)	WP	1.8		4	124	0.04	<0.01	86-0083
CA, 1986 (Colossal)	WP	1.8		4	124	<0.03	<0.01	86-0084
CA, 1986 (Colossal)	WP	1.8		a 4	124	<0.03	<0.01	86-0085
CELERY								
FL, 1985 (June Belle)	WP	1.5	0.15	a 17	0 3 5 7 10 14 21	2.7 2.0 1.6 1.3 1.1 0.81 0.56 c 0.08	0.03 0.02 0.01 0.02 <0.01 <0.01 <0.01	85-0165
CA, 1985 (5270R)	WP	1.8		ga 8	7 14 21	1.8 0.78 0.46 c 0.06	0.02 0.02 0.01	85-0350
CA, 1985 (5275)	WP	1.8		ga 8	8 14 21	2.6 2.1 0.68 c 0.41	0.01 <0.01 <0.01	85-0397

CROP State, year (Variety)	Application [1]				Day	Residues, mg/kg [2]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
OH, 1985 (Florida 683)	WP	1.8	0.38	9	7 14 21 28	0.27 0.17 0.10 0.07	0.02 0.01 <0.01 <0.01	85-0401
CA, 1985 (5270R)	WP	1.8	0.96	ga 8	7 14	0.60 0.28	<0.01 <0.01	85-0454
CA, 1985 (Florida 683)	WP	1.8		ga 8	7 14 21	0.81 0.60 0.36 c 0.20	<0.01 <0.01 <0.01	85-0455
MI, 1985 (Florida 683)	WP	1.8	0.38	7	7 14 21 28	0.53 0.28 0.20 0.12	0.01 <0.01 <0.01 <0.01	85-0503
CA, 1985 (5275)	WP	1.8		ga 8	7 14 21	0.05 0.34 0.20	0.01 0.01 0.01	85-0561
FL, 1989 (June Belle)	WP	1.8	0.68	4 a 4	14 14	s 0.15 s+l 0.84 s 0.10 s+l 0.50	s 0.043 s+l 0.16 s 0.026 s+l 0.074	89-0124

[1] a: aerial application; ga: ground and aerial application.
[2] s: analysis on stalk; s+l: analysis on stalk + leaf; c: control sample

Table 43. Mancozeb residues (as CS_2) in cereal grains from supervised trials in Brazil, Canada, France, Germany, The Netherlands, Spain and the UK. Underlined residues are from treatments according to GAP.

CROP Country, year (Variety)	Application				Day	Residues, mg/kg [1]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
BARLEY								
Brazil, 1989	WP	2.0		3	0 7 14 21	1.7 1.1 0.03 <u><0.03</u>		021/90
Brazil, 1989	WP	4.0		3	0 7 14 21	11 2.8 2.6 <0.03		021/90
Netherlands, 1986 (Hasso)	WP WP	1.6 1.6	0.27 0.27	2 2	58 58	<0.01, 0.14 <0.01, 0.38	<0.002 (2) <0.002 (2)	PH8616
Netherlands, 1987 (Hasso)	WP SC	1.6 1.6	0.27 0.27	2 2	67 67	0.28, <0.03 <0.03, 0.11	0.046, 0.016 <0.002 (2)	PH8717/2
Netherlands, 1988 (Prisma)	WP WP	1.6 1.6	0.27 0.27	2 2	58 58	<0.05 (2) 0.14, <0.05	<0.002 (2) <0.002 (2)	PH8835
Netherlands, 1988 (Trumpf)	WP WP	1.6 1.6	0.27 0.27	2 2	60 60	0.65, 0.30 0.61, 0.41 c 0.68, c 0.24	<0.002 (2) <0.002 (2) c 0.003	PH8838
RICE								
Brazil, 1990	WP	1.6		3	18 25 40	2.5 2.0 <u><0.03</u>		281/90
Brazil, 1990	WP	3.6		3	18 25 40	3.1 2.2 <u>0.34</u>		281/90
Brazil, 1990	WP	7.2		3	18 25 40	4.8 4.2 0.42		281/90
SUMMER WHEAT								
Germany, 1985	WP	1.6	0.4	2	0 27 42 63	e 9.6 e 9.6 <u><0.05</u> <u><0.05</u>	 <0.02	R60.6
Germany, 1985	WP	1.6	0.4	2	0 31 39	e 8.7 e 2.0 <u>0.4</u>		R60.6

CROP Country, year (Variety)	Application				Day	Residues, mg/kg [1]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
					63	<0.05	<0.02	
Germany, 1986 (Ralle)	SC	1.6	0.4	2	0 15 31 43	e 15 e 4.8 e 2.2 <0.05	 <0.02	R60.7
WHEAT								
Brazil, 1983	WP	2.2		3	32	0.07		R&H 3318322
Brazil, 1988	WP	4.4		3	32	0.14		R&H 3318322
Canada, 1985	WP	1.8		1	46	<0.1 b <0.05 f <0.1	<0.02 b <0.02 f <0.04	#13
Canada, 1985	WP	1.8		2	46-58	<0.1 b <0.05 f <0.1	<0.02 b <0.02 f <0.04	#13
Canada, 1985	WP	1.8 +3.6		2	40 & 60	<0.1 b <0.05 f <0.1	<0.02 b <0.02 f <0.04	#13
France, 1990 (Scipion)	WP	1.5	0.375	2	64	0.29 c 0.12		R78.17
France, 1990 (Cando)	WP	1.5	0.375	2	62	0.26 c 0.30		R78.18
France, 1990 (Scipion)	WP	1.5	0.375	2	49	0.08 c 0.11		R78.20
France, 1990 (Cando)	WP	1.5	0.375	2	47	0.11 c 0.15		R78.19
France, 1990 (Beauchamps)	WP	1.5	0.375	2	64	0.16		R78.21
France, 1991 (Hornet)	SC	2.0	0.66	1	55	<0.1		R80.3
France, 1991 (Hornet)	SC	1.5 +2.0	0.5 & 0.66	2	55	<0.1		R80.3
France, 1991 (Foxal)	SC	2.2	0.75	1	91	<0.1		R80.2
France, 1991 (Scipion)	SC	2.2	0.75	1	89	<0.1		R80.1
Spain, 1991 (Mexicali)	SC	1.6	0.45	1 2 1	91 76 76	<0.1 0.17 <0.1		R80.31
WINTER WHEAT								
Germany, 1974 (Diplomat)	WP	1.6	0.27	1	21 35 57 64	<0.2 <0.2 <0.2 <0.2		R60.5
Germany, 1974 (Kormoran)	WP	1.6	0.27	1	35 62 70	<0.2 <0.2 <0.2		R60.5
Germany, 1974 (Diplomat)	WP	1.6	0.27	1	35 43 80	<0.2 <0.2 <0.2		R60.5
Germany, 1985	WP	1.6	0.4	2	0 34 54 66	e 13 e 1.1 <0.05 <0.05	 <0.02	R60.6
Germany, 1985	WP	1.6	0.4	2	0 19 41 62	e 10 e 5.4 <0.05 <0.05	 <0.02	R60.6
Germany, 1985 (Kanzler)	SC	1.6	0.8	2	0 14 24 47	e 10 e 1.7 e 1.6 <0.05	 <0.02	R60.7
Germany, 1986 (Kanzler)	SC	1.6	0.4	2	0 20 39 46	e 13 e 1.8 <0.05 <0.05	 <0.02 <0.02	R60.7
Germany, 1986 (Diplomat)	SC	1.6	0.4	2	0 24 39 52	e 21 e 3.4 e 1.3 <0.05	 <0.02	R60.7
Germany, 1986 (Okapi)	SC	1.6	0.4	2	0 25 40	e 10 e 1.6 e 0.89		R60.7

CROP Country, year (Variety)	Application				Day	Residues, mg/kg [1]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
					56	<0.05	<0.02	
Netherlands, 1984 (Okapi)	WP	1.6	0.49	2	61	<0.02 (2)	<0.02 (2)	PH8431
	SC	1.5	0.45	2	61	<0.02 (2)	<0.02 (2)	
	SC	1.4	0.44	2	61	<0.02 (2)	<0.02 (2)	
Netherlands, 1985 (Saiga)	WP	1.6	0.40	2	68	0.75, 0.49	0.002, <0.002	PH8526
	SC	1.5	0.36	2	68	<0.01, 0.24	<0.002 (2)	
	SC	1.4	0.36	2	68	<0.01, 0.82	<0.002 (2)	
Netherlands, 1985 (Marksman)	WP	1.6	0.32	2	57	<0.01 (2)	<0.002 (2)	PH8527
	SC	1.5	0.29	2	57	<0.01 (2)	<0.002 (2)	
	SC	1.4	0.29	2	57	<0.01 (2)	<0.002 (2)	
Netherlands, 1986 (Okapi)	SC	1.5	0.36	2	66	<0.01 (2)	<0.002 (2)	PH8626
	SC	0.96	0.24	2	66	<0.01, 0.06	<0.002 (2)	
	SC	1.5	0.36	2	66	<0.01 (2)	<0.002 (2)	
Netherlands, 1987 (Arminda)	WP	1.6	0.26	2	64	<0.02 (2)	<0.002 (2)	PH8727
	SC	1.6	0.26	2	64	<0.02 (2)	<0.002 (2)	
	WG	1.6	0.26	2	64	<0.02 (2)	<0.002 (2)	
Netherlands, 1987 (Obelisk)	SC	3.2	0.64	2	0	e 12		R60.8
					28	e 3.9		
					42	e 42		
					60	<0.05	<0.02	
Netherlands, 1987 (Okapi)	SC	3.2	0.64	2	0	e 14		R60.8
					28	e 9.6		
					42	e 3.4		
					60	<0.05	<0.02	
Netherlands, 1987 (Obelisk)	SC	3.2	0.64	2	0	e 11		R60.8
					28	e 2.9		
					42	e 2.1		
					60	<0.05	<0.02	
Netherlands, 1987 (Okapi)	SC	3.2	0.64	2	0	e 14		R60.8
					28	e 13		
					42	e 7.0		
					60	<0.05	<0.02	
Netherlands, 1988 (Obelisk)	SC	1.5	0.25	2	68	<0.05, 0.05	<0.002, 0.003	PH8839
	WG	1.5	0.25	2	68	<0.05, 0.14	0.003, <0.002	
	SC	1.5	0.25	2	68	<0.05, 0.08	0.004 (2)	
Netherlands, 1990 (Obelisk)	WG	1.6	0.27	2	63	<0.03 (2)	<0.002 (2)	PH9047
	SC	1.6	0.27	2	63	0.12, <0.03	<0.002, 0.008	
Netherlands, 1990 (Obelisk)	WP	1.6	0.27	2	68	<0.03 (2)	0.013, 0.016	PH9050
	SC	1.6	0.27	2	68	<0.03 (2)	0.006, 0.012	
	WG	1.6	0.27	2	68	0.09, 0.05	0.024, 0.072	
Netherlands, 1990 (Obelisk)	WP	1.6	0.27	2	76	0.04 (2)	0.017, <0.002	PH9052
	SC	1.6	0.27	2	76	<0.03 (2)	<0.002 (2)	
	WG	1.6	0.27	2	76	<0.03, 0.03	<0.002 (2)	
Netherlands, 1990 (Pagode)	SC	1.6	0.27	2	60	0.06, 0.20	0.020, <0.002	PH9054
	WG	1.6	0.27	2	60	0.03 (2)	0.024, 0.019	
UK, 1990 (Haven)	WP	1.6	0.64	3	37	0.25	0.024	R78.1
				2	50	0.18	0.01	
						c 0.08	c 0.01	
UK, 1990 (Hornet)	WP	1.6	0.64	3	36	0.42	0.01	R78.1
				2	50	0.26	0.006	
UK, 1990 (Hornet)	WP	1.6	0.64	3	46	0.05	0.005	R78.1
				2	56	0.07	0.007	
UK, 1990 (Apollo)	WP	1.6	0.64	3	47	0.5	0.01	R78.1
				2	57	0.09	0.008	

[1] b: bran; e: ears; f: flour; c: control sample.

Table 44. Mancozeb residues (as CS_2) in cereal grains from supervised trials in the USA. Underlined residues are from treatments according to GAP. All WP.

CROP State, Year (Variety)	Application	Day	Residues, mg/kg [2]	Ref.

	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
BARLEY							
ND, 1985 (Morex)	1.8	3.2	3[1]	25	0.55	<0.01	85-0272
ND, 1985 (Robust)	1.8	3.2	3[1]	25	0.46	<0.01	85-0273
WA, 1985 (Sevin)	1.8	3.2	3[1]	20	0.19	<0.01	85-0352
MAIZE							
GA, 1983 (F-4333)	1.8	1.5	4	10 20	0.078 0.045	<0.02	83-0200
FL, 1983 (NK508)	1.3	0.41	11 14	25 11	0.028 0.16	<0.02	83-0228
IN, 1983 (PA63709)	3.4	7.2	2	10 20	0.11 0.041	0.02	83-0237
IA, 1983 (P80)	1.7 3.4	2.6 5.2	2 2	11 21 11 21	<0.03 <0.03 <0.03 <0.03	<0.02 <0.02	83-0253
IL, 1983 (Funk G4740)	1.7	3.6	2	10 20	<0.03 0.03 c 0.08	<0.02	83-0358
FL, 1983 (Pioneer)	1.3	0.14	16	7 14	<0.03 <0.03		83-0419
AR, 1985 (North Upking)	1.3	1.4	5[1]	20 29 40	e <0.03 e <0.03 e <0.03	e <0.01 e <0.01 e <0.01	85-0337
IA, 1985	1.7	0.45	4	3 7 14 39	e 0.73 e 0.19 e 0.095 e <0.03	e 0.02 e 0.01 e 0.01 e <0.01	85-0453
WHEAT							
MN, 1975 (Era)	1.8		2[1]	28	0.17	<0.02	75-421-02
MN, 1975 (Era)	1.8		2[1]	47	0.1	<0.02	75-467-02
MN, 1975 (Era)	1.8		2[1]	42	0.1	<0.02	75-468-02
AL, 1981 (Coker 747)	1.8 1.8	3.8 3.8	2 3	28 28	0.07 0.09 c 0.05	<0.01 <0.01	81-0167
AL, 1981 (Coker 747)	1.8 1.8	3.8 3.8	2 3	28 28	0.10 0.05 c 0.03	<0.01 <0.01	81-0168
TN, 1981 (McNair 1003)	1.8		2	51	0.02	<0.01	81-0212
TN, 1981 (Arthur 71)	1.8	3.8	3	42	0.04	<0.01	81-0214
TX, 1988 (NK812)	1.8	3.8	3[1]	46	0.050	<0.01	88-0105
OK, 1988 (Florida 302)	1.8	3.8	3[1]	56	0.035	<0.01	88-0131
MO, 1988 (Caldwell)	1.8	0.69	3	36	<0.03	<0.01	88-0185

[1] aerial application; [2] e: ears; c: control sample.

Table 45. Mancozeb residues (as CS_2) in dry hops from supervised trials in Germany. Underlined residues are from treatments according to GAP. All WP.

Year (Variety)	Application				Day	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
1982 (Brewer's Gold)	WP	0.32 +0.42 +0.53 +0.63 +0.79 +1.2 +1.1	0.053	2 +2 +1 +1 +3 +1 +3	42	2.2	<0.1 beer 0.04	R69.23
1982 (Tettnanger Frühhopfen)	WP	0.32 +0.42 +0.53 +0.79 +0.87 +0.95 +1.1	0.053	2 +1 +3 +1 +1 +3 +2	35	<1	<0.1 beer 0.02	R69.23

Table 46. Mancozeb residues (as CS_2) in oilseeds from supervised trials in Australia, France, The Netherlands and the USA. Underlined residues are from treatments according to GAP.

CROP Country (State), year (Variety)	Application[1]				Day	Residues, mg/kg[2]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
PEANUT								
Australia, 1992 (Virginia Bunch)	WP	1.8		6	0 7 14 21 28	<0.1 <0.1 <0.1 <0.1 <0.1		AUK-92-008
	WP	3.5		6	0 7 14 21 28	<0.1 <0.1 <0.1 <0.1 <0.1		
USA (GA), 1974 (Florunner)	WP	1.8 3.6	2.3 2.3	6 6	27 27	<0.03 <0.03	<0.02 <0.02	74-171-02
USA (AL), 1974 (Florunner)	WP	1.3 2.7	1.0 2.1	7 7	7 7	<0.03 <0.03	<0.02 0.02	74-180-02
USA (NC), 1984 (Florigiant)	WP	2.7	1.9	4	24	<0.03	<0.01	85-0383
USA (TX), 1984 (Florunner)	WP	2.7		5	47	<0.03	<0.01	85-0452
USA (TX), 1984 (Florunner)	WP	2.7		5	48	<0.03	<0.01	85-0454
USA (VA), 1984 (Florigiant)	WP	2.7	1.4	6	14	<0.03	<0.01	85-0002
RAPESEED								
France, 1985 (Jet Neuf)	SC	3.2	0.64	2	51	<0.1		R65.37
France, 1985 (Bien-Venu)	SC	3.2	0.64	2	52	<0.1		R65.35
France, 1985 (Tamdem)	SC	3.2	0.64	1	48	<0.1		R65.36
Netherlands, 1984 (Jet Neuf)	WP WP	1.6 1.6		a 2 ga 3	58 58	0.43, 2.5 0.22, 1.0 c 0.04, 1.0	0.48, 0.31 0.58, 0.51 c 0.28, 0.15	PH8418

[1] a: aerial application; ga: ground and aerial application.
[2] c: control sample.

Table 47. Mancozeb residues (as CS_2) in tree nuts, cocoa and coffee from supervised trials in Australia, Brazil and the USA. Underlined residues are from treatments according to GAP.

CROP Country (State), Year (Variety)	Form	Application			Day	Residues, mg/kg		Ref.
		kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
ALMONDS								
Australia, 1991 (Californian Papershell)	WG		0.15	7	0 7 14	0.8 0.5 0.2		AUI-91-032
	WG		0.30	7	0 7 14	2.0 <0.2 <0.2		
USA (CA), 1988 (Nonpareil)	WP	5.4	0.21	3	160	<0.03	<0.01	89-0006
USA (CA), 1988 (Nonpareil)	WG	5.4	0.21	3	160	<0.03	<0.01	89-0007
USA (CA), 1988 (Thompson)	WG	5.4	0.33	3	161	<0.03	<0.01	89-0016
USA (CA), 1988 (Thompson)	WP	5.4	0.33	3	161	<0.03	<0.01	89-0017
USA (CA), 1988 (Nonpareil)	WP	5.4	0.58	3	136	<0.03	<0.01	89-0023
COCOA								
Brazil, 1990	WP	2.4		4	0 7 14	1.7 0.34 0.34		289/90

CROP Country (State), Year (Variety)	Form	Application			Day	Residues, mg/kg		Ref.
		kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
					21	0.45		
Brazil, 1990	WP	4.8		4	0 7 14 21	2.0 0.56 0.39 0.50		289/90
COFFEE								
Brazil, 1989	WP	4.0		2	3 7 14 21	13 10 3.1 0.90		117/90
Brazil, 1989	WP	8.0		2	3 7 14 21	39 27 5.6 1.4		117/90

Table 48. Mancozeb residues (as CS_2) in cereal straws from supervised trials in Canada, France, Germany, The Netherlands and the UK. Underlined residues are from treatments according to GAP.

CROP Country, year (Variety)		Application			Day	Residues, mg/kg [1]		Ref.
		kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
BARLEY STRAW								
Netherlands, 1986 (Hasso)	WP WP	1.6 1.6	0.27 0.27	2 2	58 58	3.8 3.6 c 6.4	0.25 0.23	PH8616
Netherlands, 1987 (Hasso)	WP SC	1.6 1.6	0.27 0.27	2 2	67 67	0.20 <0.05	1.4 2.2 c 2.6	PH8717/2
Netherlands, 1988 (Trumpf)	WP WP	1.6 1.6	0.27 0.27	2 2	60 60	2.2 3.3 c 0.98	0.25 0.078 c 0.21	PH8838
WHEAT STRAW								
Canada, 1985	WP	1.8		1	46	1.3 c 1.1	<0.04	#13
Canada, 1985	WP	1.8		2	54	0.95 c 0.4	0.05	#13
Canada, 1985	WP	1.8 +3.6		2	60	2.9 c 0.4	0.05	#13
Canada, 1985	WP	1.8 +3.6		2	40	0.84 c 0.4	<0.04	#13
France, 1990 (Scipion)	WP	1.5	0.38	2	64	11 c 0.41		R78.17
France, 1990 (Cando)	WP	1.5	0.38	2	62	1.8 c 0.08		R78.18
France, 1990 (Scipion)	WP	1.5	0.38	2	49	4.8 c 0.55		R78.20
France, 1990 (Cando)	WP	1.5	0.38	2	47	1.4 c 0.18		R78.19
France, 1990 (Beauchamps)	WP	1.5	0.38	2	64	13		R78.21
France, 1991 (Hornet)	SC	2.0	0.66	1	55	1.4		R80.3
France, 1991 (Hornet)	SC	1.5 & 2.0	0.5 & 0.66	2	55	2.6 c 0.14		R80.3
France, 1991 (Foxal)	SC	2.2	0.75	1	91	0.64 c 0.53		R80.2
France, 1991 (Scipion)	SC	2.2	0.75	1	89	1.1 c 0.36		R80.1
Germany, 1974 (Diplomat)	WP	1.6	0.27	1	21 35 57 64	8.4 7.9 <0.2 <0.2		R60.5
Germany, 1974 (Kormoran)	WP	1.6	0.27	1	22	14		R60.5

CROP Country, year (Variety)		Application			Day	Residues, mg/kg [1]		Ref.
		kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
					35 62 70	7.3 <0.2 <0.2		
Germany, 1974 (Diplomat)	WP	1.6	0.27	1	25 35 43 80	9.6 2.4 <0.2 <0.2		R60.5
Germany, 1985	WP	1.6	0.4	2	66	3.0	<0.04	R60.6
Germany, 1985	WP	1.6	0.4	2	63	2.8	<0.04	R60.6
Germany, 1985	WP	1.6	0.4	2	63	2.4	<0.04	R60.6
Germany, 1985	WP	1.6	0.4	2	62	4.6	<0.04	R60.6
Germany, 1985 (Kanzler)	SC	1.6	0.8	2	47	2.5		R60.7
Germany, 1986 (Kanzler)	SC	1.6	0.4	2	46	8.9		R60.7
Germany, 1986 (Diplomat)	SC	1.6	0.4	2	52	2.9		R60.7
Germany, 1986 (Ralle)	SC	1.6	0.4	2	43	3.1		R60.7
Germany, 1986 (Okapi)	SC	1.6	0.4	2	56	6.5		R60.7
Netherlands, 1987 (Obelisk)	SC	3.2	0.64	2	60	2.0		R60.8
Netherlands, 1987 (Okapie)	SC	3.2	0.64	2	60	1.1		R60.8
Netherlands, 1987 (Obelisk)	SC	3.2	0.64	2	60	7.2		R60.8
Netherlands, 1987 (Okapie)	SC	3.2	0.64	2	60	0.9		R60.8
Netherlands, 1987 (Arminda)	WG	1.6	0.26	2	64	0.29	<0.002	PH8727
Netherlands, 1988 (Obelisk)	SC WG SC	1.5 1.5 1.5	0.25 0.25 0.25	2 2 2	68 68 68	1.0 3.0 0.4	<0.01 <0.01 0.02	PH8839
UK, 1990 (Haven)	WP	1.6	0.64	3 2	37 50	7.7 4.2 c 1.9		R78.1
UK, 1990 (Hornet)	WP	1.6	0.64	3 2	36 50	13 7.1		R78.1
UK, 1990 (Hornet)	WP	1.6	0.64	3 2	46 56	8.2 6.7		R78.1
UK, 1990 (Apollo)	WP	1.6	0.64	3 2	47 57	3.9 3.8		R78.1

[1] c: control sample.

Table 49. Mancozeb residues (as CS_2) in cereal fodder and straw from supervised trials in the USA. Underlined residues are from treatments according to GAP. All WP.

CROP State, year	Application			Day	Residues, mg/kg [2]		Ref.
	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
BARLEY STRAW							
ND, 1985 (Morex)	1.8	3.2	3[1]	25	24	0.18	85-0272
ND, 1985 (Robust)	1.8	3.2	3[1]	25	11	0.19	85-0273
ID, 1985 (Sevin)	1.8	3.2	3[1]	20 20	29 s 1.4	0.33 s <0.01	85-0351
WA, 1985 (Sevin)	1.8	3.2	3[1]	20	5.2	0.11	85-0352
MAIZE FODDER							
GA, 1983 (F-4333)	1.8	1.5	4	10 20	co <0.03, h 0.35, p 1.4 co <0.03, h 0.31, p 0.77	h <0.02	83-0200
FL, 1983 (NK508)	1.3	0.41	11 14	25 11	co <0.03, h 0.73, p 6.3 co <0.03, h 19, p 86	co <0.02, h 0.02	83-0228
IN, 1983 (PA63709)	3.4	7.2	2	10 20	co 0.03, h 1.7, p 18 co <0.03, h	h 0.02	83-0237

CROP State, year	Application			Day	Residues, mg/kg [2]		Ref.
	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
					0.76, p 5.0		
IA, 1983 (P80)	1.7	2.6	2	11 21	co <0.03, h 8.1, p 11 co <0.03, h 4.7, p 5.2	co <0.02, p 0.02	83-0253
	3.4	5.2	2	11 21	co <0.03, h 4.3, p 3.1 co <0.03, h 4.5, p 1.6	p 0.02	
IL, 1983 (Funk G4740)	1.7	3.6	2	10 20	co 0.28, h 1.5, p 0.44 co 0.77, h 1.1, p 0.35 c p 0.10	co <0.02, h <0.02, p <0.02	83-0358
FL, 1983 (Pioneer)	1.3	0.14	16	7 14	co <0.03, h 1.1 co <0.03, h 2.8	h <0.01 h <0.01	83-0419
AR, 1985 (North Upking)	1.3	1.4	5[1]	20 29 40	p 3.9 p 2.8 p 1.4	p 0.02 p 0.01 p 0.01	85-0337
IA, 1985	1.7	0.45	4	3 7 14 39	p 13 p 5.9 p 3.6 p 1.2 c p 0.09	p 0.040 p 0.026 p 0.02 p 0.01	85-0453
WHEAT STRAW							
MN, 1975 (Era)	1.8		2[1]	28	10	0.05	75-421-02
MN, 1975 (Era)	1.8		2[1]	47	4.7	<0.02	75-467-02
MN, 1975 (Era)	1.8		2[1]	42	2.0	0.02	75-468-02
AL, 1981 (Coker 747)	1.8 1.8	3.8 3.8	2 3	28 28	10 18 c 1.4	0.034 0.045	81-0167
AL, 1981 (Coker 747)	1.8 1.8	3.8 3.8	2 3	28 28	5.3 1.2	0.01 0.01	81-0168
MN, 1981 (Era)	1.8		2	28	0.38 c 0.90	<0.01 c 0.01	81-0428
ND, 1981 (Spr/Manitou)	1.8	3.8	2	26	0.55 c 1.6	<0.01	81-0429
ND, 1981 (Rough Rider)	1.8	3.8	2	27	<0.3 c 0.45	<0.01	81-0430
SD, 1981 (Olaf)	1.8		2	24	3.8 c 0.51	0.02	81-0426
SD, 1981 (James)	1.8		2	24	4.8 c 0.45	0.01	81-0427
TN) (McNair 1003)	1.8		2	51	3.2 c 0.63	0.05	81-0212
TN, 1981 (Arthur 71)	1.8	3.8	3	42	7.7	0.05	81-0214
TX, 1988 (NK812)	1.8	3.8	3[1]	46	11	0.037	88-0105
OK, 1988 (Florida 302)	1.8	3.8	3[1]	56	0.50	0.01	88-0131
MO, 1988 (Caldwell)	1.8	0.69	3	36	2.0	0.11	88-0185

[1] aerial application.
[2] s: straw heads; co: cobs; h: husks; p: plants; c: control sample.

Table 50. Mancozeb residues (as CS_2) in legume animal feeds and miscellaneous fodder and forage crops from supervised trials in Australia, Italy, Japan and the USA. Underlined residues are from treatments according to GAP.

CROP Country (State), year (Variety)	Application	Day	Residues, mg/kg	Ref.

	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
ALMONDS HULLS								
USA (CA), 1988 (Nonpareil)	WP	5.4	0.21	3	160	3.1	0.48	89-0006
USA (CA), 1988 (Nonpareil)	WG	5.4	0.21	3	160	3.5	0.43	89-0007
USA (CA), 1988 (Thompson)	WG	5.4	0.33	3	161	3.1	0.15	89-0016
USA (CA), 1988 (Thompson)	WP	5.4	0.33	3	161	3.0	0.54	89-0017
USA (CA), 1988 (Nonpareil)	WP	5.4	0.58	3	136	3.0	0.19	89-0023
BEAN PODS AND FOLIAGE								
Australia, 1991 (Fiord)	WG	1.5		1	0 3 8 20 29	28 8.7 6.3 1.5 0.7		AUA-91-021
	WG	3.0		1	0 3 8 20 29	44 16 17 6.1 3.7		
BEAN STRAW								
Australia, 1988 (Fiord)	WP WP	2.0 4.0		4 4	64 64	1.9 7.9	0.1 0.5	3137/88/5
PEANUT HAY								
USA (GA), 1974 (Florunner)	WP	3.6	2.3	6	27	3.6	0.02	74-171-02
USA (NC), 1984 (Florigiant)	WP	2.7	1.9	4	24	13	<0.01	85-0383
USA (TX), 1984 (Florunner)	WP	2.7		5	47	1.3	<0.01	85-0452
USA (TX), 1984 (Florunner)	WP	2.7		5	48	0.43	<0.01	85-0454
PEANUT FOLIAGE								
Australia, 1992 (Virginia Bunch)	WP	1.8		6	0 7 14 21 28	13 3.6 3.3 1.8 1.9		AUK-92-008
	WP	3.5		6	0 7 14 21 28	30 8.4 8.8 5.3 4.0		
SUGAR BEET LEAVES								
Italy, 1989	WP	2.0 4.0	0.7 0.14	3 3	28 28	2.8 2.8	<0.01 <0.01	R72.4
Japan, 1991 (Mono_su-s)	WP	2.8	0.19	5	14 21 30	2.8 1.8 0.10	0.11 0.09 <0.01	3P-7-246
USA (TX), 1985 (Monohy D2)	WP	1.8	3.8	6[1]	7 10 14 21 28	9.3 6.7 5.0 2.8 1.7	0.029 0.02 0.01 <0.01 <0.01	85-0329
USA (MN), 1985 (KW3394)	WP	1.8	0.33	7	7 14 21	22 17 9.3 c 0.06	0.10 0.078 0.046	85-0499
USA (MN), 1985 (KW3394)	WP	1.8	0.33	7	14	11	0.042	85-0515

Animal transfer studies

Animal transfer studies on lactating dairy cows and laying hens were made available to the Meeting.

Cows. Dithiocarbamate and ETU residues were measured in the milk and tissues of lactating Holstein cows fed with aged mancozeb residues incorporated in the feed in a US study in 1985 (Predmore and Shaffer, 1986).

Groups of 4 cows were fed 5 and 15 ppm and 3 cows were fed 45 ppm of aged mancozeb residues in the diet for 28 days. Milk was collected in the

morning and evening and composited daily for analysis. On day 29 all cows but one from each group were slaughtered for tissue and organ collection. The remaining one from each group was placed on a residue-free diet and slaughtered on day 36.

Animals weighed 410-610 kg and consumed 19 kg of feed each per day; all animals gained weight during the study. Mean milk production was 13-26 kg/cow/day. The mancozeb dose was regulated by including a portion of finely ground alfalfa containing aged mancozeb residues. Analysis of the treated alfalfa at the beginning and end of the study gave 375 and 324 mg/kg mancozeb equivalents and 1.1 and 0.81 mg/kg ETU respectively. ETU was not detected in other components of the diet, but dithiocarbamates at less than 1 mg/kg were present in some other items.

Dithiocarbamates were not detected in the milk from any group (<0.04 mg/kg as CS_2). ETU residues were not detected (<0.01 mg/kg) in milk from the 45 ppm feeding group; milk from the other groups was not analysed for ETU.

Dithiocarbamate and ETU residues in the tissues are shown in Tables 51 and 52 respectively.

The levels of dithiocarbamates (3 mg/kg) in the thyroids of the cows from the two lower feeding groups after 7 days on residue-free feed are not readily explained. The residues were much higher than those in both the thyroid from the high-dose cow taken at the same time and the thyroids of all the animals slaughtered at the end of 28 days of mancozeb intake.

ETU was detected in the thyroids of all the animals, with the highest mancozeb doses causing the highest levels. Residues in the thyroids decreased during the 7 days on residue-free feed. ETU was not detected in the fat from the highest dose group; it was present in muscle, heart, liver and kidney samples from the highest feeding group on day 29, but disappeared after 7 days on the residue-free diet.

Table 51. Dithiocarbamate residues in dairy cows on diets containing 5, 15 and 45 ppm aged mancozeb residues for 28 days (Predmore and Shaffer, 1986). Animals slaughtered on day 36 had been on residue-free feed since day 28.

Tissue/ organ	Dithiocarbamate residues, mg/kg as CS_2 [1, 2, 3]					
	5 ppm feed		15 ppm feed		45 ppm feed	
	Day 29	Day 36	Day 29	Day 36	Day 29	Day 36
Muscle	-	-	-	-	<0.02 (6)	-
Heart	-	-	-	-	<0.02 (2)	-
Liver	-	-	0.10, 0.10 0.03	-	0.07, 0.12	0.03
Thyroid	0.21, 0,22 0.16	3.3 [2.9]	<0.14, 0.22, 0.16	2.6 [2.8]	0.24, 0.21	0.44 [0.24]
Kidney	-	-	-	-	0.04, 0.04	-
Fat	-	-	-	-	0.04, 0.06 0.04, 0.04 0.04, 0.04	-

[1] Numbers in parentheses are numbers of samples.
[2] Residues in square brackets are independent re-analyses.
[3] - : no analysis.

Table 52. Ethylenethiourea residues in dairy cows on diets containing 5, 15 and 45 ppm aged mancozeb residues for 28 days (Predmore and Shaffer, 1986). Animals slaughtered on day 36 had been on residue-free feed since day 28.

Tissue/ organ	ETU residues, mg/kg [1, 2]

	5 ppm feed		15 ppm feed		45 ppm feed	
	Day 29	Day 36	Day 29	Day 36	Day 29	Day 36
Muscle	-	-	<0.01 (9)	<0.01 (3)	0.01, 0.028 0.01, 0.025 0.034, <0.01	<0.01 (3)
Heart	<0.01 (3)	<0.01	<0.01, <0.01, 0.013	<0.01	0.022, 0.028	<0.01
Liver	-	-	<0.02 (3)	-	0.031, 0.039	-
Thyroid	0.17, 0.23, 0.20	0.089	0.45, 0.68, 0.21	0.26	1.0, 2.7	0.032
Kidney	-	-	<0.01 (3)	<0.01	0.018, 0.038	<0.01
Fat	-	-	-	-	<0.01 (6)	-

[1] Numbers in parentheses are numbers of samples.
[2] - : no analysis.

Hens. Dithiocarbamate and ETU residues were measured in the eggs and tissues of laying White Leghorn hens fed with aged mancozeb residues incorporated in the feed in a US study in 1985 (Jameson and Shaffer, 1986).

Groups of 10 laying hens were fed nominal 5, 15 and 50 ppm levels of aged mancozeb residues in the diet for 28 days. Eggs were collected each day for analysis. On day 29 six hens from each group were slaughtered for tissue and organ collection. The remaining hens from each group were placed on a residue-free diet and slaughtered on days 36 and 43.

Birds consumed 130 g feed each per day; they lost weight (20-70 g, controls 30 g) during the study, probably because of the low energy ration. Egg production per day was 78-89%.

The mancozeb dose was regulated by mixing a portion of finely ground alfalfa containing aged mancozeb residues with a commercial laying mash, a pellet binder and other alfalfa meal to produce pellets. Pellet analysis during the study gave <0.2, 4.1-4.3, 12-17 and 39-45 mg/kg mancozeb equivalents and <0.04, 0.07-0.08, 0.10-0.29, and 0.57-0.81 mg/kg ETU for the control and three treatment levels.

Dithiocarbamates were not detected in the eggs from any group (<0.04 mg/kg as CS_2). ETU residues were not detected (<0.04 mg/kg) in eggs from the 5 and 15 ppm feeding groups; eggs from the 50 ppm feeding group were re-analysed with a lower detection limit and ETU residues were not detected (<0.01 mg/kg) in eggs collected on days 2, 6 and 13, but were detected on days 20 (0.013 mg/kg) and 27 (0.017 mg/kg).

Dithiocarbamate and ETU residues in the tissues and organs are shown in Tables 53 and 54 respectively.

Dithiocarbamate residues (CS_2-generating) were detected in the fat of controls as well as treated birds. The reason for this is not clear. Chicken fat from other sources also yielded CS_2 residues when analysed. In the metabolism study with ^{14}C-labelled mancozeb on laying hens by Jameson (1985) levels of ^{14}C in the fat were lower than in any other tissue suggesting that dithiocarbamates are not deposited in the fat.

ETU residues were not detected in the tissues or organs but were detected in the excreta at levels related to feed levels.

Table 53. Dithiocarbamate residues in laying hens on diets containing 5, 15 and 45 ppm aged mancozeb residues for 28 days (Jameson and Shaffer, 1986). Birds slaughtered on day 36 or 43 had been on residue-free feed since day 28.

Tissue/ organ	Dithiocarbamate residues, mg/kg as CS_2 [1]

	Control		5 ppm feed		15 ppm feed		45 ppm feed	
	Day 29	Day 36 or [43]	Day 29	Day 36 or [43]	Day 29	Day 36 or [43]	Day 29	Day 36 or [43]
Muscle	<0.02 0.03	0.03	<0.02 0.06	0.03	0.03 0.08	<0.02	<0.02 0.09	<0.02
Liver	<0.02	-	<0.02	-	0.03	<0.02	0.03	-
Heart	<0.1	-	0.17	-	0.17	-	<0.1	-
Gizzard	<0.04	<0.04	<0.04	<0.04	0.10	<0.04	0.49	<0.04
Kidney	<0.2	-	<0.2	-	<0.2	-	<0.2	-
Fat	0.25	0.36 [0.19]	0.33	0.24 [0.38]	0.62	0.56 [0.43]	1.6	0.29 [0.24]

[1] - : no analysis.

Table 54. Ethylenethiourea residues in laying hens on diets containing 5, 15 and 45 ppm aged mancozeb residues for 28 days (Jameson and Shaffer, 1986). Birds slaughtered on day 36 had been on residue-free feed since day 28.

Tissue/ organ	ETU residues, mg/kg [1]					
	Control		5 ppm feed	15 ppm feed	45 ppm feed	
	Day 29	Day 36	Day 29	Day 29	Day 29	Day 36
Muscle	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
Liver	<0.02	-	<0.02	<0.02	<0.02	-
Heart	<0.04	-	<0.04	<0.04	<0.04	-
Gizzard	<0.04	-	<0.04	<0.04	<0.04	-
Kidney	<0.08	-	<0.08	<0.08	<0.08	-
Fat	<0.04	-	<0.04	<0.04	<0.04	-

[1] - : no analysis.

FATE OF RESIDUES

In animals

Metabolism studies on lactating goats and laying hens were made available to the Meeting.

Goats. Tissue, milk and excreta residues were measured in six lactating goats (weighing 52-60 kg each) dosed for 7 days by capsule with radiolabelled mancozeb ([^{14}C]ethylenediamine) equivalent to 3, 14 and 36 ppm mancozeb in the feed (Schweitzer, 1986a; Predmore, 1985). Feed consumption was 2 kg/day. Milk was collected each day; animals were slaughtered on day 8 for tissue collection.

The concentration of ^{14}C in the milk reached a steady level by day 3 at all dosing rates.

Most of the ^{14}C (94-97% of that recovered) was excreted in the faeces and urine. Excretion levels reached a plateau by day 2. The distribution of the ^{14}C is shown in Table 55.

Table 55. Distribution of ^{14}C in lactating goats fed radiolabelled mancozeb ([^{14}C]-ethylenediamine) at 3, 14 and 36 ppm in the feed for 7 days before slaughter (Schweitzer, 1986a; Predmore, 1985).

Component	^{14}C as % of administered dose		
	3 ppm in feed	14 ppm in feed	36 ppm in feed
Faeces	50	47	41
Urine	31	32	34
Milk	0.17	0.70	1.5
Muscle	1.1	0.60	1.2
Fat	0.33	0.08	0.16

Component	^{14}C as % of administered dose		
	3 ppm in feed	14 ppm in feed	36 ppm in feed
Heart	0.02	0.02	0.03
Kidney	0.07	0.04	0.09
Liver	1.7	0.85	0.99
Gall bladder contents	0.07	0.01	0.01
Blood	0.31	0.23	0.37

Tissue concentrations of ^{14}C were higher in liver (0.82, 2.1, 6.2 mg/kg mancozeb equivalents) and kidney (0.21, 0.58, 2.8 mg/kg) than in the other tissues. Schweitzer (1986b) examined the distribution of the ^{14}C among the biochemical fractions of the kidney and liver (Table 56). The majority of the ^{14}C was incorporated into natural products.

The metabolites identified in the kidneys are listed in Table 57. About 30-40% of the metabolites (10-14% of the ^{14}C dose) were not identified.

Table 56. Distribution of ^{14}C among biochemical fractions in the kidney and liver from lactating goats fed radiolabelled mancozeb ([^{14}C]ethylenediamine) at 3, 14 and 36 ppm in the feed for 7 days before slaughter (Schweitzer, 1986b).

Biochemical fraction	^{14}C as % of total ^{14}C in the organ

	Kidney	Liver
Lipids	4-8%	1.7-9%
Glycogen	4-8%	1.8-3.3%
Creatines	6-10%	6-16%
Metabolites (Table 57)	29-37%	32-36%
Bound	34-47%	45-51%
Bound, released by protease	27-31%	32-39%
Unextractable	6-15%	10-14%

Table 57. Metabolites identified in kidneys from goats fed radiolabelled mancozeb ([^{14}C]ethylenediamine) at 3, 14 and 36 ppm in the feed for 7 days before slaughter (Schweitzer, 1986b).

Metabolite	Mancozeb metabolites, mg/kg		
	3 ppm	14 ppm	36 ppm
Glycine	0.004	0.013	0.068
N-formylglycine	0.002	0.005	0.038
Ethylenediamine (EDA)	0.001	0.002	-
N-acetylethylenediamine	0.001	0.009	0.017
Ethyleneurea (EU)	0.002	0.004	0.014
Ethylenethiourea (ETU)	0.001	0.003	0.031
Hydantoin	0.001	0.003	0.006
5,6-dihydro-3*H*-imidazo[2,1-*c*][1,2,4]dithiazole-3-thione (DIDT) or Ethylenebisisothiocyanate sulphide (EBIS)	0.001	0.001	0.005

Hens. Tissue, egg and excreta residues were measured in groups of 5 laying hens, each bird weighing 1.02-1.37 kg, dosed orally for 7 days by capsule with radiolabelled mancozeb ([^{14}C]ethylenediamine) equivalent to 3, 14 or 36 ppm mancozeb in the feed (Smith, 1986a; Jameson, 1985). The feed intake was 88-96 g/bird/day. Eggs and excreta were collected throughout, and the birds were slaughtered 24 hours after the final dose for tissue collection.

Most of the ^{14}C (and over 99% of that recovered) was excreted in the faeces. Its distribution is shown in Table 58.

Radioactivity was higher in the liver (0.097, 0.79 and 1.9 mg/kg expressed as mancozeb) and kidney (0.15, 0.75 and 2.0 mg/kg) than in the other tissues.

Residue levels in whole eggs were still increasing at the end of the dosing period, but declined rapidly from a group of hens in which dosing was discontinued.

The metabolites identified in the eggs and tissues from the 36 ppm group are listed in Table 59. Ethyleneurea was the main identified metabolite (0.02-0.06 mg/kg as mancozeb equivalents).

Tissues and eggs from the highest dosing group were also analysed chemically for dithiocarbamates and ETU. The levels of dithiocarbamates expressed as CS_2 were muscle 0.02-0.04 mg/kg, liver 0.09 mg/kg, gizzard 0.08 mg/kg, kidney 0.08 mg/kg, fat 0.07 mg/kg and eggs 0.007-0.02 mg/kg. At the highest dosing rate (36 ppm in the feed) ETU levels were at or below the limit of detection (0.007 mg/kg) in the tissues, while the level in eggs was 0.06 mg/kg. The level in eggs dropped below the limit of detection in four days when dosing ceased. ETU was not detected (<0.007 mg/kg) in

eggs from the lower dosing groups.

Bound ^{14}C was released by protease or acid hydrolysis and further investigated (Smith, 1986b). The major components identified in all the tissues and eggs were ethylenediamine and glycine, together constituting 27-42% of the bound activity in eggs, muscle and liver. ETU accounted for less than 1%.

Table 58. Distribution of ^{14}C in tissues, eggs and excreta of laying hens fed radiolabelled mancozeb ([^{14}C]ethylenediamine) at 3, 14 and 36 ppm in the feed for 7 days before slaughter (Jameson, 1985).

Component	^{14}C as % of administered dose		
	3 ppm in feed	14 ppm in feed	36 ppm in feed
Excreta	83	82	87
Whole egg	0.19	0.38	0.46
Egg yolk	0.033	-	-
Egg white	0.092	-	-
Muscle	0.039	0.048	0.068
Fat	0.0016	0.0055	0.0048
Heart	0.0051	0.0055	0.0063
Kidney	0.047	0.052	0.055
Liver	0.089	0.13	0.14
Gizzard	0.045	0.084	0.076

Table 59. Metabolites in eggs and tissues of laying hens fed radiolabelled mancozeb ([^{14}C]ethylenediamine) at 36 ppm in the feed for 7 days before slaughter (Smith, 1986a).

Metabolite	Metabolite expressed as % of ^{14}C in the eggs or tissue			
	Eggs	Breast muscle	Thigh muscle	Liver
Residue - not extractable	44	35	39	49
EBIS (DIDT)*	0.12			
Ethylenethiourea (ETU)	6.8			<0.3
Ethyleneurea (EU)	20	36	14	4.5
Glycine and ethylenediamine	1.5		2.8	4.1
N-acetylethylenediamine	3.1		1.0	0.4
Hydantoin and imidazoline			2.6	2.2

* See Table 57 for chemical name

In plants

Metabolism studies on tomatoes, soya beans, sugar beet and wheat were made available to the Meeting.

Tomatoes. A tomato crop was treated with radiolabelled mancozeb ([^{14}C]ethylenediamine) at 2.7 kg ai/ha on nine occasions at approximately weekly intervals, and ripe tomatoes were harvested 5 days after the final treatment (Mazza and Schweitzer, 1989). The distribution of the radiolabel in the ripe tomato fractions was protein 14%, soluble carbohydrate 33%, lipids 14%, ethyleneurea 13% and bound residue 9%. A high proportion of the label had been incorporated into the carbon pool and appeared in a range of natural products. The concentration of ethyleneurea was 0.085 mg/kg.

The tomatoes were analysed for residues of mancozeb (0.02 mg/kg as CS_2) and ETU (not detectable at 0.01 mg/kg) using regulatory methods.

Soya beans. A crop was treated twice with radiolabelled mancozeb ([^{14}C]ethylenediamine) at 3.4 kg ai/ha, 69 and 56 days prior to harvest (Yeh, 1985). The beans were analysed for residues of mancozeb (not detectable at 0.04 mg/kg as CS_2) and ETU (not detectable at 0.014 mg/kg) using regulatory methods. In lyophilised pods ETU was not detectable (<0.01 mg/kg) while dithiocarbamates by analysis and calculated from ^{14}C were 0.75 and 7.9 mg/kg respectively (expressed as CS_2). Dithiocarbamate concentrations, expressed as CS_2, calculated from ^{14}C levels were beans 1.3 mg/kg, pods 3.5 mg/kg and stems 1.6 mg/kg (Satterthwaite, 1985).

Pods and beans were extracted with a methanol/chloroform/water mixture for examination for possible metabolites. None of the normal range of expected metabolites was detected in the extract of the beans (53% of the ^{14}C was extractable). A major component constituting 82% of the extractable ^{14}C could not be identified. In the pods 36% of the total ^{14}C was extractable; the identified metabolites are shown in Table 60.

Much of the ^{14}C in the beans was distributed among protein (25%), oil (11%) and whey solubles including 6% of the protein (37%).

A further study (Yeh, 1986b) showed that 19% of the total pod ^{14}C was incorporated into lignin, and that at least 2% was incorporated into oligo-, di- and mono-saccharides. In the beans 9-16% of the ^{14}C was associated with proteins of molecular weight greater than 25,000.

The studies suggest that most of the carbon in the ethylenediamine portion of the dithiocarbamate molecule is incorporated into natural products.

Table 60. Metabolites identified in a solvent extract of soya bean pods from a crop treated 69 and 56 days prior to harvest with 3.4 kg ai/ha ^{14}C-labelled mancozeb (Yeh, 1985).

Metabolite	Metabolite ^{14}C expressed as % of extractable ^{14}C
1-(2-imidazolin-2-yl)-2-imidazolidinethione (Jaffe's base)	36
Ethyleneurea	15
Hydantoin	11
EBIS (DIDT)*	13

* See Table 57 for chemical name

Sugar beets. A crop was treated three times with radiolabelled mancozeb ([^{14}C]ethylenediamine) at 2.2 kg ai/ha, 63, 32 and 14 days prior to harvest (Yeh, 1986a). The ^{14}C was distributed 77% in the leaf and stem, and 23% in the root.

Samples of leaf + stem at harvest were analysed for dithiocarbamates and ETU. ETU was not detected (<0.007 mg/kg). The dithiocarbamate level (as CS_2) was 0.39 mg/kg by analysis, and 5 mg/kg calculated from the ^{14}C content. The method used for dithiocarbamates was Haines (1982), and for ETU Haines and Adler (1973).

Neither ETU nor dithiocarbamate was detected in the sugar beet root by analysis(<0.007 mg/kg and <0.02 mg/kg as CS_2 respectively). The total ^{14}C calculated as residues of CS_2 was 0.3 mg/kg.

The fate of the radiolabel in metabolites and natural products was investigated by TLC in an extract of leaf and stem (73% of the ^{14}C was extracted). The distribution expressed as a percentage of the total ^{14}C in the leaf and stem was simple and complex carbohydrates 7.0%, amino acids 13%, ethyleneurea 1.6%, ETU + hydantoin 0.19%, ethylenediamine +

2-imidazoline + *N*-formylethylenediamine 2.2%, 1-(2-imidazolin-2-yl)-2-imidazolidinethione 3.9% and EBIS 2.1%.

From the sugar beet root 80% of the ^{14}C was extractable with water. The distribution of the radiolabel expressed as a percentage of the total ^{14}C in the roots was sucrose 36%, amino acids 17%, proteins etc. 7%, ethyleneurea 3.2%.

Wheat. Radiolabelled mancozeb ([^{14}C]ethylenediamine) was applied three times at 2.2 kg ai/ha to a wheat crop, which was harvested 46 days after the final application (Reibach, 1986a). The total ^{14}C in the seed, chaff and straw was measured by combustion analysis and dithiocarbamate residues were measured by a CS_2 evolution method (Table 61). The levels of the parent dithiocarbamate in the grain would be expected to be low because its polymeric and insoluble nature should result in minimal absorption and translocation.

Samples were extracted with ethanol and other solvents, and further solubilised by hydrolysis with 2N hydrochloric acid. The distribution of the radiolabel among metabolites and natural products is summarized in Table 62. ETU was not detected (<0.007 mg/kg) as ^{14}C or by chemical analysis. Levels of EBIS and ethyleneurea did not exceed 0.03 mg/kg. Stronger acid hydrolysis released more ^{14}C but a large part of the label remained in an acid-resistant non-extractable material, identified as lignin (Reibach, 1986b).

Table 61. Dithiocarbamate residues in wheat components resulting from foliar application of ^{14}C-labelled mancozeb (Reibach, 1986a).

Wheat component	Dithiocarbamate residues as CS_2, mg/kg	
	Calculated from total ^{14}C	Analysis as evolved CS_2
Seed	1.3	0.02
Chaff	8.8	1.3
Straw	13.2	1.2

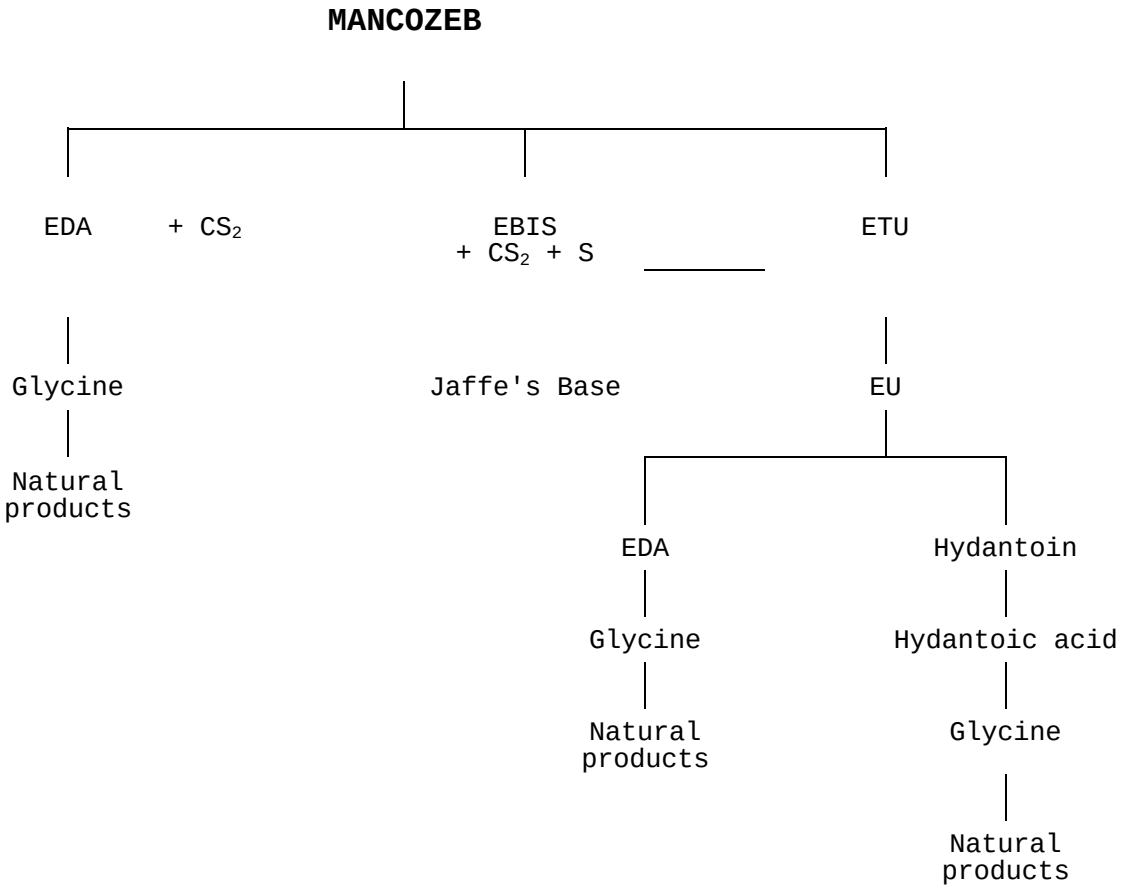
Table 62. Distribution of ^{14}C label in metabolites and natural products in wheat resulting from foliar application of ^{14}C-labelled mancozeb (Reibach, 1986a).

Metabolite	Metabolite content expressed as % of ^{14}C in the seed, chaff or straw.		
	Seed	Chaff	Straw
Sugars	32	13 (sugars + ethyleneurea)	8.2
Ethyleneurea (EU)	0.87		0.71
Amino acids	5.6	5.5	4.2
EBIS (DIDT)* + 2-imidazoline + 1-(2-imidazolin-2-yl)-2-imidazolidinethione (Jaffe's base)	1.6	2.6	2.1
Ethylenediamine (EDA)	0.84	4.1	3.1
Protein	2.5	4.2	3.7
Non-extractables	32	59	65
Solubles	68	41	35

* See Table 57 for chemical name

The metabolic pathways of mancozeb are summarized in Figure 1.

Figure 1 Mancozeb metabolism.



ETU: ethylenethiourea
EU: ethyleneurea
EBIS: 5,6-dihydro-3*H*-imidazo[2,1-*c*][1,2,4]dithiazole-3-thione (DIDT)
EDA: ethylenediamine
Jaffe's base: 1-(2-imidazolin-2-yl)-2-imidazolidinethione

In storage and processing

Processing studies were made available to the Meeting on apples, grapes, sweet corn, tomatoes, potatoes, sugar beet, barley, wheat, maize and peanuts.

Apples. Eleven applications of mancozeb (trial 85-0308, treatment $1_{\times}$: 7.2 kg ai/ha, treatment $2_{\times}$: 14.3 kg ai/ha) were made to Delicious and Macintosh apples in the USA (PA) (Ollinger *et al.*, 1986a). Apples were harvested 21 days after the final application and processed according to the scheme in Figure 2. Results are summarized in Table 63.

The washing process removed 30-50% of the mancozeb residues, and 90% of the remaining residue went with the peel fraction. ETU was generated during the heating of peels, cores and slices, and was detected in the unclarified juice and dry pomace from the $2_{\times}$ treatment. It was not detected in the other fractions or in the $1_{\times}$ treatment.

Figure 2. Processing of apples field-sprayed with mancozeb (Ollinger *et al.*, 1986a).

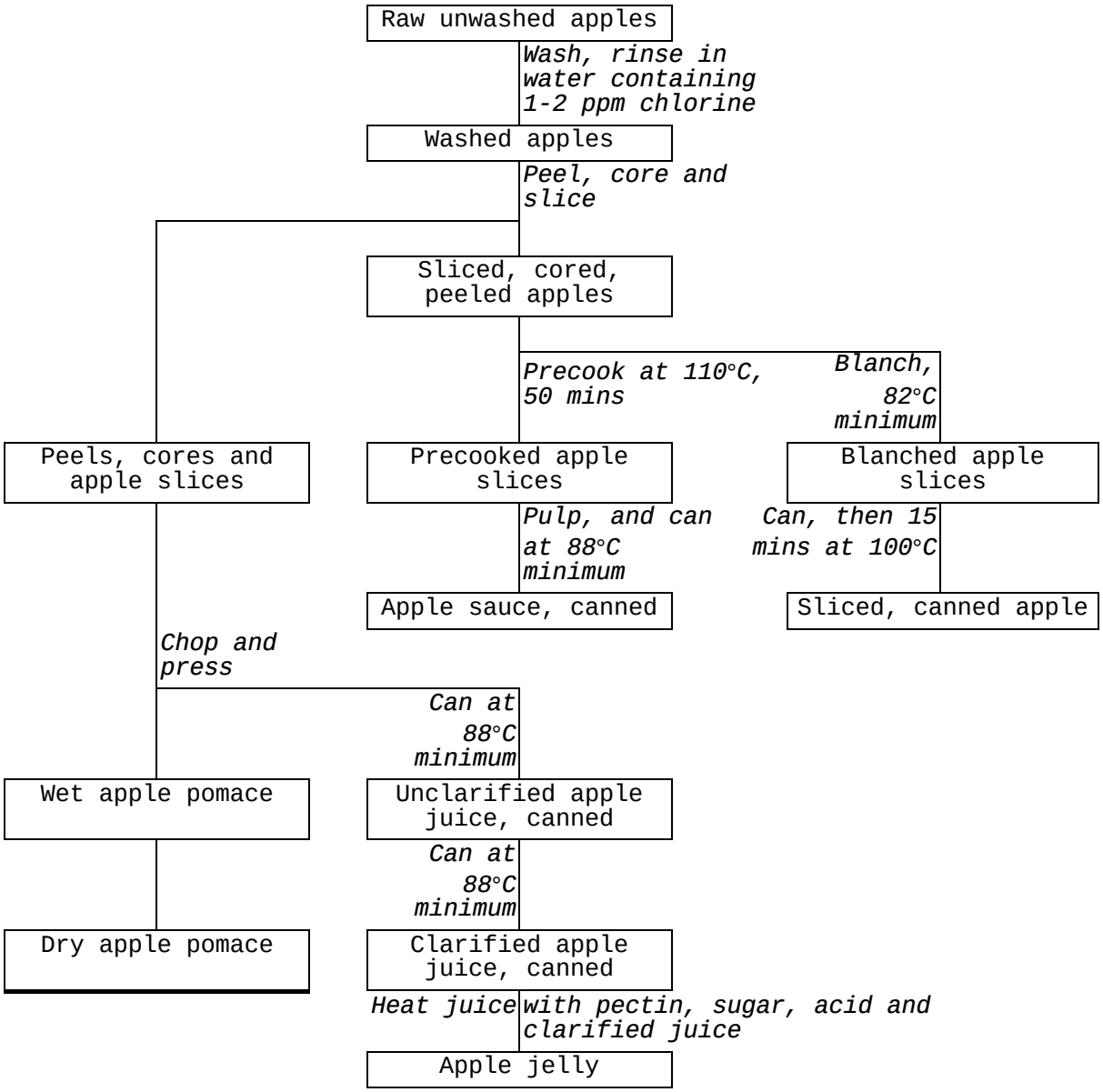


Table 63. Analysis of processed apples for dithiocarbamates and ETU (Ollinger *et al.*, 1986a). Apples had received 11 applications of mancozeb (trial 85-0308, treatment 1×: 7.2 kg ai/ha, treatment 2×: 14.3 kg ai/ha), with the final application 21 days prior to harvest. Each reported result is the mean of duplicate analyses.

Commodity	Dithiocarbamate residues, mg/kg as CS_2	ETU residues, mg/kg

	Treatment 1_X	Treatment 2_X	Treatment 1_X	Treatment 2_X
Unwashed apples	2.5	3.8	<0.03	<0.03
Washed apples	1.2	2.8	<0.03	<0.03
Sliced, cored, peeled apples	0.11	0.28	<0.03	<0.03
Peels, cores, slices before processing	5.3	5.3	<0.03	<0.03
Precooked apple slices	<0.1	<0.1	<0.03	<0.03
Apple sauce, canned	<0.1	<0.1	<0.03	<0.03
Blanched apple slices	<0.1	<0.1	<0.03	<0.03
Sliced apple, canned	<0.1	<0.1	<0.03	<0.03
Unclarified apple juice, canned	0.31	1.1	<0.03	0.04
Clarified apple juice, canned	<0.1	<0.1	<0.03	<0.03
Apple jelly	<0.1	<0.1	<0.03	<0.03
Wet apple pomace	7.6	9.7	<0.03	<0.03
Dry apple pomace	24	50	<0.03	0.10

Applications of mancozeb (in trial 84-0239, 8 at 7.2, 1 at 5.4 and 1 at 3.6 kg ai/ha; in trial 84-0262, 13 at 7.2 and 2 at 3.6 kg ai/ha; in trial 84-0468, 1 at 7.4, 4 at 7.2, 2 at 5.4 and 3 at 3.6 kg ai/ha) were made to Delicious, Winesap and Prime Gold apples in the USA (MI) (Satterthwaite, 1986n). Apples were harvested 21 days after the final application and processed on a small experimental scale. Results are summarized in Table 64.

The report makes no mention of any washing or cleaning of the apples before conversion to juice and pomace. Mancozeb residues on the surface of the apples would be expected to enter the process, and would be more likely to finish in the pomace than in the juice.

Table 64. Dithiocarbamate and ETU residues in apples, juice and pomace (Satterthwaite, 1986n).

Commodity	Dithiocarbamate residues, mg/kg as CS_2			ETU residues, mg/kg		
	Trial 84-0239	Trial 84-0262	Trial 84-0468	Trial 84-0239	Trial 84-0262	Trial 84-0468
Apples	1.1, 0.84	3.9, 3.9	4.5, 4.9	0.01, 0.015	0.01, 0.01	0.01, 0.015
Apple juice	0.29	0.55	0.44	<0.01	0.01	<0.01
Wet apple pomace	0.95	2.2	1.1	0.03	0.06	0.04
Dry apple pomace	6.7	13	6.7	0.06	0.14	0.06

Grapes. Ollinger *et al.* (1986c) treated grapes with 8 applications of mancozeb (trial 85-0353, treatment 1_X: 3 at 4.4 kg ai/ha and 5 at 2.0 kg ai/ha; treatment 2_X: 3 at 4.4 kg ai/ha and 5 at 4.0 kg ai/ha) in a processing trial in the USA. Grapes were harvested 7 days after the final application to achieve sufficiently high residues to be measured in the processed fractions. The recommended pre-harvest interval is 66 days, except in California where mancozeb cannot be applied after bloom.

Grapes were processed, one box for each treatment and process, into

juice, jelly and dried raisins (Elkins and Kim, 1986). The processes are described in Figures 3 and 4. Grapes were subjected to steam for 30 seconds and then dried in a forced air oven at 38-43°C to produce raisins. Residues of dithiocarbamate and ETU are given in Table 65.

Dithiocarbamate residue concentrations decreased through the various processing steps, except raisin production where removal of water would be expected to increase the concentration of residues. Raisins in this study were not washed; the commercial procedure is to wash the raisins, which would be likely to reduce residues. Dithiocarbamates were not detectable in clear solutions of juice or jelly.

ETU was generated in processes where dithiocarbamate residues were boiled or heated. The ETU residue level in a processed product was not related to its level in the raw commodity.

Figure 3. Processing of grapes to produce juice and pomace (Elkins and Kim, 1986).

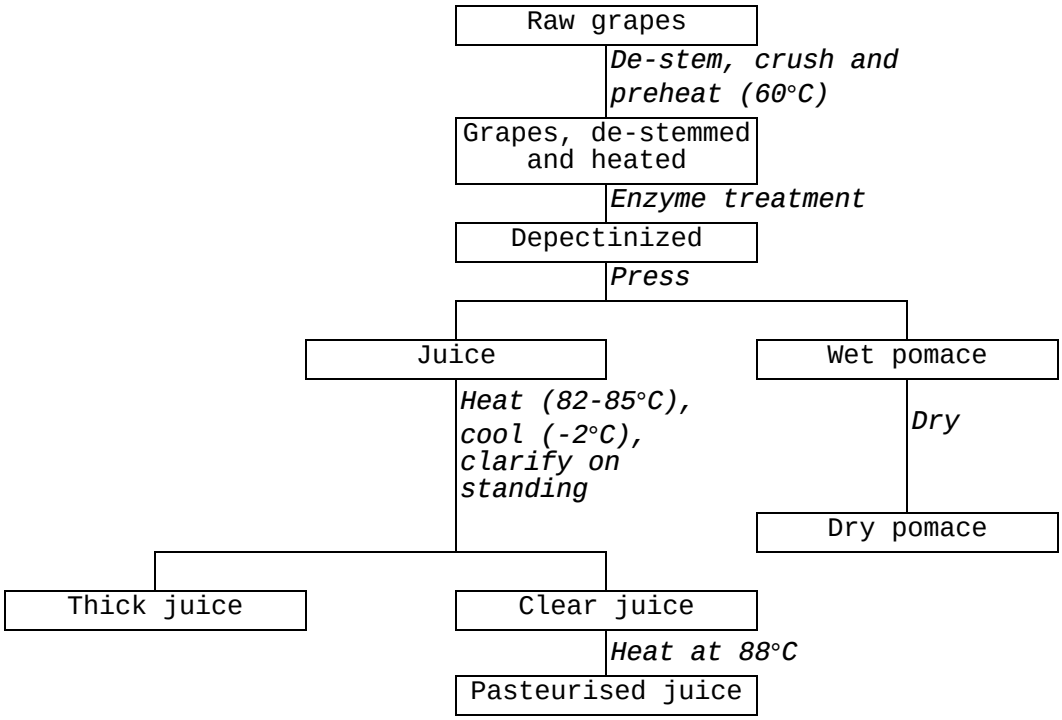


Figure 4. Processing of grapes to produce jelly (Elkins and Kim, 1986).

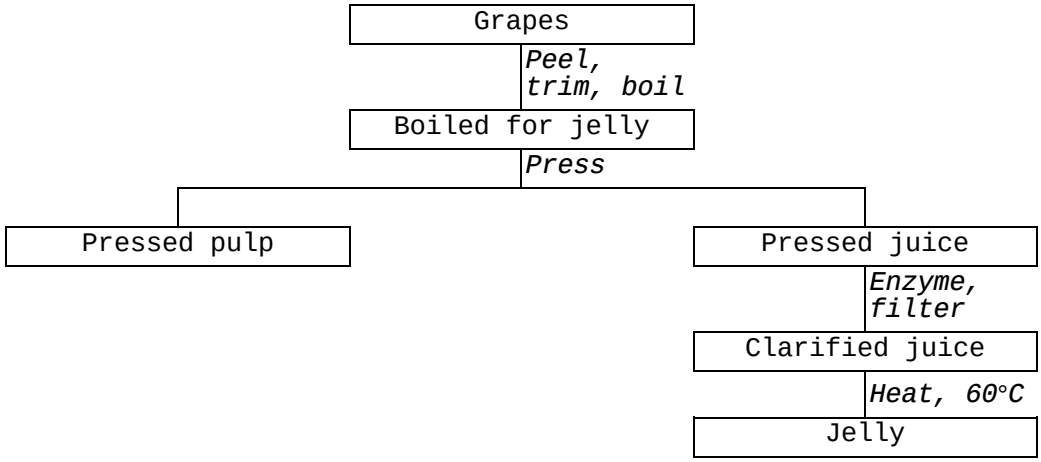


Table 65. Residues of dithiocarbamates (as CS_2) and ETU in grapes and their processed products, trial 85-0353 (Ollinger *et al.*, 1986c; Elkins and Kim, 1986). The processes are described in Figures 3 and 4. Reported results are from duplicate samples.

Commodity	Dithiocarbamate residues, as CS_2, mg/kg	ETU residues, mg/kg

	Treatment 1×	Treatment 2×	Treatment 1×	Treatment 2×
Raw grapes	21, 17	49, 36	0.01, 0.01	0.28, 0.35
De-stemmed and heated	3.9, 3.4	18, 20	0.07, 0.04	0.28, 0.33
Depectinized	3.1, 2.2	13, 13	0.04, 0.03	2.4, 2.4
Wet pomace	4.5, 5.6	9.5, 15	0.03, 0.02	0.29, 0.19
Dry pomace	12, 14	20, 18	0.20, 0.21	1.3, 0.90
Clear juice	<0.1, <0.1	<0.1, <0.1	0.19, 0.23	2.4, 2.6
Thick juice	2.4, 2.6	1.4, 1.2	0.08, 0.08	4.3, 4.3
Pasteurised juice	<0.1, <0.1	<0.1, <0.1	0.08, 0.09	0.93, 0.90
Canned juice	<0.1, <0.1	<0.1, <0.1	0.13, 0.11	1.3, 1.3
Boiled for jelly	0.84, 0.78	19, 17	0.22, 0.26	4.9, 4.2
Pressed pulp	2.2, 1.5	11, 12	0.32, 0.37	0.37, 0.29
Pressed juice	0.4, 0.5	2.2, 3.0	1.5, 1.2	2.9, 2.7
Clarified juice	<0.1, <0.1	<0.1, <0.1	0.21, 0.20	3.1, 3.0
Cooled jelly	<0.1, <0.1	<0.1, <0.1	0.71, 0.74	1.6, 1.1
Heated raisins	22, 30	34, 37	0.05, 0.05	0.09, 0.08
Dried raisins	46, 53	135, 136	0.31, 0.37	1.0, 0.92

Grapes grown in the USA (CA) for processing studies were treated once (Trial 85-0336) with mancozeb at 7.2 kg ai/ha 64 days prior to harvest (Satterthwaite, 1986f). In a second trial (85-0342), grapes were treated five times with mancozeb at 2.0 kg ai/ha (1×) or 4.0 kg ai/ha (2×) with a pre-harvest interval of 21 days. Raisins, white wine and red wine were produced from the grapes.

The grapes were dried to <16% moisture content then processed to remove chaff, stems, leaves and small fruit to produce raisins. The material removed was the raisin waste. The wine production process is shown in Figure 5. Dithiocarbamate and ETU residues in the wine, raisins and by-products from both trials are listed in Table 66.

The use patterns were not GAP and were designed to produce exaggerated residues for the processing study.

In the production of raisins some dithiocarbamate residues were lost while no ETU was generated. Dithiocarbamate residues were not found in the wine produced from the treated grapes, but ETU was generated in the process.

Figure 5. Process for wine production (Satterthwaite, 1986f). The red wine was produced by fermenting skins and juice together, with additional sugar.

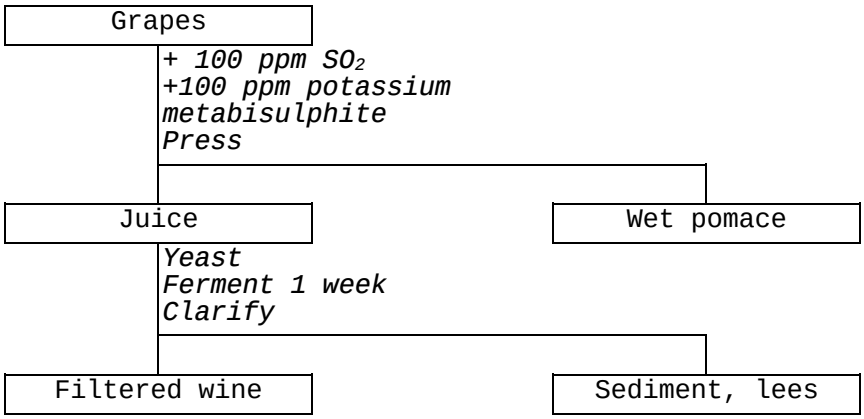


Table 66. Residues of dithiocarbamates (as CS_2) and ETU in grapes and their processed products (Satterthwaite, 1986f). The wine production process is described in Figure 5.

Commodity	Dithiocarbamate residues, mg/kg as CS_2			ETU residues, mg/kg		
	Trial 85-0336	Trial 85-0342 1×	Trial 85-0342 2×	Trial 85-0336	Trial 85-0342 1×	Trial 85-0342 2×
Fruit	1.6	3.6	9.0	<0.01	<0.01	0.02
Raisins	0.90	0.78	2.8	<0.01	<0.01	<0.01
Raisin waste	7.9	5.0	1.8	0.04	0.05	0.17
RED WINE						
Unfiltered juice	1.5	6.7	21	<0.01	<0.01	0.03
Pomace	0.29	0.84	3.7	<0.01	<0.01	<0.01
Lees		8.4	12		0.06	0.57
Red wine filtered		<0.03	0.06		0.08	0.64
WHITE WINE						
Unfiltered juice	1.2	4.5	5.3	0.01	0.01	0.02
Pomace	0.49	1.4	3.8	<0.01	<0.01	0.01
Lees	7.9	8.4	33	0.07	0.16	1.0
White wine filtered	<0.03	<0.03	<0.03	0.09	0.22	0.79

Mancozeb was applied three times at 3.6 kg ai/ha to grapes for a processing study in the USA (CA) (Satterthwaite, 1990a). The grapes were harvested 82 days after the final application and processed into raisins and juice, which were analysed for residues of dithiocarbamates and ETU (Table 67). Raisins were produced by drying the Thompson seedless grapes in the sun for 13 days, when the moisture content was less than 16%. They were then cleaned and sized. The initial analysis of the grapes for ETU showed 0.23 mg/kg, which appeared anomalous in the light of previous experience. Re-analysis showed 0.061 mg/kg.

Table 67. Residues of dithiocarbamates (as CS_2) and ETU in grapes, raisins and juice (Satterthwaite, 1990a).

Commodity	Mancozeb residues, mg/kg as CS_2	ETU residues, mg/kg
Grapes	0.23	0.061
Raisins	0.52, 0.16, 0.19, 0.24	<0.01 (4)
Raisin waste	6.9	0.20
Juice	0.28	0.43, 0.046
Fermented wet pomace	0.26	0.046
Dry pomace	0.21	0.022

Sweet corn. Sweet corn was treated with mancozeb on 7 occasions at 1.3 kg ai/ha or 6.7 kg ai/ha, and harvested 7 days after the final application (trial 87-0328) in the USA (PA) (Schweitzer, 1989b). The sweet corn was put through a small-scale cannery process. Residues of dithiocarbamates and ETU were measured in the sweet corn and its products (Table 68).

Table 68. Residues of dithiocarbamates (as CS_2) and ETU in sweet corn and processed products (Schweitzer, 1989b).

Commodity	Dithiocarbamate residues, mg/kg as CS_2		ETU residues, mg/kg	
	Applicn. rate 1.3 kg ai/ha	Applicn. rate 6.7 kg ai/ha	Applicn. rate 1.3 kg ai/ha	Applicn. rate 6.7 kg ai/ha
Whole ear	0.21	0.90	<0.01	0.022
Cob + kernel	<0.03	0.03	<0.01	0.021
Husk	1.3	6.7	0.010	0.18
Frozen corn	<0.03	0.05	<0.01	<0.01
Canned corn	<0.03	<0.03	<0.01	0.014
Cannery waste	0.39	3.0	0.015	0.11

Tomatoes. Five applications of mancozeb (trial 85-0378, treatment 1_X: 2.7 kg ai/ha; treatment 2_X: 5.4 kg ai/ha) were made to crops of tomatoes in the USA (PA) for processing studies (Ollinger *et al.*, 1986b). Tomatoes were harvested 5 days after the final application and processed according to the scheme in Figure 6. Results are summarized in Table 69.

Mancozeb residues (50% or more) were removed from the tomatoes during the washing process. ETU was generated during some of the cooking processes. The products with the highest levels of ETU were puree, paste and ketchup. Levels of ETU increased during the heat treatment of canned juice and canned puree.

Figure 6. Processing of tomatoes field-sprayed with mancozeb (Ollinger *et al.*, 1986b).

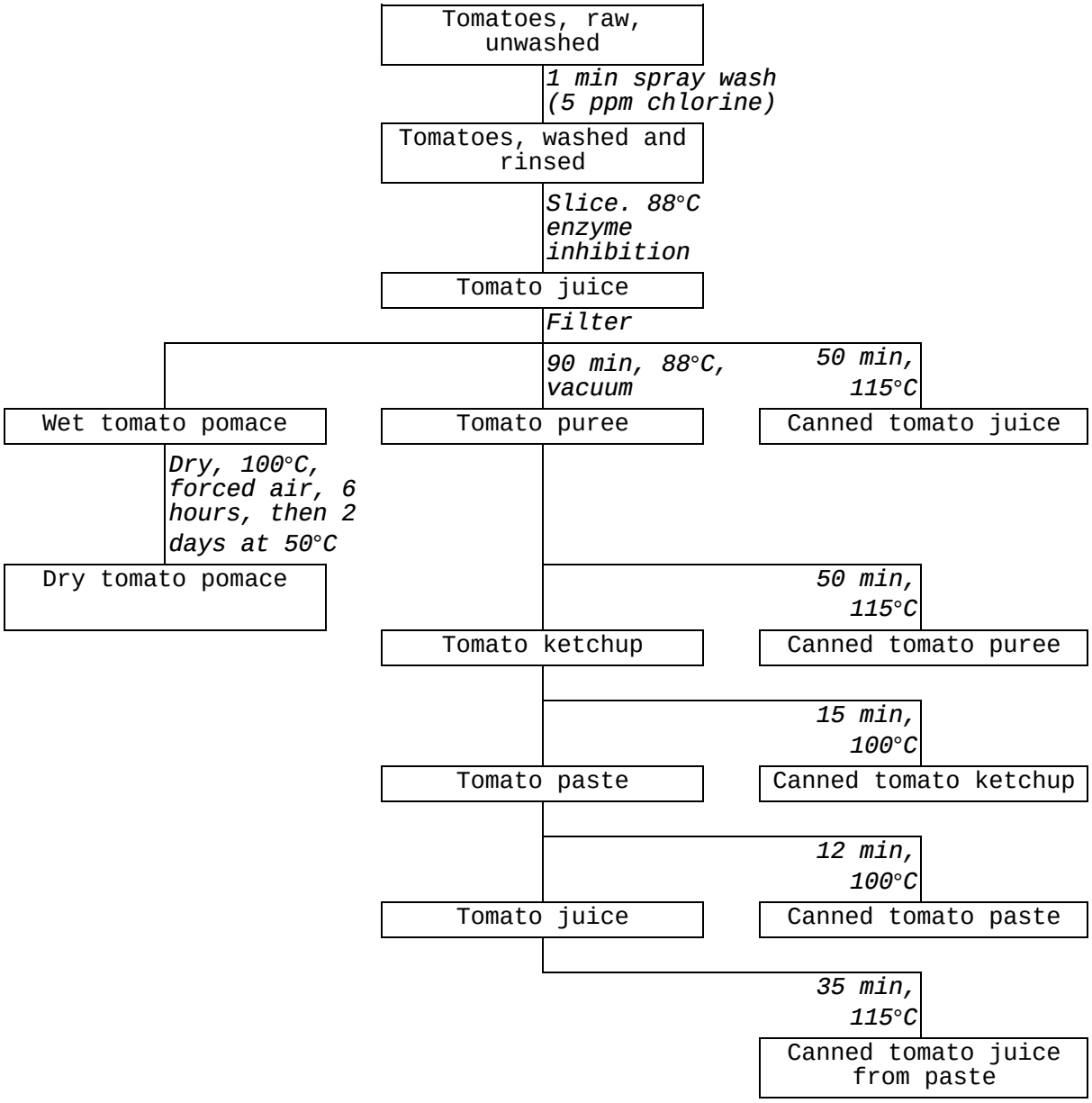


Table 69. Dithiocarbamates ETU in processed tomatoes (Ollinger *et al.*, 1986b). Tomatoes had received 5 applications of mancozeb (trial 85-0378, treatment 1_X: 2.7 kg ai/ha; treatment 2_X: 5.4 kg ai/ha), with the final application 5 days prior to harvest. Each reported result is the mean of duplicate analyses.

Commodity	Dithiocarbamate residues, mg/kg as CS_2	ETU residues, mg/kg

	Treatment 1×	Treatment 2×	Treatment 1×	Treatment 2×
Raw unwashed tomatoes	0.2	0.5	<0.01	<0.01
Washed rinsed tomatoes	<0.1	0.2	<0.01	0.02
Tomato juice	<0.1	0.1	0.015	0.05
Canned tomato juice	<0.1	<0.1	0.02	0.09
Wet tomato pomace	<0.1	<0.1	<0.01	0.01
Dry tomato pomace	<0.1	<0.1	0.03	0.05
Tomato puree	<0.1	0.2	0.07	0.12
Canned tomato puree	<0.1	0.1	0.08	0.25
Canned tomato ketchup	<0.1	0.1	0.04	0.19
Canned tomato paste	0.1	0.4	0.14	0.25
Tomato juice from paste	<0.1	0.1	0.03	0.07
Canned juice from paste	<0.1	<0.1	0.03	0.04

Tomatoes were commercially processed in 80 tonne lots to determine the fate of field-applied mancozeb (Schweitzer, 1988). The application rate was 2.7 kg ai/ha on each of 5 (trial 87-0306) or 6 (trial 87-0305) occasions, and the interval between final application and harvest was 5 days for trial 87-0305 and 11 days for trial 87-0306. Residues in the processed fractions are summarized in Table 70.

Washing removed almost all of the mancozeb residues. In the commercial procedure the tomatoes are immersed in troughs of continuously replaced water for 5-10 minutes, and are sprayed with fresh water on exit. In the previous small-scale experiment (Ollinger *et al.*, 1986b) with a 30-second water spray, only about 50-60% of the mancozeb residue was removed.

The removal of most of the dithiocarbamate before heating or cooking steps restricts the capacity to form ETU. Levels of ETU in the end products were substantially lower than in the earlier study (Ollinger *et al.*, 1986b).

Table 70. Dithiocarbamate and ETU residues in commercially processed tomatoes (Schweitzer, 1988). Numbers in parentheses are numbers of samples.

Commodity	Dithiocarbamate residues, mg/kg as CS_2		ETU residues, mg/kg	
	Trial 87-0305	Trial 87-0306	Trial 87-0305	Trial 87-0306
Unwashed tomatoes	0.41, 0.20, 0.47, 0.39, 0.42, 0.51	0.18, 0.16, 0.18, 0.73, 0.35, 0.39	<0.01 (6)	<0.01 (6)
Washed tomatoes	0.03, <0.03 (23)	0.03, 0.05, 0.04, <0.03 (21)	0.01 (10), <0.01 (13), 0.015	<0.01 (24)
Hot break juice	<0.03 (6)	<0.03 (6)	0.031, 0.023, 0.034, 0.022, 0.016, 0.016	0.020, 0.020, 0.016, 0.022, 0.025, 0.039
Wet pomace	<0.03 (6)	<0.03 (6)	<0.01 (5), 0.016	<0.01 (6)
Concentrate	<0.03 (6)	<0.03 (6)	0.027, 0.037, 0.044, 0.033, 0.049, 0.035	0.049, 0.038, 0.025, 0.042, <0.01 (2)
Tomato sauce	0.04, 0.03, <0.03		<0.01 (3)	
Tomato ketchup		<0.03, 0.03, 0.03		0.016 (2), <0.01 (3), 0.01

Potatoes. Harvested potatoes (23 kg) were sprayed in the laboratory with mancozeb at a rate estimated to produce a mancozeb residue of 1 mg/kg , and then sent for processing (Ollinger *et al.*, 1986d). Processing details are summarized in Figure 7. Dithiocarbamate and ETU residues in each of the processed potato fractions are given in Table 71.

Dithiocarbamate residues were essentially only on the peel of the potatoes. Some ETU was formed during the baking of peel containing dithiocarbamate residues.

Figure 7. Processing of potatoes sprayed with mancozeb to produce a nominal 1 mg/kg mancozeb residue. (Ollinger *et al.*, 1986d).

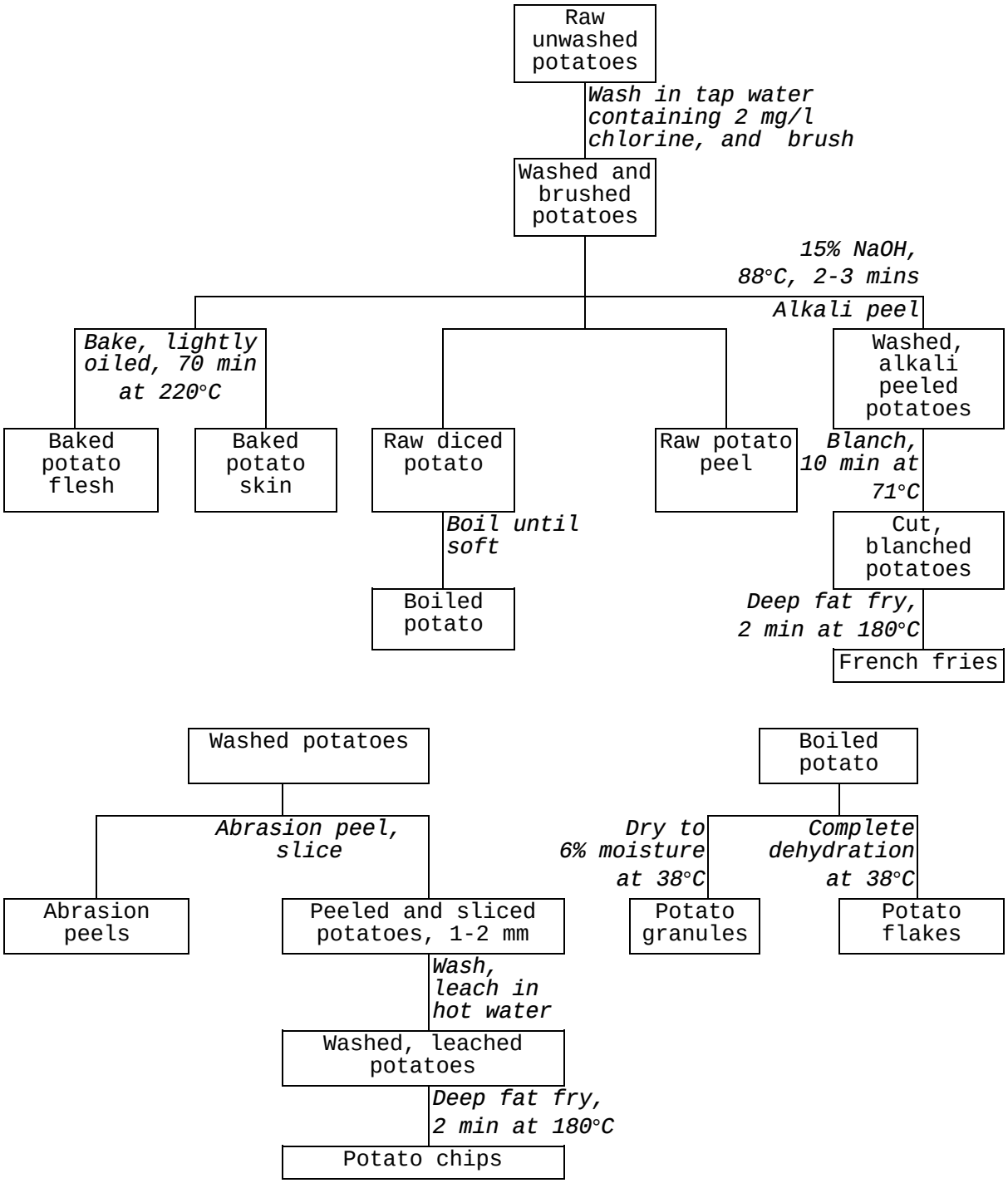


Table 71. Analysis of processed fractions from potatoes sprayed with mancozeb to produce a nominal 1 mg/kg mancozeb residue. (Ollinger *et al.*, 1986d). Each reported result is the mean of duplicate analyses.

Commodity	Dithiocarbamate residues, mg/kg as CS_2	ETU residues, mg/kg
Unwashed potatoes	0.32	<0.01
Washed and brushed potatoes	<0.06	<0.01
Baked potato pulp	<0.06	0.013
Baked potato peel	<0.06	0.04
Raw potato peels	0.53	<0.01
Raw, diced potato	<0.06	<0.01
Boiled potato	<0.06	<0.01
Washed, alkali-peeled potatoes	<0.06	<0.01
Cut, blanched potatoes	<0.06	<0.01
French fries	<0.06	<0.01
Abrasion peels	0.66	<0.01
Peeled and sliced potatoes	<0.06	<0.01
Washed, leached potatoes	<0.06	<0.01
Potato chips	<0.06	<0.01
Potato granules	<0.06	<0.01
Potato flakes	<0.06	0.01

Mancozeb was foliar-applied on two occasions to potato crops in the USA at 1.8 and 9.0 kg ai/ha, at a site in Ohio, to provide potatoes for a processing study (Schweitzer, 1989c). The potatoes were processed (approximately 5 kg each process) according to Figure 7 for potato chips, granules and flakes. Residues are shown in Table 72.

Mancozeb is not systemic, so residues in the tubers from foliar application would be expected to be a sporadic occurrence from soil contamination or exposure of tubers at the soil surface. During processing, where dithiocarbamate might be transferred from the peel by operations such as abrasion peeling, there would be an opportunity for the formation of ETU during cooking. The results show that residues are not generally detectable, but enough dithiocarbamate is sometimes present to generate ETU.

Table 72. Residues of dithiocarbamates and ETU in potatoes harvested 14 days after foliar applications of mancozeb and in the processed potato commodities (Schweitzer, 1989c). Each result is the mean of duplicate analyses.

Commodity	Dithiocarbamate residues, mg/kg as CS_2	ETU residues, mg/kg

	Appl. rate 1.8 kg ai/ha	Appl. rate 9.0 kg ai/ha	Appl. rate 1.8 kg ai/ha	Appl. rate 9.0 kg ai/ha
Raw unwashed potatoes	<0.1	<0.1	<0.02	<0.02
Washed, abrasion-peeled potatoes	<0.1	<0.1	<0.02	0.04
Abrasion peels	<0.1	<0.1	<0.02	0.03
Sliced, washed, leached, potatoes	<0.1	<0.1	<0.02	0.02
Potato chips	<0.1	0.16	<0.02	<0.02
Washed, hand-peeled potatoes	<0.1	<0.1	<0.02	<0.02
Peels from hand-peeling	<0.1	<0.1	<0.02	<0.02
Boiled potatoes	<0.1		<0.02	
Potato granules	<0.1	<0.1	<0.02	0.08
Potato flakes	0.36	<0.1	0.09	0.23

Sugar beet. Beets grown in the USA (MN) were treated with mancozeb (trial 85-0515) at 1.8 ($1\times$) and 7.2 ($4\times$) kg ai/ha on 7 occasions and harvested 14 days after the final application in a residue processing study (Satterthwaite, 1986k). The simulated commercial process used 140 kg of sugar beet. The first stage of the process was washing the roots. Residues are shown in Table 73.

Table 73. Dithiocarbamate and ETU residues in processed sugar beet products (Satterthwaite, 1986k). Beets were treated with mancozeb (trial 85-0515) at 1.8 ($1\times$) and 7.2 ($4\times$) kg ai/ha on 7 occasions and harvested 14 days after the final application.

Commodity	Dithiocarbamate residues, mg/kg as CS_2		ETU residues, mg/kg	
	Treatment $1\times$	Treatment $4\times$	Treatment $1\times$	Treatment $4\times$
Sugar beet root	0.14	0.16	0.018	0.025
Molasses	<0.03	<0.03	<0.01	<0.01
Pulp	0.12	0.45	<0.01	0.02
White sugar	<0.03	<0.03	<0.01	<0.01

Barley. A crop was treated with mancozeb on 3 occasions at 1.8 kg ai/ha, and harvested 25 days after the final application in a barley milling trial (85-0273) in the USA (ND) (Satterthwaite, 1986h). The barley was put through a small-scale flour-milling process. Residues of dithiocarbamates and ETU were measured in the barley and milled products (Table 74).

Table 74. Residues of dithiocarbamates (as CS_2) and ETU in barley and milled products (Satterthwaite, 1986h).

Commodity	Dithiocarbamate residues, mg/kg as CS_2	ETU residues, mg/kg
Whole kernels (harvested grain from which dirt and straw had been removed)	1.6	0.03
Cleaned grain	0.46	<0.01
Kernel (no husk)	0.15	0.03
Husk	3.3	0.16
Bran	<0.03	0.015
Flour	<0.03	<0.01
Rough	3.1	0.07
Shorts and germ	<0.03	0.015

Maize was treated with mancozeb on 7 occasions at 1.7 kg ai/ha or on 8 occasions at 3.4 kg ai/ha, and harvested 21 days after the final application in a maize-processing trial (85-0568) in the USA (IL) (Satterthwaite, 1986j). In a small-scale process the maize was milled to produce meal, flour, germ, grits, crude oil, refined oil, hulls and soapstock.

Neither dithiocarbamates nor ETU were detected (<0.03 mg/kg for dithiocarbamates as CS_2, and <0.01 mg/kg for ETU) in the maize kernels or any of the products.

Wheat. Mancozeb was sprayed at 1.8 kg ai/ha on 2 or 3 occasions and wheat was harvested approximately 26 days after the final application. The wheat was milled and bread baked (Table 75). More details of the location of the trials and the mancozeb application are provided in the "Residues resulting from supervised trials" section, Table 44. Residues of dithiocarbamates in the grain and milled products were less than 0.5 mg/kg, and usually much less. Residues of ETU were undetectable (<0.01 and <0.02 mg/kg).

Table 75. Residues of dithiocarbamates (as CS_2) and ETU in wheat and milled products in a series of studies in the USA in 1975 and 1981. The mancozeb application details are recorded in Table 44.

Dithiocarbamate residues, mg/kg as CS_2				ETU residues, mg/kg				Study
Grain	Bran	Flour	Bread	Grain	Bran	Flour	Bread	
0.07	0.07	0.04	0.03	<0.01	<0.01	<0.01	<0.01	81-0167
0.09	0.14	0.04	0.03	<0.01	<0.01	<0.01	<0.01	81-0167
0.10	0.05	0.04	0.02	<0.01	<0.01	<0.01	<0.01	81-0168
0.05	0.04	0.04	0.02	<0.01	<0.01	<0.01	<0.01	81-0168
0.17	0.39	0.17	0.05	<0.02	<0.02	<0.02	<0.02	75-421-02
0.1	0.2	0.05	<0.05	<0.02	<0.02	<0.02	<0.02	75-467-02
0.1	0.2	0.08	<0.05	<0.02	<0.02	<0.02	<0.02	75-468-02
-	0.02	<0.03	<0.01	-	<0.01	<0.01	<0.01	81-0428
-	0.03	0.04	0.02	-	<0.01	<0.01	<0.01	81-0429
-	0.03	0.04	<0.01	-	<0.01	<0.01	<0.01	81-0430
-	0.05	0.06	<0.01	-	<0.01	<0.01	<0.01	81-0426
-	0.1	0.06	0.02	-	<0.01	<0.01	<0.01	81-0427
0.02	0.06	0.06	0.02	-	<0.01	<0.01	<0.01	81-0212
0.04	0.12	0.07	0.04	<0.01	<0.01	<0.01	<0.01	81-0214

Peanuts. Mancozeb was applied to a peanut crop 6 times at 1.8 kg ai/ha or 3.6 kg ai/ha in a processing trial (85-0516) in the USA (GA) (Satterthwaite, 1986d). The peanuts were harvested 14 days after the final application and processed into meal, crude oil, refined oil and soapstock in a small-scale simulation of a commercial process.

Neither dithiocarbamates nor ETU were detected (<0.03 mg/kg for dithiocarbamates as CS_2, and <0.01 mg/kg for ETU) in the raw peanuts or any of the products.

Johnson (1991) reported on the effects of typical consumer practices during food preparation on residues of dithiocarbamates and ETU in potatoes, tomatoes, onions and apples.

Potatoes were treated with mancozeb to obtain a residue of 0.5 mg/kg (as mancozeb). Some were washed for 5 seconds under running water with light rubbing by the operator's fingers encased in polypropylene gloves. A second set was thoroughly scrubbed with a vegetable brush under running water for 5 seconds. A third set was treated similarly and then towel-dried with a clean cotton cloth. A fourth set, after drying, was peeled with a standard kitchen potato peeler, keeping the amount of pulp removed with the peel to an absolute minimum. Dithiocarbamate and ETU residues were measured after each process (Table 76).

Tomatoes and apples were also treated at 0.5 mg mancozeb/kg and similarly washed and dried. Onions were treated at 50 mg mancozeb/kg and peeled. It was necessary to work at a higher level because naturally-occurring sulphur compounds caused analytical interference at lower levels. Results are summarized in Table 76.

Mancozeb residues are on the surface and are removed by washing, cleaning and peeling. Combinations of washing, scrubbing and drying remove quite a high proportion of the residue (70-90%). Very little ETU is produced during these typical food preparation steps.

In Table 76 the reduction factor is defined as the ratio of the mancozeb concentration after each process to its applied concentration (0.5 or 50 mg/kg). The ETU conversion factor is defined as the ratio of the ETU residue after the process to the applied mancozeb concentration.

Table 76. Reduction factors for mancozeb and conversion factors for ETU as a result of typical consumer practices in food preparation (Johnson, 1991).

Process	Potato		Tomatoes		Apples		Onions	
	Mancozeb reduction factor	ETU conversion factor	Mancozeb reduction factor	ETU conversion factor	Mancozeb reduction factor	ETU conversion factor	Mancozeb reduction factor	ETU conversion factor
Unwashed	0.65	0.01	0.48	0.01	0.91	0.02	1.0	0.01
Washed	0.70	0.01	0.18	0.01	-	0.01		
Washed + brushed	0.42	0.01						
Washed + brushed + dried	0.30	0.01						
Washed + brushed + dried + peeled	0.02	0.02						
Washed + dried			0.09	0.01	0.32	0.01		
Peeled							0.05	0.0

Studies on the fate of mancozeb residues during food processing were included in a recent review in the open literature of the effects of processing on pesticide residues (Holland *et al.*, *in press*).

Stability of pesticide residues in stored analytical samples

Schweitzer (1989a) reported the results of a two-year freezer storage stability study on mancozeb and ETU in apples, tomatoes and wheat.

Apples, tomatoes and wheat were homogenised and analysed to establish the absence of dithiocarbamates and ETU. Samples (10 g) were weighed into separate containers, fortified with mancozeb (1 mg/kg) or ETU (0.1 mg/kg) and then stored in a freezer at -20°C. Containers were periodically removed for residue analysis. The results are summarized in Table 77.

The stability of mancozeb was within the normally acceptable range, with more than 70% remaining after for the longest storage interval. ETU was somewhat more labile, suggesting that samples containing ETU residues at this level should be analysed without excessive storage.

Table 77. Freezer storage stability of mancozeb and ETU in apple, tomato and wheat samples (Schweitzer, 1989a).

Freezer storage time	Residues, mg/kg					
	Mancozeb			ETU		
	Apples	Tomatoes	Wheat	Apples	Tomatoes	Wheat
Day 0	1.00	1.03	0.98	0.095	0.096	0.092
1 month	1.03	1.01	1.00	0.103	0.101	0.102
6 months	1.01	0.98	1.02	0.064	0.082	0.087
12 months	0.75	0.71	0.81	0.046	0.076	0.072
24 months	0.76	0.76	0.76	0.050	0.058	0.060

Loftus (1990b) reported on the freezer storage stability of mancozeb and ETU residues in matrices of vegetables, meat and milk (Tables 78 and 79). The studies showed that mancozeb was stable at -20 ± 5°C in dry beans,

corn, lettuce, meat, milk, raw potato (marginal stability), and tomatoes; ETU was stable in dry beans, corn, lettuce (marginal stability), meat, milk, raw potato (marginal stability), and tomatoes.

Oxygen plays a role in the conversion of ETU to ethyleneurea. Surface residues would be more susceptible to degradation; fortified residues would probably be more susceptible to loss than incurred residues.

The stability of ETU was tested with both coarsely and finely ground samples. Short term studies (12 days) were conducted on finely ground matrices because the analytical protocol required subsamples to be extracted for analysis within five days of grinding.

Table 78. Stabiltiy of mancozeb residues to freezer storage (Loftus, 1990b). The finely ground commodity was fortified with mancozeb and stored in individual reaction flasks at -20° ± 5°C. Results were adjusted for the analytical recovery associated with the particular type of sample before the remaining residue was calculated.

Commodity and fortification level, mg/kg	Storage period	% of initial residue remaining
Dry beans, 2.0 mg/kg	0 days	84
	14 days	123
	1 month	102
	50 days	117
	3 months	108
	4 months	98
Frozen corn, 2.0 mg/kg	0 days	85
	14 days	113
	1 month	101
	50 days	96
	3 months	90
	4 months	86
Lettuce, 2.0 mg/kg	0 days	97
	12 days	96
	30 days	91, 91
	60 days	87
	90 days	95
Raw potato, 2.0 mg/kg	0 days	100
	14 days	84
	1 months	77
	3.5 months	59
Tomatoes, 2.0 mg/kg	0 days	96
	14 days	90
	1 month	91
	3 months	92
	6 months	100
Meat, 0.50 mg/kg	0 days	100
	14 days	98
	1 month	92
	3 months	112
		112
	6 months	
Milk, 0.50 mg/kg	0 days	102
	14 days	109
	1 month	89
	3 months	98
	6 months	79

Table 79. Stabiliy of ETU residues to freezer storage (Loftus, 1990b). The coarsely or finely ground commodity was fortified with ETU and stored in individual glass jars at -20° ± 5°C. Results were adjusted for the analytical recovery associated with the particular type of sample before the remaining residue was calculated.

Commodity and fortification level, mg/kg	Coarsely ground matrix		Finely ground matrix	
	Storage period	% of initial residue remaining	Storage period	% of initial residue remaining
Dry beans, 0.50 mg/kg	0 days	91	0 days	79
	14 days	96	5 days	95
	1 month	97	12 days	95
	3 months	81		
	4 months	85		
Frozen corn, 0.50 mg/kg	0 days	83	0 days	85
	14 days	97	5 days	103
	1 month	102	12 days	101
	3 months	95		
	4 months	95		
Lettuce, 0.50 mg/kg	0 days	113	0 days	97
	14 days	94	5 days	103
	30 days	107	12 days	117
	60 days	84		
	90 days	55		
Raw potato, 0.50 mg/kg	0 days	99	0 days	92
	14 days	84	5 days	72
	1 month	64	12 days	76
	3.5 months	47		
Raw tomato, 0.50 mg/kg	0 days	100	0 days	97
	14 days	104	5 days	103
	1 month	105	12 days	97
	3 months	93		
	6 months	89		
Meat, 0.10 mg/kg	0 days	102	0 days	101
	14 days	108	5 days	112
	1 month	106	12 days	108
	3 months	95		
	6 months	110		
Milk, 0.10 mg/kg	0 days	94		
	14 days	97		
	1 month	106		
	3 months	95		
	6 months	96		

Residues in the edible portion of food commodities

Residues of dithiocarbamates in citrus fruit treated with mancozeb were mainly in the peel (Table 12). In Japanese trials with mancozeb on "summer" citrus and mandarins, residue levels in the pulp were either undetectable (<0.004 mg/kg, as CS_2), or amounted to an average of 2.8% of the levels in the peel. ETU residues in the pulp were generally undetectable (<0.01 mg/kg), but in some cases reached about 10% of the level in the peel.

Washing mandarins and oranges treated with mancozeb (in Spanish trials) removed on average 89% of the dithiocarbamate residues (Table 12).

Dithiocarbamate residues were mostly undetectable (<0.03 mg/kg, as CS_2) in orange juice produced from oranges sprayed with mancozeb (Brazil),

and were on average less than 10% of the levels in the oranges (Table 12). ETU residues were mostly undetectable (<0.01 mg/kg) in both oranges and juice.

Dithiocarbamate residues in the pulp of lemons, limes and oranges from supervised mancozeb trials in the USA were approximately 7% of the levels in the whole fruit (Table 13). The samples were taken on the day of the final spray application and the results may have reflected pulp residues arising from previous applications, with whole fruit residues present in all samples. ETU residues in the pulp were on average 17% of the levels in the whole fruit.

ETU residues in apple sauce were approximately 2-4% of the mancozeb levels (as CS_2) in the apples (Germany, Table 16) and those in pear compote were less than 2% of the mancozeb levels (as CS_2) in the pears.

In the commercial processing of apples (Table 63) washing removed 30-50% of the mancozeb residues. Most (90%) of the remaining mancozeb went into the fraction containing the peel. Neither mancozeb (<0.1 mg/kg as CS_2) nor ETU (<0.03 mg/kg) was detected in clarified apple juice produced from apples containing mancozeb at 2.5 and 3.8 mg/kg (as CS_2). Mancozeb residues were carried through the process into the wet apple pomace with dithiocarbamate levels 3-6 times those in the washed apples.

In another processing trial on mancozeb-treated apples (Table 64), where washing was apparently not included, dithiocarbamate residues in the juice were on average 18% of the levels in the apples. There was no conversion to ETU.

Residues of dithiocarbamates were mostly undetectable (<0.05, <0.1, <0.25 mg/kg) in wine produced from mancozeb-treated grapes in France and Italy (Tables 23, 24). ETU was also not detectable (<0.01 or <0.02 mg/kg) in wine produced from these grapes.

De-stemming and cleaning removed an average of about 70% of the mancozeb residues from bunches of grapes (Table 65). Dithiocarbamate residues were not detectable (<0.1 mg/kg as CS_2) in clear grape juice produced from de-stemmed grapes containing 3.4-20 mg/kg as CS_2. Residue levels in thick juice averaged about 40% of the levels in the de-stemmed grapes, but with wide variation. Dithiocarbamate residues were not detectable (<0.1 mg/kg as CS_2) in grape jelly.

ETU was generated in the production of clear grape juice (14%), thick juice (18%) and jelly (20%). Estimated mean conversion yields of mancozeb in the de-stemmed grapes to ETU in the final product are shown in parentheses, with the assumption that 1 kg of product was derived from 1 kg of grapes.

Mancozeb residue levels in dried raisins were on average 3 times as high as in the raw grapes, mainly owing to the reduction in moisture. Conversion to ETU was 1% or less.

Less than 1% of the dithiocarbamate residue in mancozeb-treated grapes reached the red and white wines produced from them (Table 66). About 7% conversion to ETU occurred during wine production. Dithiocarbamate residue levels in raisins were about 20-50% of the levels in the grapes. No ETU was generated in raisin production.

Mancozeb residues were lost during the production of raisins which were dried in the sun for 13 days and then cleaned (Table 67), although the mean residue levels in the raisins were 120% of the levels in the grapes owing to the loss of moisture. No ETU was generated in this process.

In Australian banana trials (Table 26) dithiocarbamate residues were not detected (<0.1 mg/kg as CS_2) in the pulp. ETU was not detectable (<0.1 mg/kg) in the peel or the pulp.

Washing reduced mancozeb residues in papayas by 50% (USA, Table 27) but did not influence ETU residue levels. Mancozeb residues in the pulp

were 35-40% and ETU residues were 35% of the levels in the whole fruit.

Mancozeb residues in frozen corn and canned corn were less than 10% of the levels in the raw sweet corn whole ears (Table 68). ETU was not generated in the products.

In a tomato processing trial 50% or more of the mancozeb residues were removed by a 30-second water spray wash (Table 69). Dithiocarbamate residues were undetectable (<0.1 mg/kg as CS_2) in canned tomato juice and tomato pomace produced from tomatoes with residues of 0.2 and 0.5 mg/kg. Conversion to ETU occurred in the production of canned tomato juice (20-50%). The estimated yield of ETU is shown in parentheses and was calculated with the assumption that 1 kg of washed rinsed tomatoes produced 1 kg of juice.

The commercial washing of tomatoes removed more than 90% of the mancozeb residues (Table 70), which were then not detectable (<0.03 mg/kg as CS_2) in tomato juice or pomace. ETU residues in the juice were of the same order as dithiocarbamate residue levels in the washed tomatoes.

Dithiocarbamate residues were essentially not detectable (<0.1 mg/kg as CS_2) in potatoes field-treated with mancozeb at an exaggerated application rate or in the processed potato products, except chips and flakes (Table 72). ETU was detected in potato granules (0.08 mg/kg) and flakes (0.23 mg/kg) produced from potatoes containing less than 0.1 mg/kg dithiocarbamate residues as CS_2.

Dithiocarbamate and ETU residues were undetectable (<0.03 and <0.01 mg/kg respectively) in white sugar produced from sugar beet containing dithiocarbamate residues of 0.14 and 0.16 mg/kg, as CS_2 (Table 73).

Mancozeb was undetectable in bran and flour from milled barley; the detection limit was less than 7% of the level in the cleaned grain. Cleaning the grain prior to milling reduced the residue level by 70% (Table 74).

In wheat milling and baking trials (Table 75) dithiocarbamate residues in the bread were either undetectable or, on average, 30% of the levels in the grain. ETU was not detectable in the bread.

Mancozeb was used on hops in two German trials (Table 45), leading to dithiocarbamate residues in the dry hops of 2.2 and <1 mg/kg. ETU levels in the beer produced using the hops were 0.04 and 0.02 mg/kg respectively.

Typical consumer practices were shown to reduce mancozeb residue levels in potatoes, tomatoes, apples and onions (Table 76). Residue levels in potatoes subjected to washing, brushing, drying and peeling were reduced by 97%. Residues in tomatoes and apples after washing and drying were reduced by 80% and 65% respectively. Residues in onions were reduced by 95% on peeling.

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

In a US Food and Drug Administration monitoring programme a variety of baby foods (864 samples) were monitored for pesticide residues (Yess *et al.*, 1993). ETU residues were detected in 65 samples as follows: baked goods (1 of 29 samples), cereals (6 of 56), combination meat dinners (0 of 103), combination poultry dinners (0 of 72), desserts (9 of 70), fruits and fruit juices (38 of 310), infant formulas (0 of 48) and vegetables (11 of 167). The highest levels detected were 0.06 mg/kg.

A large survey of food items in the USA in 1989-90 for dithiocarbamate and ETU residues was conducted by the four US registrants (Slesinski, 1990). Approximately 300 samples of each of 19 different raw and processed food commodities were collected according to a statistically designed protocol at biweekly intervals at urban, suburban and rural grocery stores across the USA. Attention was paid to analytical methods to achieve limits of determination for ethylenebis(dithiocarbamate)s and ETU

of 0.003 mg/kg (as CS_2) and 0.001 mg/kg respectively. The survey was conducted according to GLP. The results are summarized in Table 80.

Most of the samples (91% of 5241 samples apart from broccoli and onions, which were excluded because of endogenous CS_2 generation, did not contain measurable dithiocarbamate residues. No measurable residues of ETU were found in 82% of the samples.

Weighted means were calculated taking into account the percentage of the crop which might theoretically have been treated, the distribution of the grocery stores and their commodity volumes, and assigining residues of half the LOD to residue levels which were below that limit.

Table 80. Summary of US survey of food items for dithiocarbamate and ETU residues in 1989-90 (Slesinski, 1990).

Commodity	No. of samples	Dithiocarbamates (as CS_2)			ETU		
		No. with residues >LOD	Range, mg/kg	Weighted mean, mg/kg	No. with residues >LOD	Range, mg/kg	Weighted mean, mg/kg
Green beans, raw	22	1	<0.01-0.018	0.003	0		0.002
Green beans, frozen	26	0		0.002	0		0.002
Green beans, canned	13	0		0.002	0		0.002
Green beans, infant	13	0		0.002	3	<0.01-0.04	0.006
Dry beans	311	0		0.002	0		0.0014
Dry beans, canned	296	0		0.002	0		0.0011
Broccoli, raw	306	306	0.027-1.6	0.26	6	<0.0025-0.015	0.0013
Broccoli, frozen	298	99	<0.01-0.62	0.014	23	<0.0025-0.094	0.0028
Celery	26	7	<0.01-0.19	0.017	1	<0.01-0.024	0.002
Corn, raw	296	0		0.001	6	<0.005-0.013	0.0006
Corn, frozen	298	1	<0.01-0.016	0.001	0		0.0005
Corn, canned	297	0		0.001	2	<0.005-0.028	0.0006
Cucumbers	317	60	<0.01-0.45	0.013	70	<0.0025-0.053	0.0040
Lettuce	306	10	<0.01-0.79	0.010	4	<0.0025-0.013	0.0014
Onions	345	334	<0.01-0.31	0.10	94	<0.0025-0.043	0.0031
Potato, raw	316	7	<0.003-0.13	0.001	104	<0.002-0.045	0.0021
Potato, frozen	298	5	<0.003-0.004	0.0013	180	<0.002-0.023	0.0044
Tomatoes, raw	316	205	<0.003-0.25	0.016	146	<0.002-0.034	0.0027
Tomato juice	298	16	<0.005-0.015	0.001	74	<0.002-0.022	0.0015
Tomato ketchup	298	6	<0.005-0.031	0.001	94	<0.002-0.017	0.0016
Tomato paste	298	14	<0.01-0.17	0.004	170	<0.002-0.098	0.0061
Tomato puree	298	13	<0.005-0.011	0.001	108	<0.002-0.029	0.0031
Meat	298	19	<0.001-0.004	0.0001	0		0.000005
Milk	298	41	<0.001-0.002	0.0002	0		0.000005

Grape juice samples (100) were taken from major grape juice producers in the USA to determine likely dithiocarbamate and ETU residues in juice commercially processed from grapes grown where dithiocarbamate fungicides had been used on the 1990 crop (Honeycutt, 1991). The sampling plan aimed at a representative sample of the juices.

Samples were analysed for dithiocarbamates (limit of determination 0.01 mg/kg as CS_2) and ETU (limit of determination 0.005 mg/kg). ETU was not detected in any of the samples. Dithiocarbamate residues (as CS_2) were detected in 92 samples. The median value was approximately 0.022 mg/kg as CS_2. Residue levels in 46 of the samples fell in the 0.02-0.05 mg/kg range, 45 samples had residues up to 0.02 mg/kg, and 9 above 0.05 mg/kg.

If the dithiocarbamates were ethylenebis(dithiocarbamate)s, ETU should also have been detected because the production of grape juice involves several heating steps: 2 hours at 60°C during pressing and juice filtration, 1 minute at 88°C for filtered juice pasteurisation and again during filling, and finally 4-5 minutes at 74-77°C after bottling. There was some suggestion that ferbam, a dithiocarbamate fungicide which does not generate ETU, may have been the source of some of the dithiocarbamate residues.

A further 17 samples of grape juice produced from grapes from districts in the USA where dithiocarbamates were not used contained no detectable residues of dithiocarbamates or ETU.

In an Australian study, samples of tomatoes and commercially processed tomato products were analysed for ETU residues (Dukes, 1991; Zalewski and Edwards, 1992). In all samples ETU levels were less than the limit of determination (0.1 mg/kg). The numbers of samples included in the study were tomatoes 7, tomato paste 30, and thin pulp 4.

METHODS OF RESIDUE ANALYSIS

Methods for dithiocarbamates rely on the generation of CS_2, which can be measured by GLC or by colorimetry.

The methods used in the survey of US food items by Slesinski (1990) for ethylenebis(dithiocarbamate) residues in crops, processed commodities, meat, and milk were described by Westberg (1989a-c). The methods rely on the formation of CS_2 from dithiocarbamate residues during reaction with hydrochloric acid + stannous chloride at 100°C in a sealed reaction flask. CS_2 is then measured by GLC headspace analysis (flame-photometric detector). Calibration relies on an ethylenebis(dithiocarbamate) standard similarly prepared and injected.

The laboratory sample (wet and dry crops, meat) was chopped or ground while frozen with dry ice. Frozen milk was quick-thawed to a slush using a cold water bath. The analytical portion (4 g for crops, 10 g for meat, 20 g for milk) was placed in the reaction flask for CS_2 generation. Samples had to be kept frozen at all times until the addition of the reagent, including during weighing (samples kept on dry ice before and after weighing). The detection limit for dithiocarbamates (as CS_2) was 0.01 mg/kg in crops and 0.001 mg/kg in meat and milk.

Rogers *et al.*, (1989a-c) described methods used in the Slesinski (1990) survey for ethylenethiourea in crops, meat and milk. ETU was extracted from the sample with water (pH adjusted to 11-12 with ammonia) + ethanol or methanol, the extract was cleaned up on an alumina column, and the ETU was determined by HPLC.

Samples of crops or meat were ground while frozen with dry ice. Milk was thawed to a slush for weighing. Samples must be kept frozen until the extraction solvent is added. All glassware that comes into contact with extracts or ETU solutions must be silanized. Determination was by HPLC with electrochemical detection.

Loftus (1990a) assembled the validation data for these methods. Dithiocarbamate recoveries were tested with celery, snap beans, dry beans, frozen corn and potatoes fortified with mancozeb at 0.02, 0.2 and 2.0 mg/kg, tomatoes fortified at 0.005, 0.01 and 0.02 mg/kg, and meat and milk at 0.002, 0.005 and 0.02 mg/kg. The work was distributed among three laboratories. Recoveries exceeded 70% except from dry beans (55-62%) and frozen corn (67-73%), both analysed in the same laboratory.

ETU recoveries were tested with celery, snap beans, dry beans and corn fortified with ETU at 0.01, 0.1 and 1.0 mg/kg, potatoes, tomatoes and tomato paste fortified at 0.002, 0.005 and 0.01 mg/kg, and meat and milk fortified at 0.001, 0.003 and 0.01 mg/kg. Recoveries exceeded 70% except from meat (67-74%).

There was some evidence that ethylenebis(dithiocarbamate) residues could be converted to ETU during analysis, with estimated conversion rates of 0.22-8.5%. Experimental techniques which minimize the time taken to perform critical steps and ensure that reagents such as HPLC-grade water do not degrade the dithiocarbamates are needed to reduce the conversion.

Bulb onions (Pennwalt study BR-88-15) and broccoli (Pennwalt study BR-89-09, and Rohm and Haas data) were shown to contain endogenous CS_2 or compounds which produced CS_2 in the dithiocarbamate analytical method. Twelve samples of bulb onions (10 varieties, from 10 sites in the USA) certified not to have been treated with dithiocarbamates showed, on analysis, CS_2 residues ranging from undetectable (<0.03 mg/kg) to 0.13 mg/kg, with a median of 0.05 mg/kg. The CS_2 in eight samples of broccoli (6 varieties, from 6 sites in the USA), certified as not treated with dithiocarbamates, ranged from undetectable (<0.01 mg/kg) to 0.79 mg/kg, median 0.32 mg/kg.

Kallio and Salorinne (1990) reported carbon disulphide as one of the 27 volatile compounds identified by headspace GC-MS of onions.

Larese (1988a) analysed bananas for dithiocarbamate residues by boiling the sample with dilute acid to release CS_2, which was carried by an air stream into an ethanol trap at dry-ice temperature. The CS_2 was measured by GLC with flame-photometric detection in the sulphur mode. This method was used in the US supervised residue trials on bananas and wheat.

An earlier method (Keppel, 1971) measured the trapped CS_2 colorimetrically with a cupric acetate/diethanolamine reagent. It was used in the US supervised residue trials for the analysis of almonds, asparagus, bananas, carrots, celery, cucumbers, oranges, peanuts, potatoes, summer squash, tomatoes, wheat and winter squash.

Larese (1988b) extracted ETU from bananas with methanol, and cleaned up the extract on an aluminium oxide column. The ETU was derivatised with bromobutane to form butyl-ETU, which was determined by GLC with flame-photometric detection in the sulphur mode. The method was used in the US supervised trials to analyse almonds, asparagus, bananas, celery, cucumbers, oranges, peanuts, potatoes, tomatoes, and wheat.

Australian residue analyses were by methods for dithiocarbamates (Shields, 1990e) and ETU (McCarthy, 1990), similar to those described by Westberg (1989a) and Rogers *et al.*, (1989a).

Shields (1990e) described a GLC method for measuring the carbon disulphide evolved from dithiocarbamate residues. Samples were cut up and representative portions (100 g) taken for analysis. The maceration of crop samples was not recommended because contact between plant acids and dithiocarbamates may cause loss of residues.

Carbon disulphide was generated in a hydrolysis flask by treating the sample with 40% stannous chloride in hydrochloric acid under reflux. The evolved carbon disulphide was swept by a current of air into an ethanol trap maintained at a low temperature in a dry-ice/acetone trap. The ethanol solution was then analysed for CS_2 in a gas chromatograph equipped with a flame-photometric detector (S filter).

Recoveries of mancozeb from the trial crops were in the range 55-115%, mean 84% (n = 38).

McCarthy (1990) described an HPLC method for ETU residues in plant material. The sample was mixed with the anti-oxidant cysteine hydrochloride and extracted with water (adjusted to pH 11-12 using concentrated ammonia) and methanol. The extract was filtered and the filtrate reduced in volume by rotary evaporation. Clean-up was effected by absorption of the aqueous concentrate into 10 g of GLC column support material followed by elution of the ETU from this material with methanol/chloroform through a small alumina column.

The solvent was removed and the residue taken up in water for HPLC analysis with UV detection. The ease of oxidation of ETU and danger of loss of residues were stressed. Precautions such as the use of silanized glassware and the addition of the anti-oxidant were needed. Recoveries were in the range 44-137%, mean 81% (n = 9).

Mellet (1993a, and related reports) described the method used for measuring the dithiocarbamate residues in the French trials. The analytical sample was treated with stannous chloride in hydrochloric acid under hot conditions to liberate carbon disulphide, which was swept with a current of air into an absorption trap containing a colorimetric reagent (diethanolamine and cupric acetate). The absorbance of the coloured solution was measured at 435 nm. Known amounts of mancozeb were run through the procedure to establish the calibration.

Recoveries were determined and controls analysed with each crop in the residue trials. Some types of sample can give a false response if they contain sulphur compounds which generate CS_2 during the hydrolysis step, or if they give a false colour with the reagent. Some examples are discussed in the section on supervised trials.

A UK Panel on the Determination of Dithiocarbamate Residues (1981) examined the headspace method for dithiocarbamate residues in lettuce. The Panel drew attention to the loss of residues which can occur between beginning to cut the sample and inserting it into the reaction bottle. Vegetables and fruits must be analysed as soon as possible after cutting or picking, and any further cutting or dicing of the whole commodity should be carried out immediately before placing in the reaction flask, and should be kept to a minimum. Foodstuffs should be frozen whole, when this becomes necessary, and chopped and mixed in the frozen state immediately before taking the analytical samples.

It should be noted that the previously described freezer storage studies on spiked homogenised samples showed that mancozeb residues were stable under freezer conditions, but the evidence suggests that if storage is necessary samples should be frozen whole.

Onley *et al.*, (1977) reported a method for ETU residues in crops and food, which minimized the conversion of ethylenebis(dithiocarbamate) residues to ETU. Extracts were cleaned up by adsorption on GLC column support material and alumina. The final determination of ETU was by HPLC or GLC (as the S-butyl derivative). TLC was used for additional identification. Collaborative testing of the method was reported by Onley (1977). The HPLC method was used to determine the ETU residues in US supervised trials on carrots, celery, summer squash and winter squash.

Krause (1989) extracted ETU with a methanol/aqueous sodium acetate solution and cleaned up the extract on a diatomaceous earth column. The final analysis was by HPLC on a graphitized carbon column with electrochemical detection. The limit of determination was 0.01-0.02 mg/kg. Celery samples showed low recoveries.

Doerge and Miles (1991) extracted and cleaned up ETU residues in crop samples by the method of Krause (1989), and used particle beam liquid chromatography/mass spectrometry for quantitative determination and positive identification of ETU down to 5 ìg/kg.

NATIONAL MAXIMUM RESIDUE LIMITS

The Meeting was aware that the following national MRLs had been established.

Country (residue definition)	MRL, mg/kg	Commodity
Australia (as CS_2)	0.01	potato
	*0.2	milks
	0.2	bulb onion, peanut
	*0.5	edible offal (mammalian), eggs, meat (mammalian)
	0.5	cereal grains
	1	banana, carrot, fruiting vegetables, cucurbits
	2	beans except broad bean and soya bean, broad bean (green pods and immature seeds), brassica (cole or cabbage) vegetables
	3	chard, fig, head lettuce, leaf lettuce, pome fruits, stone fruits, tomato
	5	celery, grapes
Canada (as zineb)	nr[1]	carrot, maize, onions, potato, sugar beet (sugar)
	4	cucumber, tomato
	5	celery
	7	apple, apricot, asparagus, beet, blackberry, blackeyed pea, blueberry, broccoli, Brussels sprouts, cabbage, cauliflower, cherries, collards, common bean, cranberry, currant, date, egg plant, gooseberry, grapes, green onions, guava, head lettuce, huckleberry, kale, kohlrabi, loganberry, mango, melon (not watermelon), mushrooms, mustard greens, papaya, peach, peanuts, pear, peas, pepper, plum, pumpkin, quince , radish, raspberry, rutabaga, spinach, squash, strawberry, turnip
Germany	0.2	potatoes
	2	asparagus, pome fruits, stone fruits, wine grapes
	25	hops
Mexico (mancozeb)	0.1	asparagus, corn grain, fresh corn

Country (residue definition)	MRL, mg/kg	Commodity
	0.5	bulb onions dry, cotton seed, garlic, green onions, onions, peanuts, potatoes
	2	beets, carrots, sugar beets
	4	bananas, cucumbers, melons, squash, summer squash, tomatoes, watermelons
	5	barley grain, celery, oat grain, rye grain, wheat grain
	7	apples, grapes, pumpkin
	10	papayas, pears, spinach
Spain (as CS_2)	0.2	cereals, potatoes, sugar beet
	3	apples, citrus fruit, medlars, olives, persimmon, stone fruit, vegetables
	4	grapes, hops, strawberries
USA (mancozeb)	0	papayas edible pulp
	0.1	asparagus, corn grain
	0.5	bananas pulp without peel, cotton seed, dry bulb onions, fresh corn, kidney, liver, peanuts, popcorn, potatoes, sweet corn
	1	barley flour, oat flour, rye flour, wheat flour
	2	carrots, sugar beets
	4	bananas, cucumbers, melons, summer squash, tomatoes
	5	barley grain, celery, corn fodder, corn forage, oat grain, rye grain, wheat grain
	7	apples, cranberries, grapes
	10	crab-apples, fennel, papayas, pears, quinces
	20	barley bran, barley milled feed, oat bran, oat milled feed, rye bran, rye milled feed, wheat milled feed
	25	barley straw, oat straw, rye straw, wheat straw
	65	peanut vine hay, sugar beet tops

[1] nr: residues up to 0.1 mg/kg are acceptable

APPRAISAL

Mancozeb, evaluated in 1967 and several times since, was scheduled for review in 1993 in the CCPR periodic review programme (ALINORM 93/24A, para 71).

The Meeting received extensive information on GAP, supervised residue trials, animal transfer studies, metabolic fate in farm animals and crops, fate during processing and storage, residues in food in commerce and at consumption, and methods of residue analysis.

When lactating goats were dosed with [^{14}C]mancozeb ([^{14}C]ethylenediamine) in the feed, most of the ^{14}C was excreted in the faeces and urine. Excretion levels reached a plateau by day 2. The concentration of ^{14}C in milk reached a plateau by day 3 at all dosing levels. Concentrations of ^{14}C were higher in liver and kidney than in the other tissues or organs, most of it being incorporated into natural products. The main metabolites identified in the kidney were glycine, *N*-formylglycine, ethylenediamine, *N*-acetylethylenediamine, ethyleneurea, ethylenethiourea (ETU) and ethylenebisisothiocyanate sulphide.

When laying hens were dosed with [^{14}C]mancozeb in the feed, most of the ^{14}C was excreted in the faeces. ^{14}C levels in whole eggs were still increasing at the end of the 7-day dosing period, but declined rapidly in eggs from a group of hens in which dosing was discontinued. Ethyleneurea was the identified metabolite present at highest levels in eggs and tissues. ^{14}C was present at higher levels in liver and kidney than in other organs or tissues. In the highest dosed group (equivalent to 36 ppm mancozeb in the feed) dithiocarbamate levels (as CS_2) by direct chemical analysis were muscle 0.02-0.04 mg/kg, liver 0.09 mg/kg, and eggs 0.007-0.02 mg/kg. ETU levels in the tissues of this group were either at or below the level of detection (0.007 mg/kg), and in eggs were 0.06 mg/kg. ETU levels in eggs were not detectable (<0.007 mg/kg) in the group dosed at the equivalent of 14 ppm.

Most of the ^{14}C was incorporated into the carbon pool, appearing in a range of natural products, when a tomato crop was treated with [^{14}C]mancozeb. Ethyleneurea was the major primary metabolite identified.

When a soya bean crop was treated with [^{14}C]mancozeb the primary metabolites identified in soya bean pods were 1-(2-imidazolin-2-yl)-2-imidazolidinethione, ethyleneurea, hydantoin and ethylenebisisothiocyanate sulphide. Much of the ^{14}C was incorporated into protein, lignin and oil.

In a sugar beet crop treated with [^{14}C]mancozeb, 1-(2-imidazolin-2-yl)-2-imidazolidinethione was the major primary metabolite to be identified. The total ^{14}C label was distributed 77% in the leaf and stem, and 23% in the root.

The primary metabolites identified in wheat which had received foliar applications of [^{14}C]mancozeb were ethyleneurea, ethylenediamine, ethylenebisisothiocyanate sulphide, 2-imidazoline and 1-(2-imidazolin-2-yl)-2-imidazolidinethione. Much of the ^{14}C was incorporated into carbohydrates.

Mancozeb is registered as a protective fungicide for use on citrus fruits, pome fruits, stone fruits, berries and other small fruits, tropical and subtropical fruits, bulb vegetables, root and tuber vegetables, Brassica vegetables, leafy vegetables, stalk and stem vegetables, fruiting vegetables, legume vegetables, cereals, tree nuts, oilseeds and miscellaneous crops in very many countries.

Typical spray concentrations for high-volume application of mancozeb were 0.15-0.20 kg ai/hl to a wide variety of crops in many countries, but higher concentrations were recommended in some cases. The application rate for high-volume application depended on the volume of spray per hectare required for the particular crop and the typical spray concentration.

The Meeting received extensive residue data from supervised trials on the following crops and commodities:

grapefruit (*USA*), lemons (*Spain, USA*), limes (*USA*), mandarins (*Japan, Spain*), oranges (*Australia, Brazil, Spain, USA*);

apples (*Australia, Austria, Belgium, Brazil, France, Germany, Hungary, Italy, Japan, Netherlands, UK, USA*), pears (*Australia, Brazil, France, Germany, Italy, Japan, USA*);

apricots (*Australia*), peaches (*Australia, Brazil*), plums (*Brazil, France*);

black currants (*UK*), cranberries (*USA*), grapes (*Australia, Brazil, France, Hungary, Italy, Japan, Portugal*), strawberries (*Japan, Spain*);

avocados (*Brazil*), bananas (*Australia, Brazil, Honduras, USA*), figs (*Brazil*), mangoes (*Australia, Brazil*), papayas (*USA*), passion fruit (*Australia*), persimmons (*Japan*);

garlic (*Brazil, France, Japan*), leeks (*France, Japan*), onions (*Australia, Brazil, Finland, Japan, Netherlands, USA*);

broccoli (*Brazil*), cabbage (*Brazil, Germany, Japan*), cauliflower (*Brazil, Spain*), Chinese cabbage (*Japan, Spain*);

cantaloupes (*USA*); cucumbers (*Australia, Brazil, France, Germany, Japan, Spain, USA*), gherkins (*Germany*), melons (*France, Germany, Japan*), pumpkins (*Australia, Brazil*), squash (*France, Japan*), summer squash (*Australia, France, USA*), watermelons (*Australia, Japan, USA*), winter squash (*USA*);

egg plants (*Brazil*), peppers (*Brazil, Spain*), sweet corn (*USA*), tomatoes (*Brazil, France, Germany, Italy, Japan, Netherlands, Portugal, Spain, USA*);

kale (*Brazil*), lettuce (*Spain*);

azduki beans (*Japan*), beans (*Australia, Brazil, France, Netherlands,*

Spain), French beans (*Brazil*), kidney beans (*Japan*), peas (*Brazil, France*);

beet (*Brazil*), carrots (*Australia, Brazil, France, Germany, USA*), lotus (*Japan*), potatoes (*Australia, Brazil, Finland, France, Germany, Italy, Japan, Netherlands, UK, USA*), sugar beet (*France, Italy, Japan*), yams (*Japan*);

asparagus (*France, USA*), celery (*USA*), chard (*Australia*), witloof (*France, Netherlands*);

barley (*Brazil, Netherlands, USA*), maize (*USA*), rice (*Brazil*), summer wheat (*Germany*), wheat (*Brazil, Canada, France, Spain, USA*), winter wheat (*Germany, Netherlands, UK*);

hops (*Germany*);

peanuts (*Australia, USA*), rape seed (*France, Netherlands*);

almonds (*USA*), cocoa (*Brazil*), coffee (*Brazil*),

barley straw (*Netherlands, USA*), maize fodder (*USA*), wheat straw (*Canada, France, Germany, Netherlands, UK, USA*);

almond hulls (*USA*), bean pods and foliage (*Australia*), bean straw (*Australia*), peanut foliage (*Australia*), peanut hay (*USA*), sugar beet leaves (*Italy, Japan, USA*).

Dithiocarbamate residues are expressed as mg CS_2 /kg throughout.

Mancozeb is used as a cover fungicide, often with the same spray concentrations for high-volume application, on a wide range of crops. Because the residue is on the surface and there is no translocation from foliage to fruits, residue levels are often similar on fruits of a similar size.

Mancozeb use patterns are common across the citrus fruits in each country. Spanish trials on mandarins (GAP spray concentration 0.32 kg ai/hl, PHI 15 days) produced dithiocarbamate residues up to 4.7 and 6.6 mg/kg at 14 days. For a similar use pattern on oranges, residues of dithiocarbamates were mostly less than 1 mg/kg (highest 1.3 mg/kg). Japanese trials showed that most of the residues are in the peel while the Spanish trials confirmed that washing the fruit generally removes 90% or more of the residue. The Meeting estimated maximum residue levels of 10 mg/kg and 2 mg/kg for mandarins and oranges respectively, based on mancozeb uses.

US trials on lemons, limes and oranges demonstrated that most residues of both dithiocarbamates and ETU were on the peel with little in the pulp. US data on citrus could not be evaluated because there was no US GAP.

Residue data and mancozeb GAP for apples were available from many countries. The mancozeb spray concentrations used in high-volume applications were quite similar in most countries (0.15-0.2 kg ai/hl). GAP information from France did not include a PHI so French data were evaluated according to the German GAP for pome fruit. Residues in apples above 1 mg/kg were recorded in trials in Australia, Austria, Brazil, Germany, Italy and the UK when mancozeb was used within GAP. The highest recorded residue exceeded 4 mg/kg. The Meeting estimated a maximum residue level of 5 mg/kg for apples.

Use patterns on pears were the same as on apples, with the highest recorded residue being 2.2 mg/kg. The Meeting recommended an MRL for pome fruit of 5 mg/kg for dithiocarbamates, based on mancozeb uses.

The number of trials on apricots, peaches and plums was inadequate to recommend MRLs. No data were available for cherries. The Meeting agreed to withdraw the MRL recommendations for cherries, peaches and plums.

Grape residue data were supplied from many countries. The highest residues from the main population of data were in the 2.1-2.8 mg/kg range (Italy) suggesting an MRL of 5 mg/kg. Australian trials produced residues higher than 20 mg/kg when mancozeb was used according to GAP, and residues seemed somewhat anomalous when compared with similar uses elsewhere. The Australian use pattern is currently under review; Australian residue data were not included in the current evaluation.

The number of trials on strawberries was inadequate to permit the estimation of a maximum residue level. The Meeting recommended the withdrawal of the strawberry MRL.

A consistent series of mancozeb trials on cranberries in the USA in 1985 and 1988 suggested an MRL of 5 mg/kg.

The highest residues in black currants from the UK mancozeb trials exceeded 5 mg/kg (5.1, 5.4 mg/kg). The Meeting estimated an MRL of 10 mg/kg for currants.

Residue data on bananas and mangoes are mutually supportive with similar uses leading to a similar range of residues. The Meeting estimated a maximum residue level of 2 mg/kg for bananas and mango. Data on papayas, where the use pattern permits harvest on the same day as application, suggested an MRL of 5 mg/kg. The number of trials for avocados, figs and passion fruit was too limited for recommendations.

Residue data on garlic were made available from trials in Brazil, France and Japan. Generally, residues were not detectable (<0.05 mg/kg and lower) as would be expected from a foliar-applied non-systemic fungicide. However, residues were detected in a control sample at 0.1 mg/kg, and the possibility should not be excluded that some varieties of garlic or some conditions of production and storage could generate endogenous CS_2 as in onions. Mancozeb trials on leeks in France and Japan were made available for evaluation. The highest residue of 0.30 mg/kg and the possibility of endogenous CS_2 (a control sample registered 0.21 mg/kg of CS_2) suggested a maximum residue level of 0.5 mg/kg for garlic and leeks.

Onion trials in Brazil, Japan, The Netherlands and the USA showed residues up to 0.17 mg/kg, with control samples in Japan at 0.12 mg/kg. The highest residues in onions were in an Australian trial at 1.7 mg/kg but appeared to be an order of magnitude higher than others and difficult to explain for an immobile residue such as mancozeb. The Meeting agreed to evaluate bulb onions, garlic and leeks as a group, and estimated a maximum residue level of 0.5 mg/kg for onions resulting from mancozeb use.

Residue data from trials on broccoli and cauliflower in Brazil in 1989 according to GAP were mutually supportive, and suggested a maximum residue of 0.2 mg/kg. Broccoli has, however, been shown to contain endogenous CS_2. In a US study 8 samples of broccoli (6 varieties, 6 sites in the USA) certified to be untreated with dithiocarbamates, on analysis contained CS_2 residues ranging from undetectable (<0.01 mg/kg) to 0.79 mg/kg, median 0.32 mg/kg. The Meeting had no information on endogenous CS_2 levels in cauliflower. It did not estimate a maximum residue level for broccoli or cauliflower because of the limited number of trials. The Meeting drew attention to the endogenous CS_2 levels in broccoli and possible endogenous CS_2 in related crops.

The highest residue in cabbages from trials according to GAP in Brazil and Japan was 0.22 mg/kg. Chinese cabbage from trials in Japan contained residues of 0.1 mg/kg in the untreated control, again suggesting endogenous CS_2 in the various Brassica vegetables. The Meeting was unable to recommend MRLs for cabbage or Chinese cabbage because of the limited data.

Cucumber residue data from trials according to GAP were supplied from Australia, Brazil, France, Japan and the USA, with residues up to 0.3 mg/kg in US trials. The Meeting estimated a maximum residue level of 0.5 mg/kg for cucumbers, based on mancozeb uses.

Residues in melons from the same use patterns were generally in the same range as in cucumbers. The Meeting recommended an MRL of 0.5 mg/kg for melons except watermelon.

There were only two trials on pumpkins according to GAP, one from Australia and one from Brazil, but residues were generally consistent with those in other cucurbits. The Meeting estimated a maximum residue level of 0.2 mg/kg for pumpkins.

Summer squash in trials in Australia, France and the USA showed residues from undetectable levels to 0.83 mg/kg, the last in a US trial where the harvest took place on day 4 after the last application. Residues would have been higher than on day 5 (the recommended PHI), but the level on day 10 was still 0.65 mg/kg. The Meeting estimated an MRL of 1 mg/kg for summer squash.

US data on winter squash could not be evaluated because no US GAP was available. Residues in squash in trials in France and Japan were quite similar, even though there was quite a difference in the use patterns, with PHIs of 3 and 30 days in France and Japan respectively. The Meeting estimated an MRL of 0.1 mg/kg for winter squash.

A US watermelon trial with mancozeb used 12 applications, but this would probably have little influence on the residues since US GAP allows a maximum of 8. The residue level on day 5 after the final treatment was 0.38 mg/kg. In the Australian trials residues were not detected (<0.1 mg/kg), and in the Japanese trials residues were measured on the watermelon pulp rather than the whole fruit. Residues in the pulp were at quite low levels, 0.01-0.02 mg/kg. The Meeting estimated a maximum residue level of 0.5 mg/kg, based on mancozeb uses on watermelon.

When mancozeb was used according to GAP on peppers in Brazil and Spain the highest residues were in the 0.5-0.6 mg/kg range. The Meeting recommended an MRL of 1 mg/kg for sweet peppers.

Sixty-eight trials with mancozeb on tomatoes were available from many countries. Many of the measured residue levels were in the 0.1-1 mg/kg range, but residues up to 4.1 mg/kg were recorded in the US trials. The Meeting recommended an MRL of 5 mg/kg for tomatoes.

US trials on sweet corn showed that dithiocarbamate residues were on the husk rather than in the kernels. Residues were not detected (<0.03 mg/kg) in the cob + kernels. Additional data were available from US processing studies where application of mancozeb at the recommended US rate produced undetectable residues (<0.03 mg/kg) in cob + kernels. The residue level was 0.03 mg/kg when mancozeb was used at 5 times the recommended rate. Mancozeb, an immobile residue, would not be expected in the cob and kernels, which are protected by the husk from direct application. The Meeting recommended an MRL for sweet corn of 0.1* mg/kg as being a practical limit of quantification.

In supervised mancozeb residue trials on kale in Brazil dithiocarbamate residues 14 days after the last application were 0.95 and 1.0 mg/kg for label rate and double label rate of application, but the number of trials was too limited to allow the estimation of a maximum residue level.

When lettuce was sprayed with mancozeb at 0.16 kg ai/hl in trials in Spain and harvested 14 days after the final application residues in the 3-10 mg/kg range were found. The Meeting estimated a maximum residue level of 10 mg/kg for mancozeb use on head lettuce.

Trials in Japan on adzuki beans and kidney beans, and in Brazil on beans and French beans generally demonstrated undetectable or low residues on bean seeds, but the LOD for some of the older results was too high to be useful. The Meeting was unable to recommend an MRL for dry beans because of the limited data. It was not completely clear whether the commodity analysed in the Brazilian trials on peas included peas + pods, or peas

only. The Meeting did not recommend an MRL for beans or peas.

Information on mancozeb residues in beetroot from trials in Brazil was made available, but the number of trials was insufficient to recommend an MRL.

Most residue levels in carrots arising from approved uses of mancozeb were less than 0.2 mg/kg, but a number of values were found in the 0.5-1 mg/kg range in the Brazilian trials. The Meeting estimated a maximum residue level of 1 mg/kg for carrots.

Dithiocarbamate residues were not detected (<0.02 mg/kg) in East Indian lotus in two trials from Japan, but the data were insufficient to estimate a maximum residue level.

One hundred and seventeen mancozeb potato trials, but many not within GAP, were available from 9 countries for review. Residues were mostly undetectable even when mancozeb had been used at exaggerated application rates. Residues were sometimes detected, and the residues are more likely to depend on the inadvertent spraying of exposed potatoes than on the application rates or pre-harvest intervals. The highest residues were found in a French trial at 0.32 mg/kg and a German trial at 0.26 mg/kg, but they appeared exceptional when compared with all the other results. The Meeting estimated a maximum residue level of 0.2 mg/kg for uses of mancozeb on potatoes.

Dithiocarbamate residues from sugar beet trials in France, Italy and the USA were mostly around 0.1 mg/kg or lower, but residues in the 0.2-0.4 mg/kg range were recorded in US trials. The Meeting recommended a maximum residue level of 0.5 mg/kg for mancozeb use on sugar beet.

The US use pattern for mancozeb on asparagus requires a long PHI, 120 days in some states and 180 days in others. As expected, residues were low after this interval in the US trials. The French trials on asparagus could not be evaluated because no information on the French PHI was available. The Meeting recommended a maximum residue level of 0.1 mg/kg for asparagus.

No US GAP for mancozeb uses on celery was available to permit evaluation of US trials. Only one trial on chard according to GAP was available, from Australia, and this was insufficient in the absence of data from other similar vegetables which could have provided mutual support. Witloof trial data from France and The Netherlands could not be evaluated in the absence of GAP information.

Results of barley trials in Brazil, The Netherlands and the USA were made available to the Meeting. Dithiocarbamate residues up to 0.55 mg/kg were recorded in the US trials, and an MRL of 1 mg/kg for barley is recommended.

Results of a large number of mancozeb trials on wheat were supplied from 8 countries. The highest dithiocarbamate residues were recorded from trials in France (0.26 mg/kg), Germany (0.4 mg/kg), The Netherlands (0.82, 0.75 and 0.49 mg/kg) and the UK (0.42, 0.5 mg/kg), but in many of the trials residues were not detected. The Meeting estimated a maximum residue level of 1 mg/kg for mancozeb uses on wheat.

The PHI for the use of mancozeb on maize in the USA is 40 days; most of the residue data in the supervised trials were from shorter treatment-to-harvest intervals, and so could not be evaluated. In two trials where the longer interval was observed the commodity analysed was the "ear". Presumably this is the cob + grain. The appropriate commodity for a maize MRL is the grain.

Data from two supervised trials on rice according to the conditions of Brazilian GAP were made available to the Meeting. The data suggest a maximum residue level of 2 mg/kg, but trials covering a wider range of conditions are desirable for such an important crop. Also, if dithiocarbamate residues in this range or higher are likely, information on their fate during milling and cooking is desirable.

Two German trials with mancozeb on hops led to dithiocarbamate levels in dry hops of 2.2 and <1 mg/kg, but the information was too limited to permit the estimation of a maximum residue level.

Dithiocarbamate residues were not detected (<0.1, <0.03 mg/kg) in peanuts in Australian and US trials even when exaggerated application rates were employed. An MRL of 0.1* mg/kg was recommended.

Residues were detected in almonds in an Australian trial at the recommended application rate, but not at twice this rate. Because mancozeb is a surface residue only it is likely that any residues detected in the kernel were physically transferred during the cracking process. In the US trials dithiocarbamate residues were present in the almond hulls at 3 mg/kg, but no residues were detected (<0.03 mg/kg) in the almonds. The Meeting estimated a maximum residue level of 0.1* mg/kg for the use of mancozeb on almonds.

Mancozeb trials on cocoa and coffee in Brazil were insufficient for the Meeting to estimate maximum residue levels for cacao beans or coffee beans.

Residue data were available for wheat straw and fodder harvested at the same time as the wheat in the previously mentioned trials. Data on barley straw from trials in The Netherlands were also included for evaluation. Many of the residues were in the 2-5 mg/kg range but residues ranged up to 18 mg/kg. Two additional trials on barley with an identical use pattern were available from the USA, with residues of 24 mg/kg on barley straw from one of them. Wheat straw and barley straw should be assessed together for the same use pattern. The Meeting estimated maximum residue levels of 25 mg/kg for both. This level is compatible with animal commodity MRLs recommended on the basis of animal transfer studies.

Dithiocarbamate residues of 1.2 and 1.4 mg/kg were found in maize plants in two US trials 39 and 40 days after the final application of mancozeb. The Meeting estimated a maximum residue level of 2 mg/kg for maize fodder.

Dithiocarbamate residues up to 3.3 mg/kg on peanut foliage from previously mentioned Australian trials permitted the Meeting to estimate a maximum residue level of 5 mg/kg for peanut fodder. Data on almond hulls and peanut hay from US trials could not be evaluated because no US GAP was available for almonds and application rates on the peanuts were in excess of recommended rates.

When mancozeb was used on sugar beet crops according to US GAP, dithiocarbamate residues up to 17 mg/kg were found on sugar beet leaves. The Meeting estimated a maximum residue level of 20 mg/kg for sugar beet leaves or tops from mancozeb use.

Animal transfer studies with lactating dairy cows and laying hens were made available to the Meeting.

When dairy cows were fed a diet containing aged mancozeb residues equivalent to 5, 15 and 45 ppm mancozeb for 28 days dithiocarbamate residues were not detected (<0.04 mg/kg as CS_2) in the milk from any group. In the highest feeding group residues were not detected (<0.02 mg/kg,as CS_2) in muscle, while residues in the kidney and liver were 0.04 and 0.1 mg/kg respectively. The Meeting estimated maximum residue levels of 0.05*, 0.02* and 0.1 mg/kg for milks, meat and edible mammalian offal, respectively. These levels should accommodate animals eating 45 ppm mancozeb (25 ppm as CS_2) in the diet.

ETU residues were not detected (<0.01 mg/kg) in milk from the highest feeding group, but were detected in the thyroids of all the animals, with the highest doses causing the highest levels. ETU was detectable in muscle, liver and kidney of the highest feeding group, but had disappeared from the tissues of an animal returned to a residue-free diet for 7 days.

When laying hens were fed aged mancozeb residues (5, 15 and 45 ppm as mancozeb) for 28 days, dithiocarbamate residues were not detected (<0.04 mg/kg as CS_2) in the eggs from any feeding group. In the middle and highest feeding groups residues were 0.08 and 0.09 mg/kg (as CS_2) in muscle, while residues in the liver were 0.03 mg/kg. Measured residues in control samples were also around 0.03 mg/kg. The Meeting estimated maximum residue levels of 0.05*, 0.1 and 0.1 mg/kg for eggs, poultry meat and poultry edible offal, respectively.

ETU residues were detected in some eggs from the highest feeding group (0.01-0.02 mg/kg), but were not detected in tissues.

Processing studies were made available to the Meeting on apples, grapes, sweet corn, tomatoes, potatoes, sugar beet, barley, wheat, maize and peanuts.

In general, mancozeb residues (which are on the surface) can be substantially diminished by vigorous washing. The remaining residues tend to remain with the insoluble fractions, so that clear juices are unlikely to contain them. The remaining mancozeb residues may, however, be converted to ETU if processing includes a heating step.

In the commercial processing of apples, washing removed 30-50% of the residue, the remainder being carried through the process into the pomace. Neither mancozeb nor ETU residues were detectable in clarified apple juice.

De-stemming and cleaning removed about 70% of the mancozeb residues from bunches of grapes. Dithiocarbamate residues were not detectable in clear grape juice, but were present in the thick juice. ETU was generated in the production of the grape juices and jelly.

Less than 1% of the dithiocarbamate residues in mancozeb-treated grapes entered red and white wines produced from them. Approximately 7% conversion to ETU occurred during the wine production.

In one study mancozeb residue levels in dried raisins were on average 3 times as high as in the raw grapes, while in another study levels in the raisins were 20-50% of the levels in the grapes. No ETU was generated in raisin production.

Mancozeb residues in frozen corn and canned corn were less than 10% of the levels in the raw sweet corn whole ears; ETU was not generated in the process.

The commercial washing of tomatoes removed more than 90% of the mancozeb residues. Dithiocarbamate residues in the tomato juice and pomace produced from the washed tomatoes were undetectable. ETU residues in the juice were of the same order as the dithiocarbamate levels in the washed tomatoes.

Dithiocarbamate residues were essentially undetectable (<0.1 mg/kg) in potatoes field-treated with mancozeb at an exaggerated rate, and in the processed potato products. ETU was present in potato granules (0.08 mg/kg) and potato flakes (0.23 mg/kg).

Dithiocarbamate and ETU residues were not detected (<0.03 and <0.01 mg/kg respectively) in white sugar produced from mancozeb-treated sugar beet containing dithiocarbamate residues of 0.15 mg/kg.

The cleaning of barley grain prior to milling reduced residue levels by 70%. Mancozeb residues were not detectable in bran or flour.

Milling and baking trials on wheat harvested after foliar mancozeb applications showed that dithiocarbamate residues in the bread were either undetectable or, on average, 30% of the levels in the grain. ETU was not detectable (<0.01 mg/kg) in the bread.

Maize was field-treated with mancozeb and harvested for processing into meal, flour, germ, grits, crude oil, refined oil and soapstock.

Neither dithiocarbamates nor ETU were detected in the maize kernels or any of the products (<0.03 and <0.01 mg/kg respectively).

A peanut crop was field-treated with mancozeb and harvested for processing into meal, crude oil, refined oil and soapstock. Neither dithiocarbamates nor ETU were detected in the raw peanuts or any of the products (same limits as above).

The ETU level was 0.04 mg/kg in beer produced from mancozeb-treated hops (dithiocarbamates 2.2 mg/kg as CS_2).

Typical consumer practices were shown to reduce mancozeb residue levels in potatoes, tomatoes, apples and onions. Residues in potatoes subjected to washing, brushing, drying and peeling were reduced by 97%. Residues in tomatoes and apples subjected to washing and drying were reduced by 80% and 65% respectively. Residues in onions were reduced by 95% on peeling.

Mancozeb residues were stable (>70% remaining) in homogenised samples of apples, tomatoes and wheat stored for 2 years at -20°C. ETU residues were more labile; more than 70% of the ETU remained in tomato and wheat matrices after 12 months storage, but not after two years. ETU residues in an apple matrix had declined to less than 70% after 6 months storage and to less than 50% after 12 months.

Mancozeb residues were shown to be stable at -20 ± 5°C in stored analytical samples of dry beans, corn, lettuce, meat, milk, raw potato (marginal stability), and tomato. ETU residues were shown to be stable at -20 ± 5°C in stored analytical samples of dry beans, corn, lettuce (marginal stability), meat, milk, raw potato (marginal stability), and tomato.

Under a US Food and Drug Administration monitoring programme a variety of baby foods (864 samples) were monitored for pesticide residues. ETU residues were detected in 65 samples; the highest levels detected were 0.06 mg/kg.

In 1989-90 in the USA a large survey of food items (approximately 300 samples each of 19 different raw and processed commodities) was conducted for dithiocarbamate and ETU residues. Most of the samples (91% of 5241 samples) did not contain measurable dithiocarbamate residues (<0.003 mg/kg as CS_2); broccoli and onions were excluded because of endogenous CS_2 generation. No measurable residues of ETU (LOD 0.001 mg/kg) were found in 82% of the samples.

Grape juice samples (100), from major grape juice producers in the USA using grapes from districts where dithiocarbamates had been used on the 1990 crop, contained no detectable ETU residues (LOD 0.005 mg/kg). Dithiocarbamates were detected in 92 of the samples (median value approximately 0.022 mg/kg as CS_2). If the dithiocarbamates were ethylenebis(dithiocarbamate)s, ETU should also have been detected because the production of grape juice involves several heating steps. There was a suggestion that ferbam, a dithiocarbamate fungicide but not an ethylene-bis(dithiocarbamate), may have been the source of some of the dithiocarbamate residues.

In an Australian study in 1991, ETU residues were not detected (<0.1 mg/kg) in tomatoes, commercially produced tomato paste or thin pulp (41 samples).

Analytical methods for dithiocarbamates rely on the generation of CS_2, which can be measured by GLC or by colorimetry.

Reaction with hydrochloric acid + stannous chloride at 100°C is needed for quantitative conversion to CS_2, which can be analysed by head-space GLC. Alternatively, the evolved CS_2 can be swept by a current of air into an ethanol trap maintained at dry ice/acetone temperature, and the ethanol solution then analysed by GLC. In the colorimetric approach the

evolved CS_2 is swept into a trap of cupric acetate/diethanolamine reagent. Some types of sample can give a false response by generating a false colour in the reagent.

A UK Panel on the Determination of Dithiocarbamate Residues (1981) drew attention to the loss of dithiocarbamate residues which can occur between commencement of cutting of the sample and insertion into the reaction bottle. Vegetables and fruits must be analysed for residues as soon as possible after cutting or picking, and any further cutting or dicing of the whole commodity should be carried out immediately before placing in the reaction flask, and should be kept to a minimum. Foodstuffs should be frozen whole, when this becomes necessary, and chopped and mixed in the frozen state immediately before taking the analytical samples.

ETU methods rely on HPLC or GLC for final analysis. Samples are typically extracted with aqueous ammonia (pH 11-12) + methanol or ethanol and the extract cleaned up on an alumina column. ETU is easily oxidised or lost during the analysis; precautions are needed, such as the use of silanized glassware. Precautions must also be taken to prevent ethylenebisdithio-carbamate residues from being converted to ETU during the analysis.

The Meeting was aware of national MRLs established in Australia, Canada, Germany, Mexico, Spain and the USA.

RECOMMENDATIONS

The recommendations for mancozeb are included under DITHIOCARBAMATES (105).

FURTHER WORK OR INFORMATION

Desirable

1. Supervised trials on rice covering a wider range of conditions.

2. Fate of mancozeb residues during the milling and cooking of rice.

REFERENCES

1. Arends, L. 1991a. Mancozeb residue determinations - carrots. Trial AUK-91-008. DuPont (Australia) Ltd. Unpublished.

2. Arends, L. 1991b. Mancozeb residue determinations - onions. Trial AUK-91-009. DuPont (Australia) Ltd. Unpublished.

3. Arends, L. 1992a. Mancozeb residue determinations - cucumber. Trial AUK-92-003. DuPont (Australia) Ltd. Unpublished.

4. Arends, L. 1992b. Mancozeb residue determinations - pumpkin. Trial AUK-92-004. DuPont (Australia) Ltd. Unpublished.

5. Arends, L. 1992c. Mancozeb residue determinations - watermelon. Trial AUK-92-005. DuPont (Australia) Ltd. Unpublished.

6. Arends, L. 1992d. Mancozeb residue determination - zucchini. Trial AUK-92-006. DuPont (Australia) Ltd. Unpublished.

7. Arends, L. 1992e. Mancozeb residue determination - silverbeet. Trial AUK-92-007. DuPont (Australia) Ltd. Unpublished.

8. Arends, L. 1992f. Mancozeb residue determination - peanuts. Trial AUK-92-008. DuPont (Australia) Ltd. Unpublished.

9. Cairns, S.M. 1990. Analysis of orange samples from Brazil. Lab memo 34-90-61, includes 89-0191. Rohm and Haas Company, USA. Unpublished.

10. Doerge, D.R. and Miles, C.J. 1991. Determination of ethylenethiourea in crops using particle beam liquid chromatography/mass spectrometry. Analyt. Chem., 63, 1999-2001.

11. Dukes, N. 1991. Laboratory report - ETU in tomato products. Report P91/ . ARL, Australia. Unpublished.

12. Elkins, E.R. and Kim, E.S. 1986. Investigation of changes in mancozeb residues during food processing. Project LC 1507. National Food Laboratory, Inc., USA. Unpublished.

13. Frank, R., Braun, H.E. and Ripley. 1986. Mancozeb and its metabolite ETU analytical methodology and terminal residues. Agricultural Laboratory Services Branch, Ontario Ministry of Agriculture & Food, Canada. Unpublished.

14. Galoux, M. 1992. Evolution des résidus de mancozèbe sur pommes. Project R&H/BA 7.138/1991. Station de Phytopharmacie, Belgium. Unpublished.

15. Haines, L.D. 1978. Analytical results of pesticide residues. Report 77-0300. Rohm and Haas (France). Unpublished.

16. Haines, L.D. 1979. Analytical results of Dithane residues. Report 78-0418. Rohm and Haas (Germany). Unpublished.

17. Haines, L.D. 1982. A gas chromatographic method for measurement of Dithane M-45 or other dithiocarbamate fungicides in waste water or natural waters. Report TR36F-82-20. Rohm and Haas Company, USA. Unpublished.

18. Haines, L.D. and Adler, I.L. 1973. Gas chromatographic determination of ethylene thiourea residues. J. Assoc. Off. Analyt. Chem., 56, 333-337.

19. Hammond, T. 1990a. Mancozeb residue determinations - apples. Trial AUE-90-026. DuPont (Australia) Ltd. Unpublished.

20. Hammond, T.G. 1990b. Manzate residue trials. Trial AUE-90-002, relates to 1289/90. DuPont (Australia) Ltd. Unpublished.

21. Hammond, T.G. 1991a. Mancozeb residue determinations - pears. Trial AUE-91-026. DuPont (Australia) Ltd. Unpublished.

22. Hammond, T.G. 1991b. Mancozeb residue determinations - peaches. Trial AUE-90-027. DuPont (Australia) Ltd. Unpublished.

23. Hammond, T.G. 1992. Mancozeb residue determinations - oranges. Trial AUE-92-001. DuPont (Australia) Ltd. Unpublished.

24. Holland, P.T., Hamilton, D., Ohlin, B. and Skidmore, M. *in press*. Effects of storage and processing on pesticide residues in plant products. Pure & Appl. Chem.

25. Honeycutt, R.C. 1991. Collection and analysis of produce grape juice for ETU and EBDCs. Hazard Evaluation and Regulatory Affairs Company, USA. Study 90-113RA, report 91-104. Unpublished.

26. Jameson, C.E. 1985. Metabolism study of mancozeb in laying hens. ABC Laboratory Number 32865. Analytical Biochemistry Laboratories Inc, USA. Unpublished.

27. Jameson, C.E. and Shaffer, S.R. 1986. Residues of mancozeb and ethylenethiourea in eggs and edible tissues from hens fed field aged mancozeb residues. ABC study 33552. Analytical Biochemistry Laboratories, Inc., USA. Unpublished.

28. Johnson, G.E. 1991. EBDC/ETU consumer practice study. Project ETU 90-02. Report 90-0084ATO-1. EN-CAS Analytical Laboratories, USA. Unpublished.

29. Kallio, H. and Salorinne, L. 1990. Comparison of onion varieties by

headspace gas chromatography-mass spectrometry. J. Agric. Fd Chem., 38, 1560-1564.

30. Keppel, G.E. 1971. Collaborative study of the determination of dithiocarbamate residues by a modified carbon disulfide method. J. Assoc. Off. Analyt. Chem., 54, 528-532.

31. Krause, R.T. 1989. Liquid chromatographic electrochemical determination of ethylene thiourea in food by revised official method. J. Assoc. Off. Analyt. Chem., 72, 975-979.

32. Larese, J. 1988a. Dithane (as CS_2) residue analysis of bananas. Project RH-04-88. Enviro-Bio-Tech, Ltd, USA. Unpublished.

33. Larese, J. 1988b. ETU residue analysis of bananas. Project RH-04-88. Enviro-Bio-Tech, Ltd, USA. Unpublished.

34. Larese, J. 1988c. ETU residue analysis of bananas. Project RH 04-88. Enviro-Bio-Tech, Ltd, USA. Unpublished.

35. Larese, J. 1988d. Dithane and ETU residues analysis of bananas. Project RH-57-88. Enviro-Bio-Tech, Ltd, USA. Unpublished.

36. Larese, J. 1989. Dithane and ETU residue analysis of dry beans. Project RH-11-89 86-0200. Enviro-Bio-Tech. Ltd, USA. Unpublished.

37. Loftus, M.L. 1990a. Method validation study final report. Report ETU 90-06. Technical Assessment Systems, Inc., USA. Unpublished.

38. Loftus, M.L. 1990b. Storage stability study. Report ETU 90-11. Technical Assessment Systems, Inc., USA. Unpublished.

39. Loftus, M.L. 1991. 1990 mancozeb and metiram apple field study. Report ETU 91-02. Technical Assessment Systems, Inc., USA. Unpublished.

40. Malet, J.C. 1990. Synthèse residus mancozebe. SRPV, Midi-Pyrenées, France. Unpublished.

41. Mazza, L.S. and Schweitzer, M.G. 1989. Mancozeb metabolism in tomatoes. Report TR-34-89-19. Rohm and Haas Company, USA. Unpublished.

42. McCarthy, B. 1990. Determination of ethylene thiourea residues in various substrates. Method PR20. Analchem Consultants, Australia. Unpublished.

43. McQuinn, D.J. 1991a. Mancozeb residue determinations - almonds. Trial AUI-91-032. DuPont (Australia) Ltd. Unpublished.

44. McQuinn, D.J. 1991b. Manzate DF and Dithane M45 residue trial - apricots. Trial AUI-91-034. DuPont (Australia) Ltd. Unpublished.

45. McQuinn, D.J. 1991c. Grape residue trials - 1989/1990. Manzate DF and Dithane M45. Relates to 868/90. DuPont (Australia) Ltd. Unpublished.

46. Mellet, M. 1993a. Determination of residues of mancozeb in grapes in France in 1991. Report RF 1052-1, includes R79.45, R79.65, R79.73, R80.13, R80.24, R80.34. Anadiag, S.A., France. Unpublished.

47. Mellet, M. 1993b. Determination of residues of mancozeb in apples in France in 1991. Report RF 1052-2, includes R80.32, R80.33. Anadiag, S.A., France. Unpublished.

48. Mellet, M. 1993c. Determination of residues of mancozeb in cereals in France in 1991. Report RF 1052-3 (amendment 1), includes R80.1, R80.2, R80.3. Anadiag, S.A., France. Unpublished.

49. Mellet, M. 1993d. Determination of residues of mancozeb in potatoes in France in 1991. Report RF 1052-4, includes R78.60, R78.61, R79.55, R79.58. Anadiag, S.A., France. Unpublished.

50. Mellet, M. 1993e. Determination of residues of mancozeb in pea in France in 1991. Report RF 1052-5, includes R79.31, R79.32. Anadiag, S.A., France. Unpublished.

51. Mellet, M. 1993f. Determination of residues of mancozeb in tomato in France in 1991. Report RF 1052-6, includes R79.43, R79.44. Anadiag, S.A., France. Unpublished.

52. Mellet, M. 1993g. Determination of residues of mancozeb in carrots in France in 1991. Report RF 1052-7, includes R79.61, R79.41. Anadiag, S.A., France. Unpublished.

53. Mellet, M. 1993h. Determination of residues of mancozeb in leeks in France in 1991. Report RF 1052-8, includes R79.63, R79.42. Anadiag, S.A., France. Unpublished.

54. Mellet, M. 1993i. Determination of residues of mancozeb in grapes in Portugal in 1991. Report RF 1052-9, includes R80.27. Anadiag, S.A., France. Unpublished.

55. Mellet, M. 1993j. Determination of residues of mancozeb in tomatoes in Spain in 1991. Report RF 1052-10, includes R80.30. Anadiag, S.A., France. Unpublished.

56. Mellet, M. 1993k. Determination of residues of mancozeb in cereals in Spain in 1991. Report RF 1052-11 includes R80.31. Anadiag S.A., France. Unpublished.

57. Mesch, P. 1991. Mancozeb residue determinations - passionfruit. Trial AUH-91-012. DuPont (Australia) Ltd. Unpublished.

58. Murray, A. 1992a. Karamate blackcurrant residue studies 1990 with Karamate Dry Flo (750 g/kg mancozeb water dispersible grain (WDG) formulation). R80.35. Rohm and Haas, UK. Unpublished.

59. Murray, A. 1992b. Karamate blackcurrant residue studies 1991 with Karamate Dry Flo (750 g/kg mancozeb water dispersible grain (WDG) formulation). R80.36. Rohm and Haas, UK. Unpublished.

60. Ollinger, J., Lyman, W.R. and Larkin, R.H. 1986a. Food processing studies for apples treated with mancozeb. Report 310-86-13. Rohm and Haas Company, USA. Unpublished.

61. Ollinger, J., Lyman, W.R. and Larkin, R.H. 1986b. Food processing studies for tomatoes treated with mancozeb. Report 310-86-14. Rohm and Haas Company, USA. Unpublished.

62. Ollinger, J., Lyman, W.R. and Larkin, R.H. 1986c. Food processing studies for grapes treated with mancozeb. Report 310-86-15. Rohm and Haas Company, USA. Unpublished.

63. Ollinger, J., Lyman, W.R. and Larkin, R.H. 1986d. Food processing studies for potatoes treated with mancozeb. Report 310-86-16. Rohm and Haas Company, USA. Unpublished.

64. Onley, J.H. 1977. Gas-liquid chromatographic method for determining ethylenethiourea in potatoes, spinach, applesauce, and milk: collaborative study. J. Assoc. Off. Analyt. Chem., 60, 1111-1115.

65. Onley, J.H., Giuffrida, L., Ives, N.F., Watts, R.W. and Storherr, R.W. 1977. Gas-liquid chromatography and liquid chromatography of ethylenethiourea in fresh vegetable crops, fruits, milk and cooked foods. J. Assoc. Off. Analyt. Chem., 60, 1105-1110.

66. Panel on Determination of Dithiocarbamate Residues. 1981. Determination of residues of dithiocarbamate pesticides in foodstuffs by a headspace method. Analyst, 106, 782-787.

67. Predmore, L. 1985. Metabolism of ^{14}C-mancozeb and non-radiolabeled mancozeb in lactating dairy goats. ABC Laboratory Number 32232. Analytical Biochemistry Laboratories Inc, USA. Unpublished.

68. Predmore, L. and Shaffer, S.R. 1986. Field aged mancozeb residues on alfalfa hay feeding study in lactating dairy cows. ABC study 33553. Analytical Biochemistry Laboratories, Inc., USA. Unpublished.

69. Reibach, P.H. 1986a. Distribution of radiolabeled mancozeb metabolites in field treated wheat plants. Report TR-31L-86-03. Rohm and Haas Company, USA. Unpublished.

70. Reibach, P.H. 1986b. Distribution of radiolabeled mancozeb metabolites and degradation products in wheat plants. Report TR-310-86-54, addendum to TR-31L-86-03. Rohm and Haas Company, USA. Unpublished.

71. Ritcey, G., McEwen, F.L., Frank, R., and Ripley, B. 1981. Minor crops, fungicide residues. Canada. Unpublished.

72. Ritcey, G., McEwen, F.L., Frank, R., and Ripley, B. 1983. Fungicide residue in celery and lettuce. Canada. Unpublished.

73. Ritcey, G., McEwen, F.L., Frank, R., and Ripley, B. 1984. Fungicide residue in lettuce. Canada. Unpublished.

74. Ritcey, G., McEwen, F.L., Frank, R., and Ripley, B. 1985. Fungicide residues in onions and soil. Canada. Unpublished.

75. Rogers, E., Tufts, K. and Westberg, G.L. 1989a. Determination of ethylene thiourea in crops. Method MTF-88AM-004. Morse Laboratories, Inc., USA. Unpublished.

76. Rogers, E., Tufts, K., Normington, S. and Westberg, G.L. 1989b. Determination of ethylene thiourea in meat. Method ETU-89AM-004. Morse Laboratories, Inc., USA. Unpublished.

77. Rogers, E., Tufts, K., Normington, S. and Westberg, G.L. 1989c. Determination of ethylene thiourea in milk. Method ETU-89AM-005. Morse Laboratories, Inc., USA. Unpublished.

78. Satterthwaite, S.T. 1984a. Analytical reports of Dithane and ethylenethiourea residues on eggplant. Report RH-10-84, includes 84-0105. Rohm and Haas Company, USA. Unpublished.

79. Satterthwaite, S.T. 1984b. Analytical reports of Dithane and ethylenethiourea residues on onion. Report RH-13-84, includes 84-0425. Rohm and Haas Company, USA. Unpublished.

80. Satterthwaite, S.T. 1985. C14 Dithane M-45 soybean residue study and material for metabolism study. Report TR-31L-85-17. Rohm and Haas Company, USA. Unpublished.

81. Satterthwaite, S.T. 1986a. Analytical reports of Dithane and ETU for carrot residue samples. Report 31A-86-06, includes 85-0221, 85-0222, 85-0258, 85-0279, 85-0303, 85-0506. Rohm and Haas Company, USA. Unpublished.

82. Satterthwaite, S.T. 1986b. Supplemental report to: Analytical reports of Dithane and ETU for carrot residue samples. Report 31A-86-06. Rohm and Haas Company, USA. Unpublished.

83. Satterthwaite, S.T. 1986c. Analytical reports of mancozeb and ETU for asparagus residue samples. Report 31A-86-07, includes 85-0134, 85-0136, 85-0278. Rohm and Haas Company, USA. Unpublished.

84. Satterthwaite, S.T. 1986d. Analytical reports of mancozeb and ETU residues for processed peanut samples. Report 310-86-07. Rohm and Haas Company, USA. Unpublished.

85. Satterthwaite, S.T. 1986e. Analytical reports of mancozeb and ETU residues for field corn samples. Report 31A-86-08, includes 83-0200, 83-0228, 83-0237, 83-0253, 83-0358, 83-0419, 85-0337, 85-0453. Rohm and Haas Company, USA. Unpublished.

86. Satterthwaite, S.T. 1986f. Analytical reports of mancozeb and ETU residues for processed grape samples. Report 310-86-08. Rohm and Haas Company, USA. Unpublished.

87. Satterthwaite, S.T. 1986g. Analytical reports of Dithane and ETU residues in melons. Report 31A-86-09, includes 85-0161, 85-0280, 85-0162. Rohm and Haas Company, USA. Unpublished.

88. Satterthwaite, S.T. 1986h. Analytical reports of mancozeb and ETU residues for processed barley samples. Report 310-86-09. Rohm and Haas Company, USA. Unpublished.

89. Satterthwaite, S.T. 1986i. Mancozeb and ETU residues in cucumbers. Report 31A-86-10, includes 85-0126, 85-0163, 85-0325, 85-0339. Rohm and Haas Company, USA. Unpublished.

90. Satterthwaite, S.T. 1986j. Analytical reports of mancozeb and ETU residues for processed corn samples. Report 310-86-10. Rohm and Haas Company, USA. Unpublished.

91. Satterthwaite, S.T. 1986k. Analytical reports of mancozeb and ETU residues for processed sugarbeet samples. Report 310-86-11. Rohm and Haas Company, USA. Unpublished.

92. Satterthwaite, S.T. 1986l. Analytical reports of Dithane and ETU residues for celery samples. Report 31A-86-11, includes 85-0165, 85-0350, 85-0397, 85-0401, 85-0454, 85-0455, 85-0503, 85-0561. Rohm and Haas Company, USA. Unpublished.

93. Satterthwaite, S.T. 1986m. Analytical reports of Dithane and ETU residues for sugarbeet samples. Report 31A-86-12, includes 85-0264, 85-0292, 85-0329, 85-0363, 85-0365, 85-0499, 85-0500, 85-0501, 85-0515. Rohm and Haas Company, USA. Unpublished.

94. Satterthwaite, S.T. 1986n. Analytical reports of mancozeb and ETU residues for processed apple samples. Report 310-86-12. Rohm and Haas Company, USA. Unpublished.

95. Satterthwaite, S.T. 1986o. Analytical reports of Dithane and ETU for summer squash residue samples. Report 31A-86-13, includes 85-0127, 85-0128, 85-0310, 85-0311, 85-0312, 85-0428, 85-0484. Rohm and Haas Company, USA. Unpublished.

96. Satterthwaite, S.T. 1986p. Analytical reports of Dithane and ETU for winter squash residue samples. Report 31A-86-14, includes 85-0129, 85-0460, 85-0479, 85-0480, 85-0485. Rohm and Haas Company, USA. Unpublished.

97. Satterthwaite, S.T. 1986q. Analytical reports of Dithane and ETU residues for cranberry samples. Report 31A-86-16, includes 85-0294, 85-0295, 85-0341, 85-0456, 85-0457, 85-0458. Rohm and Haas Company, USA. Unpublished.

98. Satterthwaite, S.T. 1986r. Analytical reports of Dithane and ETU residues for onion samples. Report 31A-86-17, includes 85-0310, 85-0176, 85-0274, 85-0275, 85-0276, 85-0403, 85-0404, 85-0512. Rohm and Haas Company, USA. Unpublished.

99. Satterthwaite, S.T. 1986s. Analytical reports of mancozeb and ETU residues for barley samples. Report 31A-86-18, includes 85-0272, 85-0273, 85-0351, 85-0352. Rohm and Haas Company, USA. Unpublished.

100. Satterthwaite, S.T. 1986t. Analytical reports of Dithane and ETU for pear residue samples. Report 31A-86-19, includes 85-0223, 85-0224. Rohm and Haas Company, USA. Unpublished.

101. Satterthwaite, S.T. 1986u. Analytical reports of mancozeb and ETU residues for papaya samples. Report 31A-86-22, includes 85-0206, 85-0594, 85-0625, 85-0632, 85-0638. Rohm and Haas Company, USA. Unpublished.

102. Satterthwaite, S.T. 1986v. Analytical reports of mancozeb and ETU residues for peanut samples. Report 31A-86-26, includes 74-171-02, 74-180-02, 84-0383, 84-0452, 84-0454, 85-0002. Rohm and Haas Company, USA. Unpublished.

103. Satterthwaite, S.T. 1986w. Analytical reports of maneb, mancozeb and ETU residues for ginseng samples. Report 31A-86-73, includes 86-0321, 86-0354, 86-0322. Rohm and Haas Company, USA. Unpublished.

104. Satterthwaite, S.T. 1986x. Mancozeb and ETU residues in tomato. Report 31A-86-94, includes 85-0368, 85-0369, 85-0554, 85-0555. Rohm and Haas Company, USA. Unpublished.

105. Satterthwaite, S.T. 1987a. Dithane M-45 residues in pear. Report 31A-87-18, includes 85-0315. Rohm and Haas Company, USA. Unpublished.

106. Satterthwaite, S.T. 1987b. Dithane M-45 residues in asparagus. Report 31A-87-19, includes 86-0083. Rohm and Haas Company, USA. Unpublished.

107. Satterthwaite, S.T. 1987c. Dithane M-45 residues in ginseng. Report 31A-87-41, includes 87-0215. Rohm and Haas Company, USA. Unpublished.

108. Satterthwaite, S.T. 1987d. Dithane M-45™ fungicide residues in tomato. Report 31A-87-50, includes 85-0555. Rohm and Haas Company, USA. Unpublished.

109. Satterthwaite, S.T. 1987e. Dithane F-45™ fungicide and ETU residues in asparagus. Report 31A-87-68, includes 86-0084, 86-0085. Rohm and Haas Company, USA. Unpublished.

110. Satterthwaite, S.T. 1988a. Dithane™ fungicide residues in tomato. Report 34A-88-08, includes 86-0596. Rohm and Haas Company, USA. Unpublished.

111. Satterthwaite, S.T. 1988b. Dithane™ fungicide residues in oranges. Report 34A-88-12, includes 86-0495, 86-0599. Rohm and Haas Company, USA. Unpublished.

112. Satterthwaite, S.T. 1988c. Dithane fungicide and ETU residues in cucumber. Report 34A-88-21, includes 86-0560, 86-0645. Rohm and Haas Company, USA. Unpublished.

113. Satterthwaite, S.T. 1988d. Mancozeb and ETU residues in bananas. Report 34A-88-22, includes 88-0029, 88-0030, RH-04-88. Rohm and Haas Company, USA. Unpublished.

114. Satterthwaite, S.T. 1988e. Analytical reports of Dithane fungicide and ETU residues in lemon samples. Report 34A-88-23, includes 87-0017, 87-0024. Rohm and Haas Company, USA. Unpublished.

115. Satterthwaite, S.T. 1988f. Dithane™ fungicide and ETU residues in lemon. Report 34A-88-34, includes 87-0018. Rohm and Haas Company, USA. Unpublished.

116. Satterthwaite, S.T. 1988g. Dithane® fungicide and ETU residues in lime. Report 34A-88-38, includes 87-0019. Rohm and Haas Company, USA. Unpublished.

117. Satterthwaite, S.T. 1988h. Mancozeb and ETU residues in limes. Report 34A-88-48, includes 87-0020. Rohm and Haas Company, USA. Unpublished.

118. Satterthwaite, S.T. 1988i. Mancozeb and ETU residues in tomatoes. Report 34A-88-51, includes 88-0058. Rohm and Haas Company, USA. Unpublished.

119. Satterthwaite, S.T. 1988j. Mancozeb and ETU residues in potatoes. Report 34A-88-52, includes 88-0059. Rohm and Haas Company, USA. Unpublished.

120. Satterthwaite, S.T. 1988k. Mancozeb and ETU residues in wheat. Report 34A-88-64, includes 88-0105, 88-0131. Rohm and Haas Company, USA. Unpublished.

121. Satterthwaite, S.T. 1988l. Mancozeb and ETU residues in wheat. Report 34A-88-65, includes 88-0185. Rohm and Haas Company, USA. Unpublished.

122. Satterthwaite, S.T. 1988m. Mancozeb and ETU residues in cranberries. Report 34A-88-67, includes 88-0282. Rohm and Haas Company, USA. Unpublished.

123. Satterthwaite, S.T. 1988n. Mancozeb and ETU residues in papaya. Report 34A-88-68, includes 88-0266. Rohm and Haas Company, USA. Unpublished.

124. Satterthwaite, S.T. 1988o. Mancozeb and ETU residues in bananas. Report 34A-88-71, includes 88-0040, RH-57-88. Rohm and Haas Company, USA. Unpublished.

125. Satterthwaite, S.T. 1988p. Mancozeb and ETU residues in cucumbers. Report 34A-88-78, includes 87-0482, 87-0045. Rohm and Haas Company, USA. Unpublished.

126. Satterthwaite, S.T. 1988q. Mancozeb and ETU residues in sweet corn. Report 34A-88-84, includes 87-0384. Rohm and Haas Company, USA. Unpublished.

127. Satterthwaite, S.T. 1988r. Mancozeb and ETU residues in onions. Report 34A-89-01, includes 85-0652, 85-0653. Rohm and Haas Company, USA. Unpublished.

128. Satterthwaite, S.T. 1988s. Mancozeb and ETU residues in onion. Report 34A-89-59, includes 88-0041. Rohm and Haas Company, USA. Unpublished.

129. Satterthwaite, S.T. 1989a. Analytical reports of Dithane and ETU for Brazil mango residue samples. Report 31A-87-03, includes 86-0047. Rohm and Haas Company, USA. Unpublished.

130. Satterthwaite, S.T. 1989b. Mancozeb and ETU residues in lemons. Report 34A-88-45, includes 86-0148, 86-0149. Rohm and Haas Company, USA. Unpublished.

131. Satterthwaite, S.T. 1989c. Analytical report of mancozeb and ETU residues in banana samples. Report 34A-89-23, includes 86-0091. Rohm and Haas Company, USA. Unpublished.

132. Satterthwaite, S.T. 1989d. Analytical report of mancozeb and ETU residues in dry bean samples. AR 34A-89-24, includes RH-11-89, 86-0200. Rohm and Haas Company, USA. Unpublished.

133. Satterthwaite, S.T. 1990a. Mancozeb and ETU residues in processed grapes. Report 34A-89-26. Rohm and Haas Company, USA. Unpublished.

134. Satterthwaite, S.T. 1990b. Mancozeb and ETU residues in orange. Report 34A-90-08, includes 86-0134, 87-0025, 87-0040. Rohm and Haas Company, USA. Unpublished.

135. Satterthwaite, S.T. 1990c. Mancozeb and ETU residues in almond. Report 34A-90-12, includes 89-0006, 89-0007, 89-0016, 89-0017, 89-0023. Rohm and Haas Company, USA. Unpublished.

136. Satterthwaite, S.T. 1990d. Mancozeb and ETU residues in celery. Report 34A-90-24, includes 89-0124. Rohm and Haas Company, USA. Unpublished.

137. Schweitzer, M.G. 1986a. Distribution and identification of

radiolabeled mancozeb metabolites in dairy goats. Report TR-31L-86-04. Rohm and Haas Company, USA. Unpublished.

138. Schweitzer, M.G. 1986b. Isolation and characterization of radiolabeled mancozeb metabolites in tissues of lactating dairy goats. Report TR-310-86-45. Rohm and Haas Company, USA. Unpublished.

139. Schweitzer, M.G. 1987. Water solubility of mancozeb. Report 31C-87-36. Rohm and Haas Company, USA. Unpublished.

140. Schweitzer, M.G. 1988. Commercial tomato processing study with tomatoes treated with mancozeb. Report 34C-88-04. Rohm and Haas Company, USA. Unpublished.

141. Schweitzer, M.G. 1989a. Mancozeb and ETU storage stability study on apple, tomato and wheat - final report. Report 34C-88-56. Rohm and Haas Company, USA. Unpublished.

142. Schweitzer, M.G. 1989b. Determination of the magnitude of the residue in sweet corn processed fractions prepared from corn treated with mancozeb. Report 34-89-04. Rohm and Haas Company, USA. Unpublished.

143. Schweitzer, M.G. 1989c. Determination of the magnitude of the residue due to mancozeb and ETU in potato processed fractions. Rohm and Haas Company, USA. Report 34-89-15. Unpublished.

144. Shields, R. 1990a. Determination of mancozeb in Faba beans and straw. Report 3137/88/5. Analchem Consultants, Australia. Unpublished.

145. Shields, R. 1990b. The determination of ETU in Faba beans and straw. Report 3137/88/5. Analchem Consultants, Australia. Unpublished.

146. Shields, R. 1990c. Determination of mancozeb in bananas. Report 2495/89/5. Analchem Consultants, Australia. Unpublished.

147. Shields, R. 1990d. The determination of ETU in bananas. Report 2495/89. Analchem Consultants, Australia. Unpublished.

148. Shields, R. 1990e. Determination of dithiocarbamate residues by carbon disulphide generation and GLC. Method PR4. Analchem Consultants, Australia. Unpublished.

149. Shields, R. 1990f. Determination of mancozeb residues in grapes. Report 867/90. Analchem Consultants, Australia. Unpublished.

150. Shields, R. 1990g. The determination of ETU in grapes. Report 868/90/05. Analchem Consultants, Australia. Unpublished.

151. Shields, R. 1990h. Determination of mancozeb residues in grapes. Report 868/90. Analchem Consultants, Australia. Unpublished.

152. Shields, R. 1991a. Determination of mancozeb residues in apples. Report 91/1121, relates to AUE-90-026. Analchem Consultants, Australia. Unpublished.

153. Shields, R. 1991b. Determination of mancozeb residues in potatoes. Report 1289/90. Analchem Consultants, Australia. Unpublished.

154. Shields, R. 1991c. The determination of ETU in potatoes. Report 1289/90/05. Analchem Consultants, Australia. Unpublished.

155. Shields, R. 1991d. Determination of mancozeb residues in mangoes. Report 90/3058. Analchem Consultants, Australia. Unpublished.

156. Shields, R. 1992a. Determination of mancozeb residues in passionfruit. Report 91/1282, relates to AUH-91-012. Analchem Consultants, Australia. Unpublished.

157. Shields, R. 1992b. Determination of mancozeb residues in onions.

Report 91/2499, relates to AUK-91-009. Analchem Consultants, Australia. Unpublished.

158. Shields, R. 1992c. Determination of mancozeb residues in carrots. Report 91/2500, relates to AUK-91-008. Analchem Consultants, Australia. Unpublished.

159. Shields, R. 1992d. Determination of mancozeb residues in beans. Report 91/2502, relates to AUA-91-021. Analchem Consultants, Australia. Unpublished.

160. Shields, R. 1992e. Determination of mancozeb residues in almonds. Report 92/0287, relates to AUI-91-032. Analchem Consultants, Australia. Unpublished.

161. Shields, R. 1992f. Determination of mancozeb residues in apricots. Report 92/0288, relates to AUI-91-034. Analchem Consultants, Australia. Unpublished.

162. Shields, R. 1992g. Determination of mancozeb residues in oranges. Report 92/0960, relates to AUI-92-001. Analchem Consultants, Australia. Unpublished.

163. Shields, R. 1992h. Determination of mancozeb residues in pears. Report 92/1111, relates to AUE-91-026. Analchem Consultants, Australia. Unpublished.

164. Shields, R. 1992i. Determination of mancozeb residues in peaches. Report 92/1112, relates to AUE-91-027. Analchem Consultants, Australia. Unpublished.

165. Shields, R. 1992j. Determination of mancozeb residues in pumpkins. Report 92/1155, relates to AUK-92-04. Analchem Consultants, Australia. Unpublished.

166. Shields, R. 1992k. Determination of mancozeb residues in cucumbers. Report 92/1156, relates to AUK-92-003. Analchem Consultants, Australia. Unpublished.

167. Shields, R. 1992l. Determination of mancozeb residues in watermelons. Report 92/1157, relates to AUK-92-005. Analchem Consultants, Australia. Unpublished.

168. Shields, R. 1992m. Determination of mancozeb residues in silverbeet. Report 92/1382, relates to AUK-92-007. Analchem Consultants, Australia. Unpublished.

169. Shields, R. 1992n. Determination of mancozeb residues in peanut kernels and foliage. Report 92/1383, relates to AUK-92-008. Analchem Consultants, Australia. Unpublished.

170. Shields, R. 1992o. Determination of mancozeb residues in zucchini. Report 92/1384, relates to AUK-92-06. Analchem Consultants, Australia. Unpublished.

171. Slesinski, R.S. 1990. EBDC/ETU national food survey. Project ETU 89-01. Report ETU 90-09. Technical Assessment Systems, Inc., USA. Unpublished.

172. Smith, S., Jr. 1986a. Metabolism study of C-14 radiolabeled mancozeb in laying hens. Report TR-31L-86-07. Rohm and Haas Company, USA. Unpublished.

173. Smith, S., Jr. 1986b. Addendum to metabolism study of C-14 radiolabeled mancozeb in laying hens. Report TR-310-86-52. Rohm and Haas Company, USA. Unpublished.

174. Velthuis, R. 1991. Mancozeb residue determinations - faba beans. Trial AUA-91-021. DuPont (Australia) Ltd. Unpublished.

175. Wasser, C. 1993a. Determination of residues of mancozeb in garlic in France in 1990. Report RF 0062-1, includes R77.32. Anadiag, S.A., France. Unpublished.

176. Wasser, C. 1993b. Determination of residues of mancozeb in witloof in France in 1990. Report RF 0062-2, includes R80.9. Anadiag, S.A., France. Unpublished.

177. Wasser, C. 1993c. Determination of residues of mancozeb in leeks in France in 1990. Report RF 0062-3, includes R77.49, R80.11. Anadiag, S.A., France. Unpublished.

178. Wasser, C. 1993d. Determination of residues of mancozeb in carrots in France in 1990. Report RF 0062-4, includes R77.33, R77.34, R77.50. Anadiag, S.A., France. Unpublished.

179. Wasser, C. 1993e. Determination of residues of mancozeb in tomatoes in France in 1990. Report RF 0062-5, includes R77.35/36/37, R77.38/39/40, R77.41/42/43, R77.44/45/46, R78.53, R80.8. Anadiag, S.A., France. Unpublished.

180. Wasser, C. 1993f. Determination of residues of mancozeb in potatoes in France in 1990. Report RF 0062-7, includes R78.4, R78.5, R78.6, R78.10, R78.22, R78.23, R78.25, R79.27/28. Anadiag, S.A., France. Unpublished.

181. Wasser, C. 1993g. Determination of residues of mancozeb in cereals in France in 1990. Report RF 0062-8, includes R78.17, R78.18, R78.19, R78.20, R78.21. Anadiag, S.A., France. Unpublished.

182. Wasser, C. 1993h. Determination of residues of mancozeb in apples in France in 1990. Report RF 0062-9, includes R78.13, R78.14, R78.15/16, R78.30, R78.32, R78.34, R78.50/51/52, R78.58, R78.67, R78.69, R78.70, R79.4, R79.56/57, R79.59, R79.60. Anadiag, S.A., France. Unpublished.

183. Wasser, C. 1993i. Determination of residues of mancozeb in pears in France in 1990. Report RF 0062-10, includes R78.36, R78.38, R78.40, R78.42. Anadiag, S.A., France. Unpublished.

184. Wasser, C. 1993j. Determination of residues of mancozeb in citrus in Spain in 1990. Report RF 0062-11, includes R80.4, R80.5, R80.7. Anadiag, S.A., France. Unpublished.

185. Wasser, C. 1993k. Determination of residues of mancozeb in plums in France in 1990. Report RF 0062-12, includes R78.54/55, R78.56, R78.59. Anadiag, S.A., France. Unpublished.

186. Wasser, C. 1993l. Determination of residues of mancozeb in wine in France in 1988. Report RF 0062-13, includes R78.78, R78.89, R79.1. Anadiag, S.A., France. Unpublished.

187. Wasser, C. 1993m. Determination of residues of mancozeb in wine in France in 1989. Report RF 0062-14, includes R78.82, R78.85. Anadiag, S.A., France. Unpublished.

188. Wasser, C. 1993n. Determination of residues of mancozeb in grapes in France in 1990. Report RF 0062-15, includes R78.44, R78.46, R78.57, R78.62/63, R78.64, R78.65, R78.66, R78.68, R78.71, R79.5, R79.8, R79.13, R79.16, R79.26, R79.29, R79.30, R79.50. Anadiag, S.A., France. Unpublished.

189. Wasser, C. 1993o. Determination of residues of mancozeb in tomatoes in Portugal in 1990. Report RF 0062-16, includes R79.53, R79.54. Anadiag, S.A., France. Unpublished.

190. Wasser, C. 1993p. Determination of residues of mancozeb in tomatoes in Spain in 1990. Report RF 0062-17, includes R79.19, R79.20, R80.10. Anadiag, S.A., France. Unpublished.

191. Wasser, C. 1993q. Determination of residues of mancozeb in asparagus

in France in 1990. Report RF 0062-18, includes R77.30, R77.31, R77.47. Anadiag, S.A., France. Unpublished.

192. Wasser, C. 1993r. Determination of residues of mancozeb in asparagus in France in 1991. Report RF 1038-1, includes R78.11, R78.12. Anadiag, S.A., France. Unpublished.

193. Westberg, G.L. 1989a. Determination of ethylene bis dithiocarbamates (EBDCs) in crops and processed commodities. Method ETU-89AM-001. Morse Laboratories, Inc., USA. Unpublished.

194. Westberg, G.L. 1989b. Determination of ethylene bis dithiocarbamates (EBDCs) in meat. Method ETU-89AM-002. Morse Laboratories, Inc., USA. Unpublished.

195. Westberg, G.L. 1989c. Determination of ethylene bis dithiocarbamates (EBDCs) in milk. Method ETU-89AM-003. Morse Laboratories, Inc., USA. Unpublished.

196. Wright, R.M. 1991. Mancozeb residue trial on mangoes. DPI 6.2.91, relates to 90/3058. Department of Primary Industries, Queensland Government, Australia. Unpublished.

197. Yeh, S.M. 1985. Distribution of radiolabeled mancozeb metabolites in soybeans. Report TR-31L-85-18. Rohm and Haas Company, USA. Unpublished.

198. Yeh, S.M. 1986a. Distribution of radiolabeled mancozeb metabolites in sugar beets. Report TR-31L-86-08. Rohm and Haas Company, USA. Unpublished.

199. Yeh, S.M. 1986b. Addendum to TR-31L-85-18 - Distribution of ^{14}C radiolabeled mancozeb metabolites in soybeans. Report 310-86-55. Rohm and Haas Company, USA. Unpublished.

200. Yess, N.J., Gunderson, E.L. and Roy, R.R. 1993. US Food and Drug Administration monitoring of pesticides residues in infant foods and adult foods eaten by infants/children. J. Assoc. Off. Analyt. Chem., 76, 492-507.

201. Zalewski, J. and Edwards, N. 1992. Laboratory report - ETU in tomato paste. Report P92/ . ARL Australia. Unpublished.

Cross-index of report numbers, study numbers and references.

Reports and studies are listed in numerical and alphabetical order and each is linked to a reference. Reference numbers are signified by #. Reports in the data Tables giving only summary data or without authors or titles are not included.

1289/90/05 #154
1289/90 #153
1289/90 #20
2495/89 #147
2495/89/5 #146
310-86-07 #84
310-86-08 #86
310-86-09 #88
310-86-10 #90
310-86-11 #91
310-86-12 #94
310-86-13 #60
310-86-14 #61
310-86-15 #62
310-86-16 #63
3137/88/5 #145
3137/88/5 #144
31A-86-06 #81
31A-86-06 #82
31A-86-07 #83
31A-86-08 #85
31A-86-09 #87
31A-86-10 #89
31A-86-11 #92
31A-86-12 #93
31A-86-13 #95
31A-86-14 #96
31A-86-16 #97
31A-86-17 #98
31A-86-18 #99
31A-86-19 #100
31A-86-22 #101
31A-86-26 #102
31A-86-73 #103
31A-86-94 #104
31A-87-03 #129
31A-87-18 #105
31A-87-19 #106
31A-87-41 #107
31A-87-50 #108
31A-87-68 #109
31C-87-36 #139
32232 #67
32865 #26
33552 #27
33553 #68
34-89-04 #142
34-89-15 #143
34-90-61 #9
34A-88-08 #110
34A-88-12 #111
34A-88-21 #112
34A-88-22 #113
34A-88-23 #114
34A-88-34 #115
34A-88-38 #116
34A-88-45 #130
34A-88-48 #117
34A-88-51 #118
34A-88-52 #119
34A-88-64 #120
34A-88-65 #121
34A-88-67 #122
34A-88-68 #123
34A-88-71 #124
34A-88-78 #125
34A-88-84 #126
34A-89-01 #127
34A-89-23 #131
34A-89-24 #132
34A-89-26 #133
34A-89-59 #128
34A-90-08 #134
34A-90-12 #135
34A-90-24 #136
34C-88-04 #140
34C-88-56 #141
74-171-02 #102
74-180-02 #102
77-0300 #15
78-0418 #16
83-0200 #85
83-0228 #85
83-0237 #85
83-0253 #85
83-0358 #85
83-0419 #85
84-0105 #78
84-0383 #102
84-0425 #79
84-0452 #102
84-0454 #102
85-0002 #102
85-0126 #89
85-0127 #95
85-0128 #95
85-0129 #96
85-0134 #83
85-0136 #83
85-0161 #87
85-0162 #87
85-0163 #89
85-0165 #92
85-0176 #98
85-0206 #101
85-0221 #81
85-0222 #81
85-0223 #100
85-0224 #100
85-0258 #81
85-0264 #93
85-0272 #99
85-0273 #99
85-0274 #98
85-0275 #98
85-0278 #83
85-0279 #81
85-0280 #87
85-0292 #93
85-0294 #97
85-0295 #97
85-0303 #81
85-0310 #98
85-0310 #95

85-0311 #95
85-0312 #95
85-0315 #105
85-0325 #89
85-0329 #93
85-0337 #85
85-0339 #89
85-0341 #97
85-0350 #92
85-0351 #99
85-0352 #99
85-0363 #93
85-0365 #93
85-0368 #104
85-0369 #104
85-0397 #92
85-0401 #92
85-0403 #98
85-0404 #98
85-0428 #95
85-0453 #85
85-0454 #92
85-0455 #92
85-0456 #97
85-0457 #97
85-0458 #97
85-0460 #96
85-0479 #96
85-0480 #96
85-0484 #95
85-0485 #96
85-0499 #93
85-0500 #93
85-0501 #93
85-0503 #92
85-0506 #81
85-0512 #98
85-0515 #93
85-0554 #104
85-0555 #108
85-0555 #104
85-0561 #92
85-0594 #101
85-0625 #101
85-0632 #101
85-0638 #101
85-0652 #127
85-0653 #127
86-0047 #129
86-0083 #106
86-0084 #109
86-0085 #109
86-0091 #131
86-0134 #134
86-0148 #130
86-0149 #130
86-0200 #36
86-0200 #132
86-0321 #103
86-0322 #103
86-0354 #103
86-0495 #111
86-0560 #112
86-0596 #110
86-0599 #111
86-0645 #112
867/90 #149
868/90 #45
868/90 #151
868/90/05 #150
87-0017 #114
87-0018 #115
87-0019 #116
87-0020 #117
87-0024 #114
87-0025 #134
87-0040 #134
87-0045 #125
87-0215 #107
87-0384 #126
87-0482 #125
88-0029 #113
88-0030 #113
88-0040 #124
88-0041 #128
88-0058 #118
88-0059 #119
88-0105 #120
88-0131 #120
88-0185 #121
88-0266 #123
88-0282 #122
89-0006 #135
89-0007 #135
89-0016 #135
89-0017 #135
89-0023 #135
89-0124 #136
89-0191 #9
90/3058 #196
90-113RA #25
90/3058 #155
90-0084ATO-1 #28
91/1121 #152
91/1282 #156
91/2499 #157
91/2500 #158
91/2502 #159
91-104 #25
92/0287 #160
92/0288 #161
92/0960 #162
92/1111 #163
92/1112 #164
92/1155 #165
92/1156 #166
92/1157 #167
92/1382 #168
92/1383 #169
92/1384 #170
AUA-91-021 #174
AUA-91-021 #159
AUE-90-002 #20
AUE-90-026 #152
AUE-90-026 #19
AUE-90-027 #22
AUE-91-026 #21
AUE-91-026 #163
AUE-91-027 #164
AUE-92-001 #23
AUH-91-012 #57
AUI-91-032 #160
AUI-91-032 #43
AUI-91-034 #161
AUI-91-034 #44
AUI-92-001 #162
AUK-91-008 #1
AUK-91-008 #158
AUK-91-009 #2
AUK-91-009 #157
AUK-92-003 #166
AUK-92-003 #3
AUK-92-004 #4
AUK-92-005 #5
AUK-92-005 #167
AUK-92-006 #6
AUK-92-007 #168
AUK-92-007 #7
AUK-92-008 #169
AUK-92-008 #8
AUK-92-04 #165
AUK-92-06 #170
DPI 6.2.91 #196
ETU-89AM-001 #193
ETU-89AM-002 #194
ETU-89AM-003 #195
ETU-89AM-004 #76
ETU-89AM-005 #77
ETU 89-01 #171
ETU 90-02 #28
ETU 90-06 #37
ETU 90-09 #171
ETU 90-11 #38
ETU 91-02 #39
LC 1507 #12
MTF-88AM-004 #75
P91/ #11
P92/ #201
PR20 #42
PR4 #148
R&H/BA 7.138/1991 #14
R77.30 #191
R77.31 #191
R77.32 #175
R77.33 #178
R77.34 #178
R77.35/36/37 #179
R77.38/39/40 #179
R77.41/42/43 #179
R77.44/45/46 #179
R77.47 #191
R77.49 #177
R77.50 #178
R78.10 #180
R78.11 #192
R78.12 #192
R78.13 #182
R78.14 #182
R78.15/16 #182
R78.17 #181
R78.18 #181
R78.19 #181
R78.20 #181
R78.21 #181
R78.22 #180
R78.23 #180
R78.25 #180
R78.30 #182
R78.32 #182
R78.34 #182
R78.36 #183
R78.4 #180
R78.40 #183
R78.42 #183
R78.44 #188
R78.46 #188
R78.5 #180
R78.50/51/52 #182
R78.53 #179
R78.54/55 #185
R78.56 #185
R78.57 #188
R78.58 #182
R78.59 #185
R78.6 #180
R78.60 #49
R78.61 #49
R78.62/63 #188
R78.64 #188
R78.65 #188
R78.66 #188
R78.67 #182
R78.68 #188
R78.69 #182
R78.70 #182
R78.71 #188
R78.78 #186
R78.82 #187
R78.85 #187
R78.89 #186
R79.1 #186
R79.13 #188
R79.16 #188
R79.19 #190
R79.20 #190
R79.26 #188
R79.27/28 #180
R79.29 #188
R79.30 #188
R79.31 #50
R79.32 #50
R79.4 #182
R79.41 #52
R79.42 #53
R79.43 #51
R79.44 #51
R79.45 #46
R79.5 #188
R79.50 #188
R79.53 #189
R79.54 #189
R79.55 #49
R79.56/57 #182
R79.58 #49
R79.59 #182
R79.60 #182
R79.61 #52
R79.63 #53
R79.65 #46
R79.73 #46
R79.8 #188
R80.1 #48
R80.10 #190
R80.11 #177
R80.13 #46
R80.2 #48
R80.24 #46
R80.27 #54
R80.3 #48
R80.30 #55
R80.31 #56
R80.32 #47
R80.33 #47
R80.34 #46
R80.35 #58
R80.36 #59
R80.4 #184
R80.5 #184
R80.7 #184
R80.8 #179
R80.9 #176
RF 0062-1 #175
RF 0062-10 #183
RF 0062-11 #184
RF 0062-12 #185
RF 0062-13 #186
RF 0062-14 #187
RF 0062-15 #188
RF 0062-16 #189
RF 0062-17 #190
RF 0062-18 #191
RF 0062-2 #176
RF 0062-3 #177
RF 0062-4 #178
RF 0062-5 #179
RF 0062-7 #180
RF 0062-8 #181
RF 0062-9 #182
RF 1038-1 #192
RF 1052-1 #46
RF 1052-10 #55
RF 1052-11 #56
RF 1052-2 #47
RF 1052-3 #48
RF 1052-4 #49
RF 1052-5 #50
RF 1052-6 #51
RF 1052-7 #52
RF 1052-8 #53
RF 1052-9 #54
RH-04-88 #32
RH-04-88 #33
RH-04-88 #113
RH-10-84 #78
RH-11-89 #36
RH-11-89 #132
RH-13-84 #79
RH-57-88 #124
RH-57-88 #35
RH 04-88 #34
TR-31L-85-17 #80
TR-31L-85-18 #199
TR-31L-85-18 #197
TR-31L-86-03 #70
TR-31L-86-03 #69
TR-31L-86-07 #172
TR-31L-86-04 #137
TR-31L-86-08 #198
TR-310-86-45 #138
TR-310-86-52 #173
TR-310-86-54 #70
TR-34-89-19 #41
TR36F-82-20 #17

1289/90/05 #154
1289/90 #153
1289/90 #20
2495/89 #147
2495/89/5 #146
310-86-07 #84
310-86-08 #86
310-86-09 #88
310-86-10 #90
310-86-11 #91
310-86-12 #94
310-86-13 #60
310-86-14 #61
310-86-15 #62
310-86-16 #63
3137/88/5 #145
3137/88/5 #144
31A-86-06 #81
31A-86-06 #82
31A-86-07 #83
31A-86-08 #85
31A-86-09 #87
31A-86-10 #89
31A-86-11 #92
31A-86-12 #93
31A-86-13 #95
31A-86-14 #96
31A-86-16 #97
31A-86-17 #98
31A-86-18 #99
31A-86-19 #100
31A-86-22 #101
31A-86-26 #102
31A-86-73 #103
31A-86-94 #104
31A-87-03 #129
31A-87-18 #105
31A-87-19 #106
31A-87-41 #107
31A-87-50 #108
31A-87-68 #109
31C-87-36 #139
32232 #67
32865 #26
33552 #27
33553 #68
34-89-04 #142
34-89-15 #143
34-90-61 #9
34A-88-08 #110
34A-88-12 #111
34A-88-21 #112
34A-88-22 #113
34A-88-23 #114
34A-88-34 #115
34A-88-38 #116
34A-88-45 #130
34A-88-48 #117
34A-88-51 #118
34A-88-52 #119
34A-88-64 #120
34A-88-65 #121
34A-88-67 #122
34A-88-68 #123
34A-88-71 #124
34A-88-78 #125
34A-88-84 #126
34A-89-01 #127
34A-89-23 #131
34A-89-24 #132
34A-89-26 #133
34A-89-59 #128
34A-90-08 #134
34A-90-12 #135
34A-90-24 #136
34C-88-04 #140
34C-88-56 #141
74-171-02 #102
74-180-02 #102
77-0300 #15
78-0418 #16
83-0200 #85
83-0228 #85
83-0237 #85
83-0253 #85
83-0358 #85
83-0419 #85
84-0105 #78
84-0383 #102
84-0425 #79
84-0452 #102
84-0454 #102
85-0002 #102
85-0126 #89
85-0127 #95
85-0128 #95
85-0129 #96
85-0134 #83
85-0136 #83
85-0161 #87
85-0162 #87
85-0163 #89
85-0165 #92
85-0176 #98
85-0206 #101
85-0221 #81
85-0222 #81
85-0223 #100
85-0224 #100
85-0258 #81
85-0264 #93
85-0272 #99
85-0273 #99
85-0274 #98
85-0275 #98
85-0278 #83
85-0279 #81
85-0280 #87
85-0292 #93
85-0294 #97
85-0295 #97
85-0303 #81
85-0310 #98
85-0310 #95
85-0311 #95
85-0312 #95
85-0315 #105
85-0325 #89
85-0329 #93
85-0337 #85
85-0339 #89
85-0341 #97
85-0350 #92
85-0351 #99
85-0352 #99
85-0363 #93
85-0365 #93
85-0368 #104
85-0369 #104
85-0397 #92
85-0401 #92
85-0403 #98
85-0404 #98
85-0428 #95
85-0453 #85
85-0454 #92
85-0455 #92
85-0456 #97
85-0457 #97
85-0458 #97
85-0460 #96
85-0479 #96
85-0480 #96
85-0484 #95
85-0485 #96
85-0499 #93
85-0500 #93
85-0501 #93
85-0503 #92
85-0506 #81
85-0512 #98
85-0515 #93
85-0554 #104
85-0555 #108
85-0555 #104
85-0561 #92
85-0594 #101
85-0625 #101
85-0632 #101
85-0638 #101
85-0652 #127
85-0653 #127
86-0047 #129
86-0083 #106
86-0084 #109
86-0085 #109
86-0091 #131
86-0134 #134
86-0148 #130
86-0149 #130
86-0200 #36
86-0200 #132
86-0321 #103
86-0322 #103
86-0354 #103
86-0495 #111
86-0560 #112
86-0596 #110
86-0599 #111
86-0645 #112
867/90 #149
868/90 #45
868/90 #151
868/90/05 #150
87-0017 #114
87-0018 #115
87-0019 #116
87-0020 #117
87-0024 #114
87-0025 #134
87-0040 #134
87-0045 #125
87-0215 #107
87-0384 #126
87-0482 #125
88-0029 #113
88-0030 #113
88-0040 #124
88-0041 #128
88-0058 #118
88-0059 #119
88-0105 #120
88-0131 #120
88-0185 #121
88-0266 #123
88-0282 #122
89-0006 #135
89-0007 #135
89-0016 #135
89-0017 #135
89-0023 #135
89-0124 #136
89-0191 #9
90/3058 #196
90-113RA #25
90/3058 #155
90-0084ATO-1 #28
91/1121 #152
91/1282 #156
91/2499 #157
91/2500 #158
91/2502 #159
91-104 #25
92/0287 #160
92/0288 #161
92/0960 #162
92/1111 #163
92/1112 #164
92/1155 #165
92/1156 #166
92/1157 #167
92/1382 #168
92/1383 #169
92/1384 #170
AUA-91-021 #174
AUA-91-021 #159
AUE-90-002 #20
AUE-90-026 #152
AUE-90-026 #19
AUE-90-027 #22
AUE-91-026 #21
AUE-91-026 #163
AUE-91-027 #164
AUE-92-001 #23
AUH-91-012 #57
AUI-91-032 #160
AUI-91-032 #43
AUI-91-034 #161
AUI-91-034 #44
AUI-92-001 #162
AUK-91-008 #1
AUK-91-008 #158
AUK-91-009 #2
AUK-91-009 #157

AUK-92-003 #166
AUK-92-003 #3
AUK-92-004 #4
AUK-92-005 #5
AUK-92-005 #167
AUK-92-006 #6
AUK-92-007 #168
AUK-92-007 #7
AUK-92-008 #169
AUK-92-008 #8
AUK-92-04 #165
AUK-92-06 #170
DPI 6.2.91 #196
ETU-89AM-001 #193
ETU-89AM-002 #194
ETU-89AM-003 #195
ETU-89AM-004 #76
ETU-89AM-005 #77
ETU 89-01 #171
ETU 90-02 #28
ETU 90-06 #37
ETU 90-09 #171
ETU 90-11 #38
ETU 91-02 #39
LC 1507 #12
MTF-88AM-004 #75
P91/ #11
P92/ #201
PR20 #42
PR4 #148
R&H/BA 7.138/1991 #14
R77.30 #191
R77.31 #191
R77.32 #175
R77.33 #178
R77.34 #178
R77.35/36/37 #179
R77.38/39/40 #179
R77.41/42/43 #179
R77.44/45/46 #179
R77.47 #191
R77.49 #177
R77.50 #178
R78.10 #180
R78.11 #192
R78.12 #192
R78.13 #182
R78.14 #182
R78.15/16 #182
R78.17 #181
R78.18 #181
R78.19 #181
R78.20 #181
R78.21 #181
R78.22 #180
R78.23 #180
R78.25 #180
R78.30 #182
R78.32 #182
R78.34 #182
R78.36 #183
R78.4 #180
R78.40 #183
R78.42 #183
R78.44 #188
R78.46 #188
R78.5 #180
R78.50/51/52 #182
R78.53 #179
R78.54/55 #185
R78.56 #185
R78.57 #188
R78.58 #182
R78.59 #185
R78.6 #180
R78.60 #49
R78.61 #49
R78.62/63 #188
R78.64 #188
R78.65 #188
R78.66 #188
R78.67 #182
R78.68 #188
R78.69 #182
R78.70 #182
R78.71 #188
R78.78 #186
R78.82 #187
R78.85 #187
R78.89 #186
R79.1 #186
R79.13 #188
R79.16 #188
R79.19 #190
R79.20 #190
R79.26 #188
R79.27/28 #180
R79.29 #188
R79.30 #188
R79.31 #50
R79.32 #50
R79.4 #182
R79.41 #52
R79.42 #53
R79.43 #51
R79.44 #51
R79.45 #46
R79.5 #188
R79.50 #188
R79.53 #189
R79.54 #189
R79.55 #49
R79.56/57 #182
R79.58 #49
R79.59 #182
R79.60 #182
R79.61 #52
R79.63 #53
R79.65 #46
R79.73 #46
R79.8 #188
R80.1 #48
R80.10 #190
R80.11 #177
R80.13 #46
R80.2 #48
R80.24 #46
R80.27 #54
R80.3 #48
R80.30 #55
R80.31 #56
R80.32 #47
R80.33 #47
R80.34 #46
R80.35 #58
R80.36 #59
R80.4 #184
R80.5 #184
R80.7 #184
R80.8 #179
R80.9 #176
RF 0062-1 #175
RF 0062-10 #183
RF 0062-11 #184
RF 0062-12 #185
RF 0062-13 #186
RF 0062-14 #187
RF 0062-15 #188
RF 0062-16 #189
RF 0062-17 #190
RF 0062-18 #191
RF 0062-2 #176
RF 0062-3 #177
RF 0062-4 #178
RF 0062-5 #179
RF 0062-7 #180
RF 0062-8 #181
RF 0062-9 #182
RF 1038-1 #192
RF 1052-1 #46
RF 1052-10 #55
RF 1052-11 #56
RF 1052-2 #47
RF 1052-3 #48
RF 1052-4 #49
RF 1052-5 #50
RF 1052-6 #51
RF 1052-7 #52
RF 1052-8 #53
RF 1052-9 #54
RH-04-88 #32
RH-04-88 #33
RH-04-88 #113
RH-10-84 #78
RH-11-89 #36
RH-11-89 #132
RH-13-84 #79
RH-57-88 #124
RH-57-88 #35
RH 04-88 #34
TR-31L-85-17 #80
TR-31L-85-18 #199
TR-31L-85-18 #197
TR-31L-86-03 #70
TR-31L-86-03 #69
TR-31L-86-07 #172
TR-31L-86-04 #137
TR-31L-86-08 #198
TR-310-86-45 #138
TR-310-86-52 #173
TR-310-86-54 #70
TR-34-89-19 #41
TR36F-82-20 #17

MANEB

EXPLANATION

Maneb was first evaluated in 1967. The MRLs for dithiocarbamates, including Ümaneb, were consolidated into a combined list in 1977.

Maneb was scheduled for re-evaluation in 1993 in the CCPR periodic review programme.

The Meeting was provided with information on use patterns, supervised residue trials, fate of residues and miscellaneous studies by the manufacturer. GAP information was provided by Canada, Germany and Spain.

IDENTITY

ISO common name: Maneb
Chemical name:

IUPAC Manganese ethylenebis(dithiocarbamate)

CA [1,2-ethanediylbis(carbamodithioato)(2-)]manganese

CAS No: 12427-38-2
CIPAC No: CIPAC-61
Synonyms:

Empirical formula: $C_4H_6MnN_2S_4$

Structural formula:

$$[\text{-MnSC(:S)NHCH}_2\text{CH}_2\text{NHC(:S)S-}]_x$$

Molecular weight per monomer unit: 265.3

Physical and chemical properties

Physical state: greenish yellow powder with a characteristic odour.
Vapour pressure: negligible at 25°C.
Solubility: insoluble in water and most organic solvents.
Melting point: decomposes at approximately 198°C before melting.
Bulk density: 450 kg/m^3

USE PATTERN

Maneb is effective against a broad spectrum of fungi and fungal plant diseases. It is registered in many countries for use on agricultural and horticultural crops. The Meeting was

aware of the registered uses summarized in Table 1.

Table 1. Registered uses of maneb.

CROP	COUNTRY	APPLICATION	PHI, days

		Max no.	Rate per applic. kg ai/ha	Spray concn. kg ai/hl	
Almonds	USA	4	5.4-7.2	0.14-0.19	145
Apple	France			0.16	
Apple	Germany	3	0.24	[1] 0.016	
Apple	Netherlands			[1] 0.16 [2] 0.08	56
Apple	USA	4 7	[1] 5.4 [3] 2.7	[1] 0.14 [3] 0.072	77
Apricot	Canada	2	7.2	2.0	
Asparagus	Netherlands		2.4-3.2		
Banana	Spain		1.6		8
Banana	USA	10	1.8-2.7		0
Barley	UK	2	1.6		
Beans	France		2.0		21
Beans	Netherlands		3.2		
Beans	Spain			0.2	8
Beans (dry)	USA	6	1.3-1.8		30
Berries	Netherlands	2		0.28	15
Broccoli	USA	6	1.3-1.8		7
Brussels sprouts	USA	6	1.3-1.8		7
Cabbage	Canada		1.8		7
Cabbage	USA	6	1.3-1.8		7
Carrots	Canada		0.9-2.6		5
Carrots	Germany	3	0.11		35
Cauliflower	USA	6	1.3-1.8		7
Celeriac	Germany	3	0.09		28
Celery	Canada		0.9-2.6		14
Celery	France		1.6		
Celery	Spain			0.2-0.3	8
Cherries	France		0.16		
Chinese cabbage (tight head)	USA		1.3-1.8		7
Chinese cabbage (loose head)	USA		0.9-1.3		7
Cranberry	USA	3	4.3-5.4		30
Cucumber	Canada		0.9-2.6		14
Cucumber	USA	8	1.3-1.8		5
Egg plant	USA	7	1.3-1.8		5
Endive	Canada		1.8		7
Endive	USA	6	1.3-1.8		10
Figs, kadota	USA	1	2.7	0.072	10
Garlic	France		1.6		15
Grapes, wine	France		0.28	0.28	
Grapes, wine	Italy			0.16-0.32	
Grapes	Spain	4	1	0.2	15
Grapes	USA	6	1.3-3.6	0.036-0.1	66
Hops	Germany	2		0.018-0.036	
Hops	Spain			0.2	15
Kale	USA	2	1.3-1.8		10
Kohlrabi	USA	6	1.3-1.8		7
Lettuce	Canada		1.8		7
Lettuce	France		1.6		45
Lettuce	UK	5	1.6		21
Lettuce (head and leaf)	USA	6	1.3-1.8		10
Maize	Spain			0.2	8
Melons	France		2.0		3
Melons	Spain			0.2-0.4	8
Melons (honeydew, cantaloupe)	USA	8	0.9-1.8		5
Onion	Canada		0.9-2.6		10
Onion	Netherlands		1.6		28
Onion	Spain			0.2-0.3	8
Onion (dry bulb)	USA	10	1.8-2.7		7
Onion (green)	USA	7	2.7		7
Onion	USA		2.7 furrow drench		
Papaya	USA	14	1.8-2.2	0.05-0.06	0
Peach	Canada	2	7.2	2.0	
Peppers	USA	6	1.3-2.7		7
Plums	France			0.16	
Pome fruits	Germany	3	0.24	[1] 0.016	
Popcorn	USA	15	1.3		7

CROP	COUNTRY	APPLICATION			PHI, days
		Max no.	Rate per applic. kg ai/ha	Spray concn. kg ai/hl	
Potato	Canada		0.9-1.8		1
Potato	France		1.6		
Potato	Germany	4	0.96-1.4		7
Potato	Netherlands	3	1.6		
Potato	Spain	4	2	0.2	15
Potato	UK	8	1.4		7
Potato	USA	7	1.3-1.8		3, 14
Pumpkins	USA	8	1.3-1.8		5
Red pepper	Spain			0.2-0.3	8
Strawberry	Spain	4	gf 3	0.2	15
Sugar beet	Canada		1.8		14
Sugar beet	Spain	4	0.6	0.2	15
Sugar beet	USA	7	1.3-1.8		14
Summer squash	USA	8	1.3-1.8		5
Sweet corn	USA	15	1.3		7
Tomato	Canada		1.8-2.6		7
Tomato	France		2		15
Tomato	Germany	4	1.4-2.9		14
Tomato	Italy		1.6-3.2		28
Tomato	Spain			0.2-0.3	8
Tomato	Netherlands			0.08-0.16	
Tomato	UK	5	1.8		7 g 2
Tomato	USA	7	gf[4] 1.3-2.7		5
Tree fruit	Spain	4	3	0.2	15
Vegetables	Spain	4	gf 3	0.2	15
Watermelon	USA	8	0.9-1.8		5
Wheat	France		3.2		
Wheat	Netherlands	2	1.6		28
Wheat	UK	2	1.6		
Winter squash	USA	8	1.3-1.8		5

[1] pre-blossom. [2] post-blossom. [3] extended application. [4] gf: glasshouse and field use. g: glasshouse use.

RESIDUES RESULTING FROM SUPERVISED TRIALS

Maneb and ETU (ethylenethiourea) residue data from supervised maneb trials on horticultural and agricultural crops are summarized in Tables 2 to 17.

Table 2. Apples. Netherlands, USA.
Table 3. Peaches. USA.
Table 4. Grapes. USA.
Table 5. Onions. Netherlands, USA.
Table 6. Brassica vegetables. USA.
Table 7. Cucurbits, USA.
Table 8. Leafy vegetables. Canada, USA.
Table 9. Beans. USA.
Table 10. Fruiting vegetables. Canada, Netherlands, USA.
Table 11. Root and tuber vegetables. Netherlands, UK, USA.
Table 12. Celery. Canada, USA.
Table 13. Cereal grains. Netherlands, UK, USA.
Table 14. Almonds. USA.
Table 15. Cereal straw and fodder. Netherlands, UK, USA.
Table 16. Bean vines. USA.
Table 17. Miscellaneous fodder commodities (almond hulls, sugar beet tops). USA.

Most supervised residue trials were fully or adequately described. Residues reported in the Tables are not adjusted for analytical recoveries. Analytical recoveries were mostly high (>80%) for dithiocarbamates, and were generally acceptable (>70%) for ETU, so using adjusted or unadjusted results should not influence the interpretations. Attention is drawn to cases where recoveries were less than 70%.

Dithiocarbamate residues are expressed as mg CS_2/kg throughout the Tables and text. EBDC is used as the abbreviation for ethylenebis(dithiocarbamates) in the Tables.

Where residues were not detected, results are recorded in the Tables as less than the limit of determination (LOD), e.g. <0.1 mg/kg. Residues have generally been rounded to 2 significant figures or, near the LOQ, to 1 significant figure. When residues were detected in control samples they are recorded in the Tables. In the majority of cases no residues were detected in control samples and are not recorded.

In apple trials in The Netherlands 5 trees constituetd a plot. Individual ETU recoveries in some trials were as low as 62-65%, but the mean was acceptable.

Barley plot sizes were 20-25 m^2 in The Netherlands. Wheat plot sizes varied from 25 m^2 to 1 ha. ETU recoveries from barley and wheat grain were often in the 60-70% range. Barley straw and wheat straw caused difficulties in the determination of ETU with some recoveries recorded as low as 22-35%.

Potato plots were 20-25 m^2 in supervised trials in The Netherlands. ETU recoveries from potatoes were marginally low. Plot sizes in the glasshouse tomato trials were 3.5 m^2 and ETU recoveries were again marginally low. Some onion trials in The Netherlands were on 20 m^2 plots and some plots were as small as 3.3 m^2. Analytical recoveries of ETU from onions were marginal.

Maneb was applied to celery, lettuce and tomatoes in the Canadian trials with a tractor-mounted sprayer. Plot sizes were the equivalent of 40-130 m of row.

In the US trials wherever aerial spraying was used the minimum plot size was 0.1 ha. Helicopter and fixed-wing aircraft were used to apply maneb in comparison trials on some commodities.

Plot sizes in US trials where maneb was applied by portable or ground equipment were usually 10 m^2 minimum, and mostly larger. For almonds and apples, plot size was 2-4 trees, with 4 replications.

Analytical recoveries of dithiocarbamates in the US trials were almost always quite good. Problems were experienced with bean vines (recoveries 59-104%), lettuce (62-116%), sweet corn (62-107%), sweet corn forage (42-105%), and watermelons (60-80%). Analytical recoveries of ETU were generally satisfactory; low ETU recoveries occurred in the analysis of bean vines (recoveries 58-93%), celery (30-84%, mean 65%), grapes (56-84%, mean 74%), lettuce (58-86%), sugar beet tops (61-84%), sweet corn forage (26-85%, mean 58%), sweet corn (64-84%), and tomatoes (59-92%). ETU residues were not determined in almond hulls because of the analytical difficulties.

CS_2 was evolved from control samples of onions (Table 5), and broccoli and cabbage (Table 6) during the dithiocarbamate method of analysis. Residues up to 0.5 mg/kg were recorded for onions, up to 0.55 mg/kg for broccoli and up to 0.59 mg/kg for cabbage. These results are comparable to residues reported in onions and broccoli (Pennwalt studies BR-88-15 and BR-89-09).

Bulb onions (Pennwalt study BR-88-15) and broccoli (Pennwalt study BR-89-09 and Rohm and Haas data) were shown to contain endogenous CS_2 or compounds which produced CS_2 in the dithiocarbamate analytical method. Twelve samples of bulb onions (10 varieties, 10 sites in the USA), certified to be untreated with dithiocarbamates, on analysis contained CS_2 residues ranging from undetectable (<0.03 mg/kg) to 0.13 mg/kg, median 0.05 mg/kg, while eight samples of broccoli (6 varieties, 6 sites in the USA) certified to be untreated with dithiocarbamates showed CS_2 residues ranging from undetectable (<0.01 mg/kg) to 0.79 mg/kg, median 0.32 mg/kg.

Kallio and Salorinne (1990) reported carbon disulphide as one of the

27 volatile compounds identified in the headspace analysis of onions.

Dithiocarbamate levels (as CS_2) in control kale and spinach samples from the USA (Table 8) ranged up to 0.57 mg/kg and 0.40 mg/kg respectively, and in control bean vines (Table 16) up to 1.8 mg/kg.

US trials on cabbages (Table 6) included analyses of trimmed and untrimmed samples, i.e. with and without inclusion of wrapper leaves. Dithiocarbamate residue levels in trimmed samples were, on average, about 70% of those in untrimmed samples, but there was wide variation.

Removal of wrapper leaves from lettuce (Table 8) substantially reduced dithiocarbamate residue levels.

Dithiocarbamate residues in washed spinach (Table 8) were about 25% lower than in unwashed spinach, but with considerable variation.

Table 2. Maneb residues (as CS_2) in apples from supervised trials in The Netherlands and the USA.

Country, year (Variety)	Application				Day	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
Netherlands, 1984 (Golden Delicious)	WP SC SC		0.12-0.16 0.12-0.16 0.12-0.16	10 10 10	49 49 49	0.10, <0.01 0.03, 0.11 <0.01 (2)	<0.002 (2) 0.0036, <0.002 <0.002 (2)	PH8410
Netherlands, 1984 (Golden Delicious)	WP SC SC		0.12-0.16 0.12-0.16 0.12-0.16	9 9 9	58 58 58	<0.01, 0.22 <0.01 (2) <0.01, 0.87	<0.002 (2) <0.002 (2) <0.002 (2)	PH8411
Netherlands, 1985 (Golden Delicious)	WP SC SC	1.2-1.6 1.2-1.6 1.2-1.6		9 9 9	81 81 81	0.55, <0.01 <0.01 (2) <0.01 (2)	0.008, 0.011 0.039, 0.014 0.009, 0.037	PH8510
Netherlands, 1985 (Golden Delicious)	WP SC SC	1.2-1.6 1.2-1.6 1.2-1.6		10 10 10	85 85 85	<0.01 (2) <0.01 (2) <0.01, 0.02	0.020, 0.032 0.009, <0.002 <0.002, 0.056	PH8512
Netherlands, 1986 (Golden Delicious)	WP SC SC SC	1.2-1.6 1.2-1.6 1.2-1.6 1.2-1.6		7 7 7 7	88 88 88 88	<0.01, 0.09 <0.01 (2) <0.01 (2) <0.01 (2)	<0.002 (2) <0.002 (2) <0.002 (2) <0.002 (2)	PH8610
Netherlands, 1987 (Golden Delicious)	WP SC SC	1.2-1.6 1.2-1.6 1.2-1.6		8 8 8	79 79 79	0.10, 0.08 0.12, 0.10 <0.02, 0.17	<0.002 (2) 0.0028, 0.002 0.002, 0.0033	PH8711
Netherlands, 1987 (Golden Delicious)	WP SC SC	1.2-1.6 1.2-1.6 1.2-1.6		10 10 10	81 81 81	0.04, 0.10 <0.02 (2) <0.02 (2)	<0.002, 0.012 <0.002, 0.0091 0.0036, 0.0077	PH8712
Netherlands, 1988 (Golden Delicious)	WP SC WG	1.2-1.6 1.2-1.6 1.2-1.6		9 9 9	71 71 71	0.14, 0.06 0.14, 0.24 <0.05 (2)	<0.001, 0.003 0.006, 0.004 0.004 (2)	PH8845
Netherlands, 1988 (Golden Delicious)	WP SC WG	1.2-1.6 1.2-1.6 1.2-1.6		9 9 9	>63 >63 >63	0.45, 0.39 0.34, 0.31 0.36, 0.48	0.007, 0.006 0.004, 0.005 0.007, 0.006	PH8847
USA (CA), 1988 (Newton Pippin)	SC	5.0	0.61	10	21 30	3.0, 2.9, 5.6, 7.9 5.4, 4.7, 4.1, 3.5	0.082, 0.075 (2), 0.058 0.11, 0.038, 0.088, 0.058	10A-88
USA (CA), 1988 (Newton Pippin)	SC	5.0	0.75	10	21 30	0.96, 0.68, 1.6, 0.64 1.4, 0.68, 0.61, 1.2	0.014, 0.01, <0.01 (2) 0.018, 0.013, <0.01 (2)	10B-88
USA (VA), 1988 (Golden Delicious)	SC	5.0	1.1	10	21 30	2.6 1.6	0.025 0.049	10C-88

Table 3. Maneb residues (as CS_2) in peaches from supervised trials in California (USA), 1988. All WP formulation and 7.2 kg ai/ha.

Variety			Day	Residues, mg/kg		Ref.
	kg ai/hl	No.		EBDC as CS_2	ETU	
Fairtimes	0.78	4	28	1.6, 0.86, 0.98, 1.1	<0.01(4)	39A-88
Fairtimes	8.0	a[1] 4	28	0.049, 0.057, 0.045, 0.045	<0.01(4)	39B-88
June Gold)	1.6	5	28	1.3	0.01	39C-88

[1] aerial application

Table 4. Maneb residues (as CS_2) in grapes from supervised trials in the USA. Underlined residues are from treatments according to GAP.

State, year (Variety)	Application				Day	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
CA, 1987 (Gewurtztraminer)	WP	3.6	0.77	5	8	3.4, 2.2, 4.2, 5.2	0.02, 0.01, 0.040, 0.02	048
CA, 1987 (Alicante)	WP	1.3	0.29	3	30	0.34, 0.26, 0.33, 0.34	<0.01 (4)	024
	WP	2.7	0.58	3	30	0.65, 0.53, 0.94, 0.58	<0.01 (4)	024
	WP	3.6	0.77	3	30	1.2, 0.90, 0.48, 0.79	<0.01 (4)	024
CA, 1990 (Thompson)	SC	3.6	0.4-0.76	4	66	0.65, 0.80, 0.60, 0.54	0.01, 0.01, <0.01 (2)	24A-90
CA, 1990 (Thompson Seedless)	SC	3.6	0.7	4	67	0.63, 1.3, 1.0, 1.8	0.01, 0.042, 0.038, 0.033	24B-90
NY, 1990 (Catawba)	SC	3.6	0.38	4	66	0.22	<0.01	24E-90
NY, 1990 (Aurora)	SC	3.6	0.38	4	66	0.21	<0.01	24F-90
CA, 1991 (Chardonnay)	SC	3.6	0.67	4	66	1.9	<0.01	24C-90
CA, 1991 (Pinot Noir)	SC	3.6	0.67	4	66	1.3	<0.01	24D-90

Table 5. Maneb residues (as CS_2) in onions from supervised trials in The Netherlands and the USA. Underlined residues are from treatments according to GAP.

CROP Country, year (Variety)	Application				Day	Residues, mg/kg [1]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
GREEN ONION								
USA (CA), 1987 (Green Bunching)	WP	1.8		7	7	0.57, 0.43, 1.1, 0.69 c <0.03	<0.01 (4) c <0.01	23387
USA (FL), 1987 (Tokoyo Bunching)	SC	1.8		7	7	0.45, 2.0, 1.4 c 0.09	0.02, 0.06, 0.05 c <0.01	23487
USA (TX), 1987 (White Eclipse)	SC	1.8	0.48	7	7	6.3, 7.4, 6.3 c 0.03	0.13, 0.14, 0.14 c <0.01	25487
USA (AZ), 1989 (Sweet Spanish)	SC	1.8		7	7	6.9, 6.9, 4.9 c 0.50	0.69, 0.58, 0.41 c <0.01	88137
BULB ONION								
Netherlands, 1984 (Jumbo)	WP WG SC SC	2.4 2.4 2.4 2.4	0.96 0.96 0.96 0.96	8 8 8 8	29 29 29 29	0.01 (2) 0.01 (2) 0.01 (2) 0.01 (2)	0.064, 0.002 0.017, 0.002 0.002 (2) 0.019, 0.002 (2)	PH8426
Netherlands, 1985 (Balstora)	WP SC	2.4 2.4	1.2 1.2	7 7	31 31	<0.01 (2) 0.10, <0.01	0.007, <0.002 0.006, <0.002	PH8523
Netherlands, 1985 (Jumbo)	WP SC	2.4 2.4	1.2 1.2	7 7	26 26	<0.01 (2) <0.01 (2)	0.002, <0.002 <0.002 (2)	PH8524
Netherlands, 1986	WP	2.4	1.2	7	42	0.02	<0.002	PH8623

CROP Country, year (Variety)	Application				Day	Residues, mg/kg [1]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
(Balstora)	SC	2.4	1.2	7	42	<0.01	<0.002	
USA (TX), 1988 (Ben Shamon)	SC	2.7	1.8	10	0	0.63 c 0.10	0.03 c <0.01	38A-88
USA (CA), 1988 (Ula)	SC	2.7	1.1	10	0	0.92, 0.52, 0.69, 0.86 c 0.13	0.05, 0.05, 0.05, 0.10 c <0.01	38B-88
USA (CO), 1988 (Winters)	SC	2.7	0.27	10	0	0.28 c <0.03	0.02 c <0.01	38C-88
USA (MI), 1988 (Sweet Sandwich)	SC	2.7	0.29	10	0	0.80 c 0.069	0.05 c <0.01	38D-88
USA (NY), 1988 (Early Yellow Globe)	SC	2.7	1.1	10	0	1.1 c <0.03	0.07 c <0.01	38E-88
USA (TX), 1988 (Ben Shamon)	SC	2.7	5.8	a 10	0	0.37 c 0.097	0.03 c <0.01	38F-88
USA(CA), 1988 (Yellow Bulb)	SC	2.7	2.9	a 10	0	0.57, 0.48, 0.11, 0.080 c 0.05	0.08, 0.05, 0.04, 0.01 c <0.01	38G-88
USA (CA), 1988 (Ula)	SC	2.7	1.1	10	0	0.80, 0.92, 1.8, 0.63 c 0.080	0.06, 0.07, 0.10, 0.04 c <0.01	38H-88
USA (OR), 1988 (Italian Red)	SC	2.7	0.58	10	0	0.97 c 0.057	0.06 c <0.01	38I-88
USA (OR), 1988 (Simco)	SC	2.7	0.58	10	0	0.46 c <0.03	0.10 c <0.01	38J-88
USA (ID), 1988 (Yellow Sweet Spanish)	SC	2.7	1.2	10	0	1.2 c <0.03	0.19 c <0.01	38K-88
USA (OH), 1988 (New Holland)	SC	2.7	1.2	10	0	0.34 c <0.03	0.02 c <0.01	38L-88

[1] c: control sample.

Table 6. Maneb residues (as CS_2) in Brassica vegetables from supervised trials in the USA. Underlined residues are from treatments according to GAP.

CROP State, year (Variety)	Application [1]				Day	Residues, mg/kg [2]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
BROCCOLI								
CA, 1987 (DeCicco)	WP	1.8	0.48	6	3	4.6, 3.0, 4.5, 3.3 c 0.15, 0.17	0.15, 0.12, 0.06, 0.09 c <0.01 (2)	25187
CA, 1989 (DeCicco)	SC	1.8	1.9	a 6	3	1.7, 1.9, 2.2, 1.4 c 0.42	0.028, 0.042, 0.064, 0.032 c <0.01	53A-89
CA, 1989 (Mercedes)	SC	1.8	0.96	a 6	4	0.80, 0.46, 0.75, 0.48 c 0.24	0.02, 0.01, 0.02, 0.01 c <0.01	53B-89
FL, 1989 (Green Comet)	SC	1.8	3.2	a 7	3	1.8 c 0.55	0.05 c <0.01	53C-89
CABBAGE								
MI, 1987 (Danish Ballhead)	WP	1.8		8	7	u 0.24, 0.19, 0.63 uc 0.14 t 0.17, 0.14, 0.097 tc 0.036	u <0.01 (3) uc <0.01 t <0.01 (3) tc <0.01	22587
NY, 1987 (King Cole Hybrid)	SC	1.8		6	7	u 0.49, 0.36, 0.75 uc 0.27 t 0.32, 0.44, 0.38 tc 0.37	u <0.01 (2), 0.01 uc <0.01 t <0.01 (3) tc <0.01	23287
CA, 1987 (Round Dutch)	WP	1.8		6	7	u 0.73, 0.77, 1.5 uc 0.24	u 0.050, 0.080, 0.090 uc <0.01	21587
TX, 1987 (Early Round Dutch)	SC	1.8		6	7	u 10, 5.0 uc 0.34 t 1.4, 1.3, 1.1 tc 0.33	u 0.077, 0.043 uc <0.01 t 0.02, <0.01, 0.01	21987
FL, 1987 (Abbot & Cobb)	WP	1.8		7	7	u 0.41, 0.35, 0.42 uc 0.44 t 0.80, 0.69, 0.51 tc 0.44	u <0.01 (3) uc <0.01 t 0.01 (2), <0.01 tc <0.01	21887
NY, 1989 (Bravo)	SC	2.7	5.1	a 4	21	wl 0.76, wo 0.42 c 0.28	wl <0.01, wo <0.01 c <0.01	43E-89
CA, 1989 (Head Start)	SC	2.7	1.4	a 4	20	wl 0.91, 0.65, 0.61, 0.76 wo 0.48, 0.34, 0.60, 0.29 c 0.31	wl 0.02, <0.01, 0.02, 0.02 wo <0.01 (4)	43J-89

CROP State, year (Variety)	Application [1]				Day	Residues, mg/kg [2]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
							c <0.01	
CA, 1989 (Copenhagen)	SC	2.7	6.0	a 4	21	wl 0.38, 0.67, 1.7, 0.59 wo 0.34, 0.44 c 0.083	wl 0.01, 0.01, 0.02, <0.01 wo <0.01 (2) c <0.01	43L-89
TX, 1990 (Baxter 1100)	SC	2.7	5.4	a 4	21	wl 0.77, wo 0.83 c 0.59	wl <0.01, wo <0.01 c <0.01	43B-89

[1] a: aerial application.
[2] c: control sample; u: untrimmed; t: trimmed; uc: untrimmed control; tc: trimmed control; wl: includes wrapper leaves; wo: without wrapper leaves.

Table 7. Maneb residues (as CS_2) in cucurbits from supervised trials in the USA. Underlined residues are from treatments according to GAP.

CROP State, year (Variety)	Application[1]				Day	Residues, mg/kg[2]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
CUCUMBER								
MI, 1987 (Marketmore 76)	WP	1.8		8	5	0.069, 0.04, 0.080	0.031, 0.032, 0.039	22687
NC, 1987 (Ashley)	WP	1.8		8	5	<0.03 (3)	0.01, <0.01, 0.01	22787
SC, 1987 (Dasher II)	SC	1.8		5	5	<0.03 (3)	0.01 (3)	22887
TX, 1987 (Galaxy)	SC	1.8		8	5	1.3, 1.2, 0.92	0.04 (3)	25387
CA, 1987	WP	1.8		7	5	0.10, 0.11, 0.13, 0.15	0.055, 0.047, 0.041, 0.046	25287
FL, 1987 (Dasher 2)	SC	1.8		8	5	0.75, 0.57, 0.57	0.088, 0.11, 0.065	21787
CA, 1989 (Burpless)	SC	1.8	1.9	a 8	5	0.072, 0.065, 0.046, 0.13	0.01, <0.01 (2), 0.01	51A-89
CA, 1989 (Ashley)	SC	1.8-2.2	1.8-3.8	a 8	5	0.28, 0.50, 0.73, 0.34	0.01, 0.02, 0.029, 0.02	51B-89
FL, 1989 (Dasher II)	SC	1.8	3.2	a 8	5	<0.03	0.03	51C-89
WATERMELON								
GA, 1987	SC	1.8	0.48	8	5	0.03, <0.03, 0.052	<0.01, 0.01, <0.01	22987
TX, 1987 (Charleston Gray)	SC	1.8	0.48	9	5	0.57, 0.18, 0.21	0.01, 0.01, <0.01	23087
CA, 1987 (California Sweet)	WP	1.8	0.55	8	5	<0.03 (4)	<0.01 (4)	23487
CA, 1989 (California Sweet)	SC	1.8	2.2	a 8	5	<0.03 (4)	<0.01 (4)	50B-89
FL, 1989 (Jubilant)	SC	1.8	3.3	a 8	5	0.19	0.02	50C-89
CA, 1990 (Peacock)	SC	1.8	2.1	a 8	5	0.048, 0.041, <0.03 (2) qm <0.03 (2) p 0.046, 0.042 pu <0.03 (2)	<0.01, 0.01, <0.01 (2)	50A-89

[1] a: aerial application.
[2] qm: quartered melon. p: peel. pu: pulp.

Table 8. Maneb residues (as CS_2) in leafy vegetables from supervised trials in Canada and USA. Underlined residues are from treatments according to GAP.

CROP Country, year (Variety)	Application [1]	Day	Residues, mg/kg [2]	Ref.

	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
ENDIVE								
Canada, 1989 (Green Curled)	WP	1.8	0.32	3	0 1 3 7 10 14	9.0 16 20 1.1 0.71 0.39		84100761
KALE								
USA (CA), 1987 (Siberian)	WP	1.3	0.69	4	7 10	6.0, 11, 8.1, 4.9 6.6, 4.9, 6.6, 4.0	0.13, 0.18, 0.18, 0.080 0.25, 0.17, 0.10, 0.16	26687
USA (CA), 1987 (Siberian)	WP	1.8	0.91	4	7 10	4.9, 13, 3.9, 5.8 4.6, 6.2, 5.8 c 0.14, 0.46, 0.13, 0.14	0.070, 0.11, 0.10 (2) 0.10, 0.070, 0.080, 0.060 c <0.01 (4)	26687
USA (NJ), 1987 (Vates)	SC	1.3	0.33	4	7 10	5.0, 8.1, 8.5 7.2, 7.5, 6.6	0.14, 0.080, 0.060 0.14, 0.13, 0.14	26987
USA (NJ), 1987 (Vates)	SC	1.8	0.45	4	7 10	14, 12, 13 5.3, 14, 28 c 0.34, 0.57, 0.23	0.23, 0.080, 0.13 0.090, 0.10, 0.12 c <0.01 (3)	26987
USA (TX), 1987 (Vates Blue Culled Scotch)	SC	1.8	0.96	4	7	3.0, 2.4, 2.7 c 0.23	0.030, 0.02, 0.02	27187
USA (FL), 1988 (Curley Blue Dwarf)	WP	1.3	0.72	4	7 10	1.1, 1.3, 0.92 0.20, 0.41, 0.30	0.04 (3) <0.01, 0.01 (2)	26787
USA (FL), 1988 Curley Blue Dwarf)	WP	1.8	0.96	4	7 10	0.92, 0.86, 0.75 0.25, 0.35, 0.31 c 0.080	0.04, 0.02, 0.03 <0.01, 0.01 (2) c <0.01	26787
LETTUCE								
Canada, 1989 (Ithaca)	WP	1.8		1	0 1 3 7 14 21	6.6 6.9 0.55 0.39 0.05 <0.05		84100761
Canada, 1989 (Ithaca)	WP	1.8		1	1 14 21 28 35	39 0.24 <0.05 <0.05 <0.05		84100761
Canada, 1989 (leaf lettuce, Grand Rapids)	WP	1.8	0.32	3	0 1 3 7 10 14	24 12 12 0.97 0.75 0.16		84100761
Canada, 1989 (cos lettuce, Parris Island Cos)	WP	1.8	0.32	3	0 1 3 7 10 14	8.3 4.8 5.5 1.1 0.48 0.32		84100761
Canada, 1989 (cos lettuce, Parris Island)	WP	1.8	0.45	2	0 1 2 7 9 14 21	8.2 7.2 6.6 2.7 1.6 0.88 0.32		84100761
Canada, 1989 (cos lettuce, Parris Island)	WP	1.8	0.45	3	0 1 2 7 9 14 21	4.6 5.6 3.2 1.2 0.50 0.61 0.94		84100761
Canada, 1989 (cos lettuce, Parris Island)	WP	1.8	0.45	5	0 1 2 7 9 14 21	13 13 5.1 3.9 2.5 1.9 1.9		84100761
Canada, 1989 (cos lettuce, Parris Island)	WP	1.8	0.45	5	0 1 3 7 9 14 21	19 28 11 8.8 6.1 6.0 3.7		84100761
Canada, 1989 (cos lettuce, Parris Island)	WP	0.9	0.23	5	0 1 3 7 9	4.4 4.7 2.4 1.6 1.4		84100761

CROP Country, year (Variety)	Application [1]				Day	Residues, mg/kg [2]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
					14 21	1.4 0.94		
Canada, 1989 (cos lettuce, Parris Island)	WP	3.6	0.90	5	0 1 3 7 9 14 21	26 15 8.0 6.9 4.2 5.2 2.6		84100761
USA (CA), 1987 (Salinas S-1)	WP	1.8	0.48	6	10	<0.03 (4)	<0.01 (4)	26587
USA (CA), 1987 (Van Sel)	WP	1.8	0.48	6	10	<0.03 (3), 0.07	<0.01 (4)	23187
USA (CA), 1989 (Marque)	SC	1.8	0.96	a 6	10	wl 0.089, 0.21 wo <0.03 (4)	wl <0.01 (2) wo <0.01 (4)	46J-89
USA (AZ), 1990 (Salinas MI)	SC	1.8	1.9	a 6	10	wl 2.2, 0.12, 2.2, 0.70 wo 0.094, 0.070, 0.28, 0.13	wl 0.02, <0.01, 0.02, <0.01 wo <0.01 (4)	46B-89
USA (CA), 1990 (Empire)	SC	1.5-1.9	3.7	a 6	10	wl 6.9, 5.8, 5.5, 6.1 wo 0.28, 0.42, 0.74, 0.33	wl 0.055, 0.097, 0.082, 0.063 wo <0.01 (4)	46L-89
SPINACH								
USA (NJ), 1987 (Sevin R)	SC	1.8	0.45	4	10	7.2, 6.1, 5.8 w 6.6, 3.9, 5.6	0.039, 0.02, 0.061 w 0.026, 0.026, 0.032	25987
USA (TN), 1987	SC	1.8	0.96	5	14	9.4, 6.5, 6.1 c 0.40 w 5.4, 5.3, 4.9 cw 0.20	0.054, 0.057, 0.051 c <0.01 w 0.046, 0.051, 0.052 cw <0.01	26187
USA (TX), 1987 (Dixie Market)	SC	1.8	0.96	4	10	2.2, 1.4, 5.3 w 2.1, 1.8, 0.99	0.026, 0.02, 0.028 w 0.031, 0.025, 0.02	26287
USA (CA), 1988 (Viro Flay)	WP	1.8	1.0	4	10	17, 14, 14, 15 c 0.03 w 10, 8.7, 11, 14 cw <0.03	0.16, 0.086, 0.15, 0.21 c <0.01 w 0.056, 0.094, 0.061, 0.052 c <0.01	25587
USA (AZ), 1988 (Polka)	SC	1.3	0.52	4	7 10	8.0, 8.6, 8.0 6.9, 5.7, 6.9	0.57, 0.78, 0.98 0.34, 0.21, 0.46	88136
USA (AZ), 1988 (Polka)	SC	1.8	0.70	4	7 10	6.9, 16, 13 9.2, 13, 8.6	0.22, 1.4, 0.99 0.71, 1.1, 0.68	88136

[1] a: aerial application.
[2] c: control sample; wl: includes wrapper leaves; wo: without wrapper leaves; w: washed commodity; cw: control sample of washed commodity.

Table 9. Maneb residues (as CS_2) in beans from supervised trials in the USA.

CROP State, year (Variety)	Application [1]				Day	Residues, mg/kg [2]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
DRY BEANS, SNAP BEANS								
CA, 1987 (Snap dry bean)	WP WP	1.8 1.8	0.96 0.38	6 6	4 4	2.0, 2.0 1.9, 1.9	0.02, 0.02 0.02, 0.01	25387
CO, 1987	WP WP	1.8 1.8	0.96 0.38	6 6	4 4	0.04 0.04	0.02 0.02	20787
MI, 1987	WP WP	1.8 1.8	0.96 0.38	6 6	4 4	0.75 0.66	0.049 0.036	20887
ND, 1987 (Agate Pinto)	WP WP	1.8 1.8	0.96 0.38	6 6	4 4	0.080 0.052	<0.01 <0.01	21087
NE, 1987 (Great Northern)	WP WP	1.8 1.8	0.96 0.38	6 6	4 4	0.12 0.22	0.01 0.01	20987
CA, 1989 (Pinto)	SC	1.8	1.9	a 6	4	0.080, 0.086, 0.11, 0.063	<0.01, 0.01, <0.01 (2)	54A-89
CA, 1989 (Green Crop)	SC	2.7	1.4	a 6	4	0.10, 0.30, 0.086, 0.26	0.02 (4)	54B-89
FL, 1989 (Fordhook)	SC	1.8	3.3	a 6	4	0.37	0.03	54C-89
CA, 1989 (Blue Lake)	SC	1.8	1.9	a 7	4	0.040, 0.092, 0.063, 0.046	0.02, 0.057, 0.042, 0.026	52A-89
CA, 1989 (Green Crop)	SC	1.8	2.3	a 7	4	3.9, 4.4, 3.3, 3.4	0.38, 0.78, 0.32, 0.29	52B-89
FL,1989 (Triumph)	SC	1.8	3.2	a 7	4	0.017	0.01	52C-89
SUCCULENT BEANS								

CROP State, year (Variety)	Application [1]				Day	Residues, mg/kg [2]		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
NY, 1987 (Improved Tendergreen)	WP	1.8		7	4	0.092, 0.069, 0.080 w 0.35, 0.25, 0.03	0.028, 0.031, 0.027 w 0.032, 0.026, 0.026	21287
WI, 1987 (Amity)	WP	1.8		6	4	0.45, 0.46, 0.28 w 0.17, 0.54, 0.46	<0.01, 0.02, <0.01 w <0.01, 0.02, 0.01	21487
MI, 1987 (Tendercrop)	WP	1.8		7	4	0.63, 0.52, 0.75 w 0.57, 0.52, 0.35	0.036, 0.046, 0.033 w 0.037, 0.030, 0.034	21187
OR, 1987 (OSU9/S)	WP	1.8		7	4	0.05, 0.03 (2) w <0.03 (3)	0.02 (2), 0.01 w <0.01 (3)	21387
CA, 1987 (Throughgrain)	WP	1.8		6	4	3.5, 2.8, 3.6, 3.0	0.13, 0.11, 0.15, 0.11	25087
DE, 1987 (8-78)	WP	1.8		6	4	<0.03	<0.01	21687

[1] a: aerial application.
[2] w: washed.

Table 10. Maneb residues (as CS_2) in fruiting vegetables from supervised trials in Canada, The Netherlands and the USA. Underlined residues are from treatments according to GAP.

CROP Country, year (Variety)	Application [1]				Day	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
PEPPERS								
USA (CA), 1987 (Bell)	SC	1.8		6	7	0.69, 0.75, 1.0, 0.86	0.01, <0.01 (3)	25687
USA (FL), 1987 (California Wonder)	SC	1.8		6	7	0.41, 0.28, 0.14	0.05, 0.04, 0.019	25887
USA (TX), 1987 (Grande R066)	SC	1.8		6	7	<0.03, 0.03 (2)	<0.01 (3)	26387
USA (NC), 1988 (California Wonder)	WP	1.8		6	7	0.091, 0.058, 0.22	<0.01 (2), 0.02	10688
USA (CA), 1989 (Green Bell Pepper)	SC	1.8	1.9	a 6	7	0.050, 0.040, 0.056, 0.066	<0.01 (4)	47A-89
USA (CA), 1989 (Emperial Giant)	SC	1.8	0.96	a 8	7	0.39, 0.47, 0.57, 0.32	<0.01 (2), 0.01, 0.02	47B-89
USA (FL), 1989 (Early Cal Wonder)	SC	1.8	3.2	a 6	7	0.15	0.01	47C-89
SWEET CORN (cob + kernel)								
USA (OR), 1987 (Jubilee)	WP	1.8		5	4	<0.03 (3)	<0.01 (3)	22087
USA (IL), 1987 (Illini Super Sweet)	SC	1.8		5	4	0.13, 0.052, 0.052	<0.01 (3)	22187
USA (MN), 1987 (Golden Beauty)	SC	1.8		5	4	0.03, <0.03, 0.05	<0.01 (3)	22287
USA (NY), 1987 (Early Sunray)	SC	1.8		5	4	<0.03 (3)	<0.01 (3)	22387
USA (WI), 1987	WP	1.8		5	4	<0.03 (3)	<0.01 (3)	22487
USA (GA), 1987 (Merit)	SC	1.8		5	4	0.03, <0.03, 0.03	<0.01 (3)	24887
USA (CA), 1989 (Yellow Sweet Corn)	SC	1.8	1.9	a 5	5	0.070, 0.036, 0.066, 0.056	<0.01 (4)	49A-89
USA (CA), 1989 (Hybrid Jubilee)	SC	1.8	1.8-3.9	a 5	5	0.13, 0.26, 0.11, 0.094	<0.01 (4)	49B-89
USA (FL), 1989 (7210)	SC	1.8	3.2	a 5	5	<0.03	<0.01	49C-89
TOMATO								
Canada, 1989 (Heinz 318)	WP	2.6	0.33	1	1 3 7 14 21 28	0.065 0.05 <0.05 0.05 0.11 <0.05		84100761
Netherlands, 1984	WP SC	1.6-2.4 1.6-2.4		g 6	15	<0.01, 0.13 <0.01 (2)	<0.002 (2) <0.002, 0.002	PH8405

CROP Country, year (Variety)	Application [1]				Day	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
(Abunda)	SC	1.6-2.4				<0.01 (2)	<0.002 (2)	
Netherlands, 1984 (Abunda)	WP WP SC SC SC SC	1.6 1.6 1.6 1.6 1.6 1.6		g 3 g 8 g 3 g 8 g 3 g 8	15 4 15 4 15 4	<0.01, 0.05 0.02, 0.15 <0.01, 0.05 0.07, <0.01 <0.01 (2) <0.01, 0.14	0.0032, <0.002 0.003, 0.059 0.0048, <0.002 <0.002, 0.020 0.0052, 0.002 <0.002, 0.025	PH8406
USA (CA), 1987 (P-19)	SC	2.7	0.72	7	3	<0.03, 0.17, 0.04, <0.03	<0.01 (4)	24487
USA (FL), 1987 (Sunny)	SC	2.7		7	3	1.6, 1.9, 1.4	<0.01 (3)	24587
USA (MI), 1987 (Pik Red)	WP	2.7	0.58	7	3	0.13, 0.21, 0.21	<0.01 (3)	24687
USA (TX), 1987	SC	2.7	0.72	7	3	0.069, 0.057, 0.052	<0.01 (3)	24787
USA (CA), 1989 (UC 82)	SC	2.7	2.9	a 7	3	0.51, 0.58, 0.24, 0.35	0.02, 0.01, <0.01 (2)	48A-89
USA (CA), 1989 (Ace 55)	SC	2.7	1.4	a 7	3	0.13, 0.097, 0.053, 0.072	<0.01 (4)	48B-89
USA (FL), 1989 (Sunny)	SC	2.7	4.9	a 7	3	0.096	<0.01	48C-89
USA (CA), 1989 (Ace)	WP	2.2	0.33	1	7	0.072, 0.089	<0.01 (2)	61A-89
USA (CA), 1989 (Ace)	WP	2.2	0.33	4	5	0.39, 0.24	<0.01 (2)	61A-89
USA (CA), 1989 (Ace)	WP	2.2	0.33	1	7	0.050, 0.029	<0.01 (2)	61B-89
USA (CA), 1989 (Ace)	WP	2.2	0.33	4	5	0.093, 0.20	<0.01 (2)	61B-89
USA (CA), 1989 (Royal Flush)	WP	2.2	0.35	1	7	0.28, 0.16	<0.01 (2)	61D-89
USA (CA), 1989 (Royal Flush)	WP	2.2	0.35	4	5	0.50, 0.63	<0.01 (2)	61D-89
USA (CA), 1989 (Blaze)	WP	2.2	0.48	1	7	0.25, 0.18	<0.01 (2)	61E-89
USA (CA), 1989 (Blaze)	WP	2.2	0.48	4	5	0.12, 0.22	<0.01 (2)	61E-89
USA (FL), 1990 (Sunny)	SC	2.7	4.7	a 8	3	0.12	<0.01	48D-89
USA (CA), 1990 (Cal-Ace 55VF)	WP	2.2	0.32	1	7	0.24, 0.30	<0.01 (2)	61C-89
USA (CA), 1990 (Cal-Ace 55VF)	WP	2.2	0.32	4	5	0.32, 0.16	<0.01 (2)	61C-89
USA (CA), 1990 (Roma)	WP	2.2	0.51	1	7	0.21, 0.15	<0.01 (2)	61F-89
USA (CA), 1990 (Roma)	WP	2.2	0.51	4	5	1.0, 1.1	0.022, 0.023	61F-89
USA (CA), 1990 (Sunny)	WP	2.2	0.24	1	7	0.23	<0.01	61G-89
USA (CA), 1990 (Sunny)	WP	2.2	0.24	4	5	2.0	0.039	61G-89
USA (CA), 1990 (Sunny)	WP	2.2	3.9	a 1	7	<0.03	<0.01	61H-89
USA (CA), 1990 (Sunny)	WP	2.2	3.9	a 4	5	0.037	<0.01	61H-89

[1] a: aerial application; g: glasshouse trial.

Table 11. Maneb residues (as CS_2) in root and tuber vegetables from supervised trials in The Netherlands, the UK and the USA. Underlined residues are from treatments according to GAP.

CROP Country, year (Variety)	Application [1]	Day	Residues, mg/kg	Ref.

	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
POTATO								
Netherlands, 1984 (Bintje)	WP SC SC	1.6-3.2 1.6-3.2 1.6-3.2		5 5 5	14 14 14		0.002, 0.0045 0.004, 0.002 0.0054, 0.004	PH8419
Netherlands, 1984 (Bintje)	WP SC SC	1.6-3.2 1.6-3.2 1.6-3.2		5 5 5	11 11 11		0.003 (2) 0.0077, 0.0054 0.013, 0.0077	PH8420
Netherlands, 1984 (Bintje)	WP SC SC	1.6-3.2 1.6-3.2 1.6-3.2		10 10 10	7 7 7		<0.002 (2) 0.004 (2) 0.0066, 0.0083	PH8421
Netherlands, 1985 (Bintje)	WP SC SC	1.6-3.2 1.6-3.2 1.6-3.2		9 9 9	9 9 9	<0.01 (2) <0.01 (2) <0.01 (2)	0.017, 0.0081 0.0085, 0.0043 0.0065, 0.002	PH8518
Netherlands, 1985 (Bintje)	WP SC SC	1.6-3.2 1.6-3.2 1.6-3.2		8 8 8	17 17 17	<0.01 (2) <0.01 (2) <0.01 (2)	0.0090, 0.0065 0.0075, 0.011 0.004, 0.0099	PH8520
Netherlands, 1986 (Bintje)	WP SC SC SC	1.6-3.2 1.6-3.2 1.6-3.2 1.6-3.2		9 9 9 9	20 20 20 20	0.15, <0.01 <0.01 (2) <0.01, 0.04 <0.01, 0.06	0.0058, 0.0067 <0.002, 0.0045 <0.002, 0.0046 <0.002, 0.0086	PH8620
Netherlands, 1987 (Bintje)	SC SC	1.6-3.2 1.6-3.2		8 8	12 12	<0.02 (2) <0.02 (2)	0.0088, 0.0046 0.003, 0.0067	PH8719
Netherlands, 1988 (Bintje)	WP SC WG	1.6-3.2 1.6-3.2 1.6-3.2		7 7 7	22 22 22	0.06, <0.05 0.08, <0.05 <0.05 (2)	0.004, 0.010 0.004, 0.013 0.010, 0.011	PH8827
Netherlands, 1988 (Bintje)	WP SC WG	1.6-3.2 1.6-3.2 1.6-3.2		10 10 10	18 18 18	0.10, <0.05 <0.05 (2) <0.05 (2)	0.004, 0.005 0.009, 0.006 0.005, 0.004	PH8829
UK, 1991 (Mario Piper)	WG WG	1.3 2.6	0.51 1.0	5 5	20 20	<0.01 <0.01	<0.01 <0.01	R52678/7 R52678/8
UK, 1991 (Mario Piper)	WG WG	1.3 2.6	0.51 1.0	5 5	26 26	<0.01 (3) <0.01 (3)	<0.01 (3) <0.01 (3)	R52628/2 R52628/3
UK, 1991 (King Edward)	WG WG	1.3 2.6	0.51 1.0	5 5	37 37	<0.01 <0.01	<0.01 <0.01	R52628/12 R52628/13
USA (CA), 1987 (White Rose)	WP	1.8	0.48	12	14	<0.03 (4)	<0.01 (4)	23587
USA (ID), 1987 (Russet Burbank)	SC	1.8	0.48	12	14	<0.03 (3)	<0.01 (3)	23687
USA (ME), 1987 (Katahdin)	SC	1.8	0.19	12	13	<0.03 (3)	<0.01 (3)	23787
USA (ND), 1987 (Norchip)	WP	1.8	0.48	12	14	<0.03 (3)	<0.01, 0.01 (2)	23887
USA (OR), 1987 (Russet)	WP	1.8	0.48	11	14	<0.03 (3)	0.01 (2), <0.01	23987
USA (CA), 1989 (White Rose)	SC	1.8	1.9	a 12	14	<0.03 (3)	<0.01 (3)	38A-89
USA (CA), 1989 (White Russet)	SC	1.8	1.9	a 12	14	0.037, 0.052, 0.097, 0.23	<0.01 (3)	38B-89
USA (FL), 1989 (Red Pontiac)	SC	1.8	3.2	a 12	14	<0.03	<0.01	38C-89
USA (FL), 1990 (Red Pontiac)	SC	1.8	3.1	a 12	14	<0.03	<0.01	38D-89

SUGAR BEET								
USA (CA), 1987	SC	1.8	0.96	7	14	0.37, 0.28, 0.51, 0.99	<0.01 (3)	24087
USA (ID), 1987 (Great Western R2)	SC	1.8	0.96	7	14	0.052, 0.03, 0.03	<0.01 (3)	24187
USA (MN), 1987 (Ultramono)	SC	1.8	0.96	7	14	0.040, 0.069, 0.057	<0.01 (3)	24287
USA (ND), 1987 (Monorica)	WP	1.8	0.96	7	14	0.10, <0.03 (2)	<0.01 (3)	24387

[1] a: aerial application.

Table 12. Maneb residues (as CS_2) in celery from supervised trials in Canada and the USA. All WP.

Country, year (Variety)	Application			Day	Residues, mg/kg		Ref.
	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
Canada, 1989 (Florida 683)	1.8		1	0 1 7 14 21	0.52 0.22 0.22 0.22 0.19		84100761
Canada, 1989 (Florida 683)	1.8		1	0 21 28 35 42	1.4 0.17 0.069 0.055 0.055		84100761
USA (CA), 1989 (Bud of California)	1.8	0.81	1 4	7 14	1.5, 3.7 1.4, 2.0	<0.01, 0.01 0.01, <0.01	62A-89
USA (CA), 1989 (Bud of California)	1.8	0.81	1 4	7 14	0.31, 0.69 1.5, 1.4	<0.01 (2) 0.01, 0.03	62B-89
USA (CA), 1989 (Bud of California)	1.8	0.38	1 4	7 14	1.4, 1.1 1.0, 0.55	<0.01 (2) 0.01, <0.01	62C-89
USA (CA), 1989 (Tall Utah)	1.8	0.38	1 4	7 14	0.38, 0.77 1.5, 2.1	<0.01 (2) <0.01 (2)	62D-89
USA (CA), 1989 (Tall Utah)	1.8	0.38	1 4	7 14	0.60, 0.70 1.8, 1.4	0.01, <0.01 0.01, 0.01	62E-89
USA (CA), 1989 (Tall Utah)	1.8	0.96	1 4	7 14	0.20, 0.25 1.5, 1.9	<0.01 (2) 0.01, <0.01	62F-89

Table 13. Maneb residues (as CS_2) in cereal grains from supervised trials in The Netherlands, the UK and the USA. Underlined residues are from treatments according to GAP.

CROP Country, year (Variety)	Application				Day	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
BARLEY								
Netherlands, 1986 (Hasso)	WP	1.6		2	58	<0.01 (2)	<0.002 (2)	PH8616
Netherlands, 1987 (Hasso)	WP SC	1.6 1.6		2 2	67 67	0.06, <0.03 <0.03, 0.04	0.016, 0.054 0.056, 0.028	PH8717/2
Netherlands, 1988 (Prisma)	WP	1.6		2	57	0.09, 0.13	<0.002 (2)	PH88-35
Netherlands, 1988 (Trumpf)	WP	1.6		2	60	0.60, 0.20	<0.002 (2)	PH88-38
WINTER WHEAT								
Netherlands, 1984 (Okapi)	WP SC SC	1.6 1.6 1.6	0.49 0.49 0.49	2 2 2	61 61 61	<0.02 (2) <0.02 (2) 0.02, <0.02	<0.02 (2) 0.02, <0.02 <0.02 (2)	PH8431
Netherlands, 1985 (Saiga)	WP SC SC	1.6 1.6 1.6	0.40 0.40 0.40	2 2 2	68 68 68	<0.01, 0.65 <0.01 (2) <0.01 (2)	<0.002 (2) <0.002 (2) <0.002 (2)	PH8526
Netherlands, 1985 (Marksman)	WP SC	1.6 1.6	0.32 0.32	2 2	57 57	<0.01 (2) <0.01 (2)	<0.002 (2) <0.002 (2)	PH8527
Netherlands, 1986 (Okapi)	WP SC	1.6 1.6	0.40 0.40	2 2	66 66	<0.01, 0.07 <0.01, 0.03	<0.002 (2) <0.002 (2)	PH8626
Netherlands, 1987	SC	1.6	0.27	2	64	<0.02 (2)	<0.002 (2)	PH8727

CROP Country, year (Variety)	Application				Day	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
(Arminda)								
Netherlands, 1988 (Obelisk)	WP SC WG	1.6 1.6 1.6	0.27 0.27 0.27	2 2 2	68 68 68	<0.05, 0.07 <0.05 (2) 0.12, 0.05	0.002 (2) 0.004, <0.002 <0.002, 0.003	PH8839
UK, 1991 (Riband) (Riband)	WG WG WG	1.5 3.0 1.5	0.63 1.3 0.63	1 1 1	24 24 51	0.10, 0.10, 0.11 0.23, 0.15, 1.0 0.03	<0.01 (3) <0.01 (3) <0.01	R52628/27 R52628/28 R52628/42
(Riband)	WG	3.0	1.3	1	51	0.10	<0.01	R52628/43
USA, 1991 (Haven)	WG WG	1.5 3.0	0.63 1.3	1 1	46 46	<0.01 0.10	<0.01 <0.01	R52628/57 R52628/58

Table 14. Maneb residues (as CS_2) in almonds from supervised trials in the USA. Underlined residues are from treatments according to GAP. All SC.

State, year (Variety)	Application[1]			Day	Residues, mg/kg		Ref.
	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
CA, 1988 (Non Pariel)	7.2	0.74	4	138	<0.03 (4)	<0.01 (4)	36A-88
CA, 1988 (Non Pariel)	7.2	7.7	a 4	142	<0.03 (4)	<0.01 (4)	36B-88
CA, 1988 (Non Pariel)	7.2	0.74	4	135	<0.03 (4)	<0.01 (4)	36C-88
CA, 1988 (Non Pariel)	7.2	3.8	a 4	129	0.03, <0.03 (3)	<0.01 (4)	36D-88

[1] a: aerial application.

Table 15. Maneb residues (as CS_2) in cereal straw and forage from supervised trials in The Netherlands, the UK and the USA. Underlined residues are from treatments according to GAP.

CROP Country, year (Variety)	Application				Day	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
BARLEY STRAW								
Netherlands, 1986 (Hasso)	WP	1.6		2	58	2.6	0.11	PH8616
Netherlands, 1987 (Hasso)	WP SC	1.6 1.6		2 2	67 67	<0.05 <0.05	1.4 2.2	PH8717/2
Netherlands, 1988 (Trumpf)	WP	1.6		2	60	2.2	0.089	PH88-38
MAIZE FORAGE								
USA (OR), 1987 (Jubilee)	WP	1.8		5	4	4.0, 3.1, 4.1	0.02, 0.01, 0.02	22087
USA (IL), 1987 (Illini Super Sweet)	SC	1.8		5	4	32, 32, 23	0.063, 0.060, 0.062	22187
USA (MN), 1987 (Golden Beauty)	SC	1.8		5	4	5.6, 6.2, 3.9	<0.01 (2), 0.01	22287
USA (NY), 1987 (Early Sunray)	SC	1.8		5	4	2.3, 3.2, 3.6	0.021, 0.029, 0.027	22387
USA (WI), 1987	WP	1.8		5	4	6.2, 3.4, 4.8	0.026, 0.015, 0.021	22487
USA (GA), 1987 (Merit)	SC	1.8		5	4	20, 28, 28	0.098, 0.10, 0.10	24887
WHEAT STRAW								
Netherlands, 1987 (Arminda)	SC	1.6	0.27	2	64	<0.01	<0.002	PH8727
Netherlands, 1988 (Obelisk)	WP SC WG	1.6 1.6 1.6	0.27 0.27 0.27	2 2 2	68 68 68	2.1 1.8 1.8	0.065 0.071 <0.01	PH8839
UK, 1991 (Riband)	WG WG	1.5 3.0	0.63 1.3	1 1	24 24	0.29, 0.30, 0.35 2.6, 2.4, 2.9	<0.01 (2), 0.01 <0.01, 0.03, <0.01	R52628/27 R52628/28

CROP Country, year (Variety)	Application				Day	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
UK, 1991	WG	1.5	0.63	1	51	<0.01	<0.01	R52628/42
(Riband)	WG	3.0	1.3	1	51	0.51	<0.01	R52628/43
UK, 1991	WG	1.5	0.63	1	46	<0.01	<0.01	R52628/57
(Haven)	WG	3.0	1.3	1	46	0.55	0.01	R52628/58

Table 16. Maneb residues (as CS_2) in bean vines from supervised trials in the USA. Residue data are expressed on a dry weight basis. All at WP at 1.8 kg ai/ha.

CROP Country, year (Variety)	Application		Day	Residues, mg/kg [1]		Ref.
	kg ai/hl	No.		EBDC as CS_2	ETU	
DRY BEAN VINES						
CA, 1987	0.96	6	4	145	4.9	25387
(Snap bean)	0.38	6	4	165	6.9	
CO, 1987	0.96	6	4	28	0.15	20787
	0.96	6	7	28	0.60	
	0.38	6	4	16	0.59	
	0.38	6	7	17	0.27	
MI, 1987	0.96	6	4	17	0.83	20887
	0.96	6	7	14	0.16	
	0.38	6	4	11	0.69	
	0.38	6	7	11	0.063	
ND, 1987	0.96	6	4	28	3.6	21087
(Agate Pinto)	0.96	6	7	18	1.1	
	0.38	6	4	42	6.1	
	0.38	6	7	19	0.77	
NE, 1987	0.96	6	4	15	1.0	20987
(Great Northern)	0.96	6	7	12	1.2	
	0.38	6	4	8.9	0.89	
	0.38	6	7	8.9	0.90	
SUCCULENT BEAN VINES						
NY, 1987		7	4	25, 18, 19	0.74, 1.1, 0.90	21287
(Improved Tendergreen)			7	9.7, 9.7, 11	0.48, 0.66, 0.79	
WI, 1987		6	4	48, 115, 83	1.5, 4.3, 0.60	21487
(Amity)			7	14, 17, 15	1.0, 0.98, 0.73	
MI, 1987		7	4	37, 49, 52	0.91, 1.4, 0.68	21187
(Tendercrop)			7	53, 32, 34	0.30, 0.48, 0.46	
OR, 1987		7	4	1.3, 0.80, 3.6	0.027, 0.02 (2)	21387
(OSU9/S)						
CA, 1987		6	4	263, 201, 270, 209	0.02, 1.1, 0.65, 0.80	25087
(Throughgrain)				c 0.51, 0.27	c <0.01 (2)	
			7	482, 402, 464, 402	0.79, 0.53, 0.55, 0.83	
				c 1.3, 1.8	c 0.71, <0.01	
DE, 1987		6	4	0.80	0.047	21687
(8-78)						

[1] c: control sample.

Table 17. Maneb residues (as CS_2) in miscellaneous fodder commodities from supervised trials in the USA. Underlined residues are from treatments according to GAP.

CROP State, year (Variety)	Application [1]	Day	Residues, mg/kg	Ref.

	Form	kg ai/ha	kg ai/hl	No.		EBDC as CS_2	ETU	
ALMOND HULLS								
CA, 1988 (Non Pariel)	SC	7.2	0.74	4	138	9.3, 6.7, 7.3, 6.5		36A-88
CA, 1988 (Non Pariel)	SC	7.2	7.7	a 4	142	4.8, 3.9, 4.1, 2.7		36B-88
CA, 1988 (Non Pariel)	SC	7.2	0.74	4	135	0.034, 0.035, 0.053, 0.066		36C-88
CA, 1988 (Non Pariel)	SC	7.2	3.8	a 4	129	6.0, 7.9, 7.3, 9.7		36D-88
SUGAR BEET TOPS								
CA, 1987	SC	1.8	0.96	7	14	88, 76, 34, 29	0.37, 0.13, 0.040, 0.061	24087
ID, 1987 (Great Western R2)	SC	1.8	0.96	7	14	1.1, 0.85, 1.4	<0.01 (3)	24187
MN, 1987 (Ultramono)	SC	1.8	0.96	7	14	8.9, 8.4, 13	0.01 (2), 0.048	24287
ND, 1987 (Monorica)	WP	1.8	0.96	7	14	2.6, 2.3, 3.0	<0.01 (2), 0.01	24387

[1] a: aerial application.

FATE OF RESIDUES

In animals

Metabolism studies on lactating goats and laying hens were made available to the Meeting.

Tissue and milk residues were measured in 2 lactating goats (body weights 49 and 52 kg) dosed for 5 days by capsule with radiolabelled maneb ([^{14}C]ethylenediamine) at 75 mg/day equivalent to 50 ppm maneb in the feed (Wu, 1990a). Feed consumption was 1.5 kg/day. Milk was collected twice daily; animals were slaughtered for tissue collection 8 hours after the final dose.

Total ^{14}C residues in milk were close to a steady state concentration (0.2-0.4 mg/kg as CS_2) by days 3 and 4. The levels in the morning milk samples, collected just prior to the daily dose, were always considerably lower than in the evening samples, which suggested that levels in milk would decrease rapidly when dosing ceased. Total ^{14}C residues were distributed among the tissues and milk, with the highest levels in the liver and kidney (Table 18).

Table 18. Total ^{14}C residues (expressed as mg CS_2/kg) in tissues and milk from lactating goats dosed for 5 days with [^{14}C]ethylenediamine-labelled maneb equivalent to 50 ppm in the feed (Wu, 1990a).

Substrate	Total ^{14}C, expressed as mg CS_2/kg
Liver	5.8
Kidney	3.1
Loin muscle	0.23
Leg muscle	0.47
Fat	0.10
Milk (day 4)	0.33

Metabolites were identified by two-dimensional TLC, radiochromatography and HPLC. The metabolite distribution in the milk and tissues is summarized in Table 19. Ethyleneurea, Jaffe's Base (1-(2-imidazolin-2-yl)-2-imidazolidinethione) and ethylenethiourea were identified in each tissue and milk. The main primary metabolite was Jaffe's Base. Much of the ^{14}C had been incorporated into natural products.

ETU levels in tissues and milk were determined by direct analysis and by ^{14}C measurement (Table 20). Levels were low in all the tissues and the milk.

Table 19. Metabolite distribution in milk and tissues of lactating goats dosed for 5 days with [^{14}C]ethylenediamine-labelled maneb equivalent to 50 ppm in the feed (Wu, 1990a).

Metabolite	Metabolite expressed as % of total ^{14}C in the tissue or milk				
	Liver	Kidney	Muscle	Fat	Milk
Ethylenethiourea (ETU)	0.68	2.6	1.8	1.5	1.8
Ethyleneurea (EU)	4.9	4.4	5.1	7.5	4.6
1-(2-imidazolin-2-yl)-2-imidazolidinethione (Jaffe's Base)	11	47	7.0	9.7	23
Ethylenebisisothiocyanate sulphide[1] (EBIS) + ethylenethiourea-*N*-thiocarboxamide (ETT)	1.2	2.6	2.5	0.49	-
1,2-ethylenediamine (EDA)	4.6	1.5	-	2.1	6.1
N-acetyl-1,2-ethylenediamine	-	0.72	1.2	-	1.5
Hydantoin	-	-	3.1	-	0.53
Allantoin	3.0	-	5.3	4.5	-
Glycine	13	0.74	-	4.1	8.1
Creatine	1.8	-	8.3	-	-
Creatinine	1.1	0.68	2.1	2.9	2.2
Lipids	2.5	3.5	3.8	37	6.1
Bound residues	7.0	4.6	7.1	7.0	0.70

[1] IUPAC name: 5,6-dihydro-3*H*-imidazo[2,1-*c*][1,2,4]dithiazole-3-thione (DIDT)

Table 20. ETU distribution in milk and tissues of lactating goats dosed for 5 days with [^{14}C]ethylenediamine-labelled maneb equivalent to 50 ppm in the feed (Wu, 1990a).

Tissue or milk	ETU by direct chemical analysis, mg/kg	ETU by ^{14}C measurement, mg/kg
Liver	0.075	0.068
Kidney	0.050	0.14
Loin muscle	-	0.008
Leg muscle	0.035	-
Fat	<0.01	0.003
Milk	0.037	0.011

^{14}C residues were measured in the tissues and eggs of 30 laying hens (weighing 1.5 kg each) dosed for 7 days by capsule with radiolabelled maneb ([^{14}C]ethylenediamine) at 6.1 mg/day, equivalent to 51 ppm maneb in the feed (Wu, 1990b). Feed consumption was 0.12 kg/day. Eggs were collected daily; birds were slaughtered for tissue collection 8 hours after the final dose.

Total ^{14}C residues in the egg whites had reached a plateau by days 5-6, while the total ^{14}C in the yolks was still increasing at the end of the study. Total ^{14}C residues were distributed among the tissues and eggs, with the highest levels in the liver and kidney (Table 21).

Table 21. Total ^{14}C residues (expressed as mg CS_2/kg) in tissues and eggs from laying hens dosed for 7 days with [^{14}C]ethylenediamine-labelled maneb equivalent to 51 ppm in the feed (Wu, 1990b).

Substrate	Total ^{14}C, expressed as mg CS_2/kg
Liver	1.5
Kidney	1.8
Breast muscle	0.24
Thigh muscle	0.23
Fat	0.072
Skin	0.52
Egg white, day 7	0.42
Egg yolk, day 7	0.61

Metabolites were identified by two-dimensional TLC, radiochromatography and HPLC. The metabolite distribution in the eggs and tissues is summarized in Table 22. Ethyleneurea, 1-(2-imidazolin-2-yl)-2-imidazolidinethione and ethylenediamine were identified in all of the tissues, egg white and egg yolk. Ethyleneurea was the main primary metabolite. Much of the ^{14}C had been incorporated into natural products.

ETU was identified in all of the tissues (except skin) and eggs. ETU levels in tissues and eggs were determined by direct analysis and by ^{14}C measurement (Table 23). Levels of ETU were low in all tissues, but the direct method of analysis gave higher results.

Table 22. Metabolite distribution in eggs and tissues of laying hens dosed for 7 days with [^{14}C]ethylenediamine-labelled maneb equivalent to 51 ppm in the feed (Wu, 1990b).

Metabolite	Metabolite expressed as % of total ^{14}C in the tissue or egg component.

	Liver	Kidney	Breast muscle	Fat	Egg white	Egg yolk
EBIS[1] + ethylenethiourea-*N*-thiocarboxamide (ETT)	1.1	0.51	1.5	3.4	5.5	2.2
1-(2-imidazolin-2-yl)-2-imidazolidinethione (Jaffe's Base)	3.6	3.9	4.2	4.5	1.6	26
Ethylenethiourea (ETU)	2.6	3.3	1.3	4.7	1.8	1.4
Ethyleneurea (EU)	14	11	36	13	59	16
1,2-ethylenediamine (EDA)	9.9	9.9	2.5	2.1	1.1	2.1
N-acetyl-1,2-ethylenediamine	0.98	0.80	-	-	0.95	1.5
Creatine	2.7	-	-	-	-	0.43
Creatinine	-	1.2	-	-	1.0	-
Hydantoin	-	-	17	-	-	-
Allantoin	-	3.5	-	-	-	-
Glycine	17	12	-	3.8	-	1.4
N-formylglycine	-	-	-	2.7	-	-
Lipids	2.5	0.79	0.81	51	-	9.5
Proteins	39	39	26	11	23	31
Bound residues	3.1	9.2	4.4	2.6	1.6	4.0

[1] See Table 19 for chemical name.

Table 23. ETU in eggs and tissues of laying hens dosed for 7 days with [^{14}C]ethylenediamine-labelled maneb equivalent to 51 ppm in the feed (Wu, 1990b).

Tissue or egg component	ETU by direct chemical analysis, mg/kg	ETU by ^{14}C measurement, mg/kg
Liver	0.14	0.070
Breast muscle	0.044	0.005
Egg white	0.098	0.013
Egg yolk	0.039	0.015
Fat	<0.01	0.006

In plants

Metabolism studies were made available to the Meeting for lettuce, potato and tomato.

Lettuce plants were treated with 4 foliar sprays (3.1, 3.1, 6.3, and 6.3 kg ai/ha) of maneb, ^{14}C-labelled in both ethylene carbons, at approximately 7-days intervals, and harvested 13 days after the final application for metabolite identification and analysis (Ballantyne, 1992).

The harvested lettuce were surface rinsed with an EDTA solution to identify the components of the dislodgeable residue. The rinse contained approximately 33% of the ^{14}C residues; the remainder was in the tissue. The distribution of metabolites and terminal ^{14}C residues in the lettuce and the rinsings is summarized in Table 24.

Surface residues included mainly maneb and the primary metabolites, EBIS[1], ethyleneurea and ETU.

Identified metabolites in the lettuce tissue included EBIS, ethyleneurea, ethylenethiourea, ethylenediamine and *N*-acetyl ethylenediamine. Amino acids and protein were found to contain ^{14}C, which demonstrated that metabolites had been incorporated into the natural carbon pool.

[1] See Table 19 for chemical name

Table 24. Distribution of ^{14}C among the metabolites of maneb in lettuce tissues and washings of lettuce treated with maneb, ^{14}C-labelled in both ethylene carbons, and harvested 13 days after the final application (Ballantyne, 1992).

Metabolite	Metabolite as % of surface residue	Metabolite as % of residue in rinsed lettuce	Metabolite as % of total residue
Maneb	13		4.2
Ethylenethiourea (ETU)	16	2.8	7.0
EBIS[1]	49	5.3	19
Ethyleneurea (EU)	4.7	18	14
1,2-ethylenediamine (EDA)		2.7	1.8
N-acetyl-1,2-ethylenediamine		0.10	0.05
Amino acids		38	26
Unknowns		22	15
Polar origin		17	12

In a 1989 US (WI) metabolism study, potato plants, cultivar Norland, were treated with 4 foliar sprays (3.5, 3.5, 6.9, and 6.9 kg ai/ha) of maneb, ^{14}C-labelled at the ethylene carbon, at approximately 3-week intervals, and harvested 17 days after the final application for metabolite identification and analysis (Wright and Malik, 1992).

Most of the ^{14}C residues were in the foliage, with less than 0.3% in the tubers. Total ^{14}C residue levels (expressed as maneb) in the foliage, tuber pulp and tuber peel were 330, 0.92 and 0.84 mg/kg respectively. The nature of the residues in the tubers was further investigated. The distribution of the ^{14}C among primary metabolites and natural products in potato tuber pulp and peel is shown in Table 25.

The primary metabolites constituted only a minor part of the ^{14}C residues in the tuber, less than 9%. ETU, identified only in the potato peel at 0.004 mg/kg (0.02 mg/kg by direct chemical analysis), was thought to be the result of surface contamination, rather than a product of metabolism.

The results may be interpreted in terms of a relatively rapid conversion of the primary metabolites to a common plant metabolite such as glycine, which provides the mechanism for the ^{14}C to be incorporated widely into natural products.

Table 25. Distribution of ^{14}C among the metabolites of maneb in potato tuber pulp and peel from potato plants subjected to foliar application of ^{14}C- labelled maneb, and harvested 17 days after the final application (Wright and Malik, 1992).

Metabolite	Metabolite expressed as % of total ^{14}C in pulp	Metabolite expressed as % of total ^{14}C in peel
Ethylenethiourea (ETU)	-	0.49
Ethyleneurea (EU)	1.3	0.69
Ethylenethiourea-*N*-thiocarboxamide (ETT)	6.2	3.9
1,2-ethylenediamine (EDA)	0.90	-
N-acetyl-1,2-ethylenediamine	-	2.8
Hydantoin	2.1	-
Creatinine	15	-
Creatine	10	11
Allantoin	19	20
Glycine	9.0	5.4
Nonpolar lipids	0.65	0.18
Polar lipids	2.0	-
Amino acids	3.9	17
Starch	20	12
Cellulose, lignin and hemicellulose	1.1	12

In a 1989 US (WI) metabolism study, tomato plants, cultivar Ace, were treated with 4 foliar sprays of maneb, ^{14}C-labelled at the ethylene carbon, at exaggerated rates of 5.5, 10, 22, and 22 kg ai/ha at approximately 2- to 6-week intervals, and harvested 5, 17 and 24 days after the final application for metabolite identification and analysis (Wright and Ussary, 1993).

Much of the ^{14}C residue (49-63%) was removed from harvested tomatoes when they were washed with a 1% EDTA solution to determine the nature and quantity of dislodgeable residues. Maneb and EBIS were the main dislodgeable residues; the major part of the ETU residue in the whole tomatoes was in the rinsings.

Tomatoes were separated into peel and pulp for metabolite analysis. The peel contained 23-26% of the whole tomato ^{14}C, while the pulp contained 11-27%. The 5-day PHI sample, which contained the highest percentage of the ^{14}C, was chosen for detailed analysis for metabolites (Table 26).

The processes in tomato metabolism are similar to those in the other crops studied. The ^{14}C enters the metabolic carbon pool probably via glycine, from which it is incorporated into natural products.

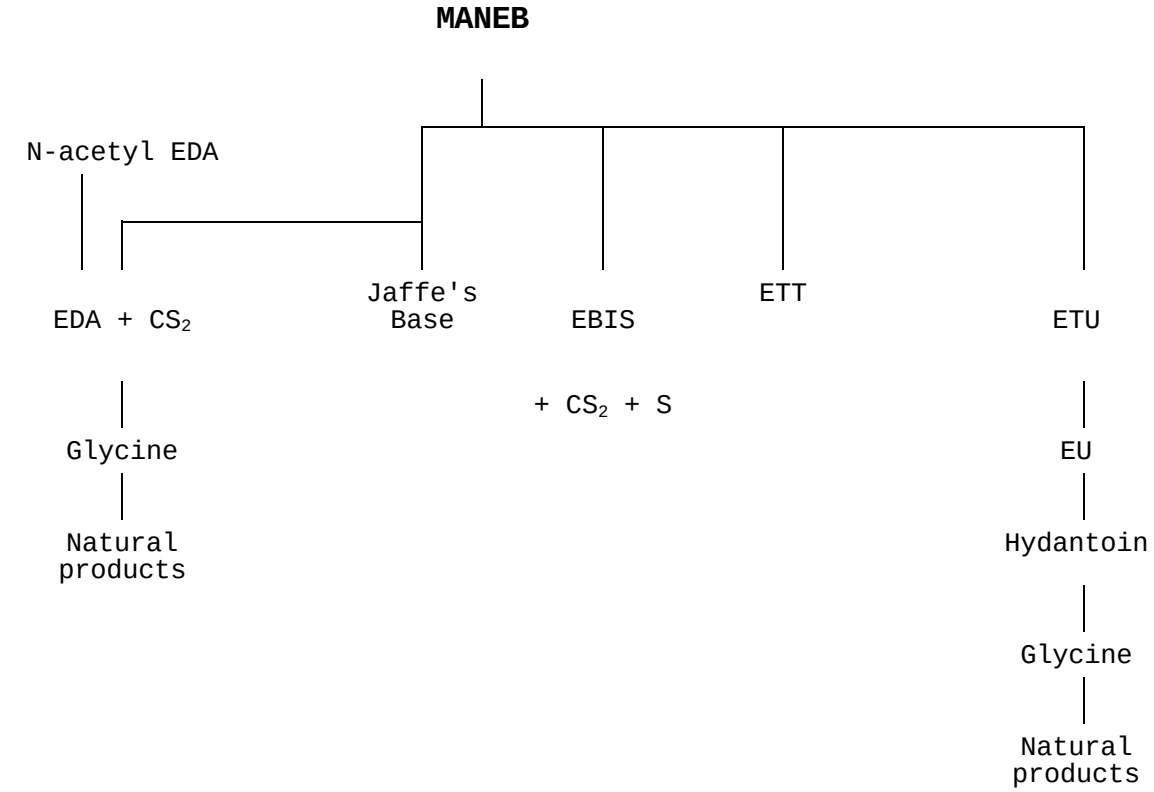
Table 26. Distribution of ^{14}C among the metabolites of maneb in tomato washings (1% EDTA), pulp and peel from tomatoes harvested 5 days after the final foliar application to a crop of ^{14}C-labelled maneb (Wright and Ussary, 1993).

Metabolite	Metabolite expressed as % of total ^{14}C in

	Rinsings	Peel	Pulp	Whole tomato
Maneb	28			14
Ethylenethiourea (ETU)	15	0.8	0.97	7.9
EBIS[2]	39	1.4	0.04	20
Ethyleneurea (EU)		6.0		1.4
N-acetyl-1,2-ethylenediamine		4.2	4.1	2.1
Allantoin		11	16	6.8
1,2-ethylenediamine (EDA)		11		2.7
Glucose			16	4.4
Amino acid			3.4	0.93
Unknowns, undefined	19	3.1	3.5	11
Polar material		4.8	27	8.6

Metabolic pathways of maneb are shown in Figure 1.

Figure 1. Metabolic pathways of maneb.



ETU: ethylenethiourea
EU: ethyleneurea
EBIS[1]: ethylenebisisothiocyanate sulphide
EDA: ethylenediamine
JB, Jaffe's Base: 1-(2-imidazolin-2-yl)-2-imidazolidinethione
ETT: ethylenethiourea-*N*-thiocarboxamide

[2]See Table 19 for IUPAC chemical name

In storage and processing

Processing studies were made available to the Meeting for apples, beans, grapes, sugar beet, sweet corn and tomatoes.

Pitt (1989b) studied the fate of maneb and ETU during the simulated commercial processing of apples (variety Monroe) subjected to 10 applications of maneb, each of 25.2 kg ai/ha (5 × commercial rate, 2.7 kg ai/hl spray concentration), in two trials in the USA (NY) in 1988. Apples were harvested 15 days after the final application.

Apples (75 kg) were ground in a hammer-mill, and the resultant wet mash was pressed to produce juice and wet pomace. Wet pomace was dried in a current of warm air (77-88°C) to yield a dry pomace with <10% moisture. The dry pomace was 20-25% by weight of the wet pomace. Although the process generally simulated commercial practice no washing was included; the intention was to represent a "worst case" situation. The resultant residues in apples and the processed fractions are shown in Table 27.

Dithiocarbamate residues accumulated in the pomace and were depleted in the juice, as would be expected from maneb's water solubility. ETU levels in the juice were lower than in the raw agricultural commodity.

Table 27. Residues of dithiocarbamates (as CS_2) and ETU in apples and processed fractions (Pitt 1989b). Apples were harvested for processing 15 days after the last of 10 applications of maneb, each of 25.2 kg ai/ha (5× commercial rate) in two trials in the USA (NY) in 1988.

Commodity	Trial 37A-88		Trial 37B-88	
	Dith residues, mg/kg as CS_2	ETU residues, mg/kg	Dith residues, mg/kg as CS_2	ETU residues, mg/kg
Apple	9.7	0.15	5.9	0.13
Wet pomace	10	0.46	17	0.70
Dry pomace	52	2.5	70	2.5
Fresh juice	2.0	0.018	2.8	0.037

In a US (NY) 1987 processing study maneb was applied on 6 occasions at 18 kg ai/ha (10 × maximum label rate) to beans which were harvested 4 days after the final treatment (Bookbinder, 1988e). Beans (34 kg) were processed into frozen and canned products according to a simulated commercial operation (Figure 2).

Residues of dithiocarbamates and ETU in the beans and processed commodities are summarized in Table 28. The report does not make it clear whether the sample of raw bean pods analysed was washed or unwashed.

Figure 2. Processing of beans field-sprayed with maneb (Bookbinder, 1988e).

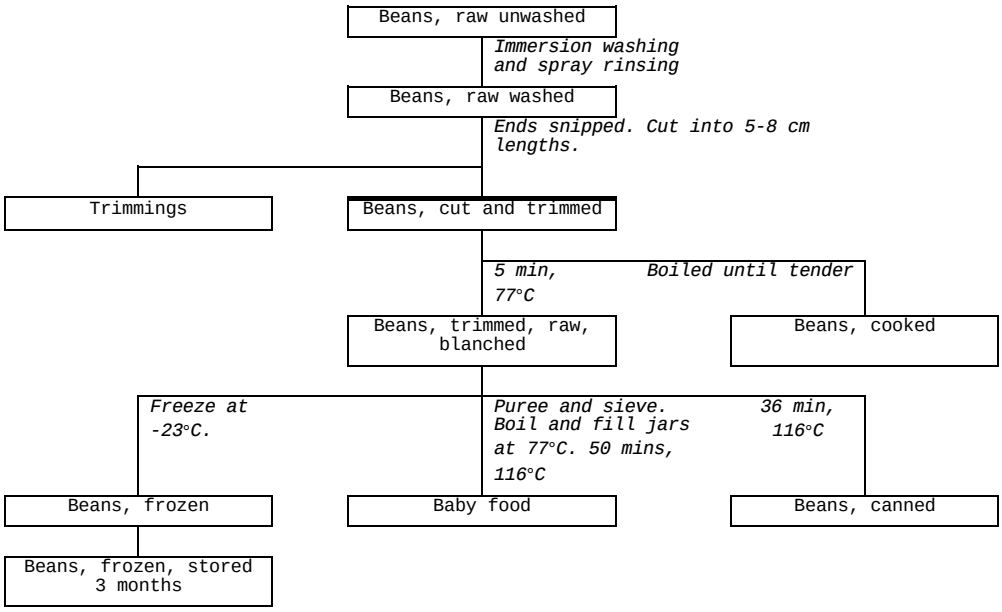


Table 28. Residues of dithiocarbamates (as CS_2) and ETU in snap beans foliar-sprayed with maneb and taken through the processing scheme in Figure 2 (Bookbinder, 1988e).

Commodity	Dithiocarbamate residues, mg/kg as CS_2	ETU residues, mg/kg
Raw bean pods	3.5	0.040
Beans, canned	0.03	0.49
Beans, frozen (not stored)	0.35	0.19
Baby food	<0.03	0.35
Trimmings (cannery waste)	3.6	0.12

In a US (CA) 1987 study maneb was applied once at 14 kg ai/ha (4 × maximum label rate) to grapes which were harvested 8 days after the final treatment (Bookbinder, 1988j). The label PHI is 66 days, but exaggerated conditions were used to produce high residues for the processing studies. Grapes (44 kg) were sent for processing into juice and pomace (Figure 3) and raisins. For the production of raisins, grapes were removed from the vine, but left firmly attached to the stem. Grapes were steam-heated for 30-60 seconds, then held at 71°C in a convection oven until dried. Raisin waste consists of stems and raisins of poor quality.

Residue data for grapes and grape products are summarized in Table 29.

Figure 3. Processing of grapes to produce juice and pomace

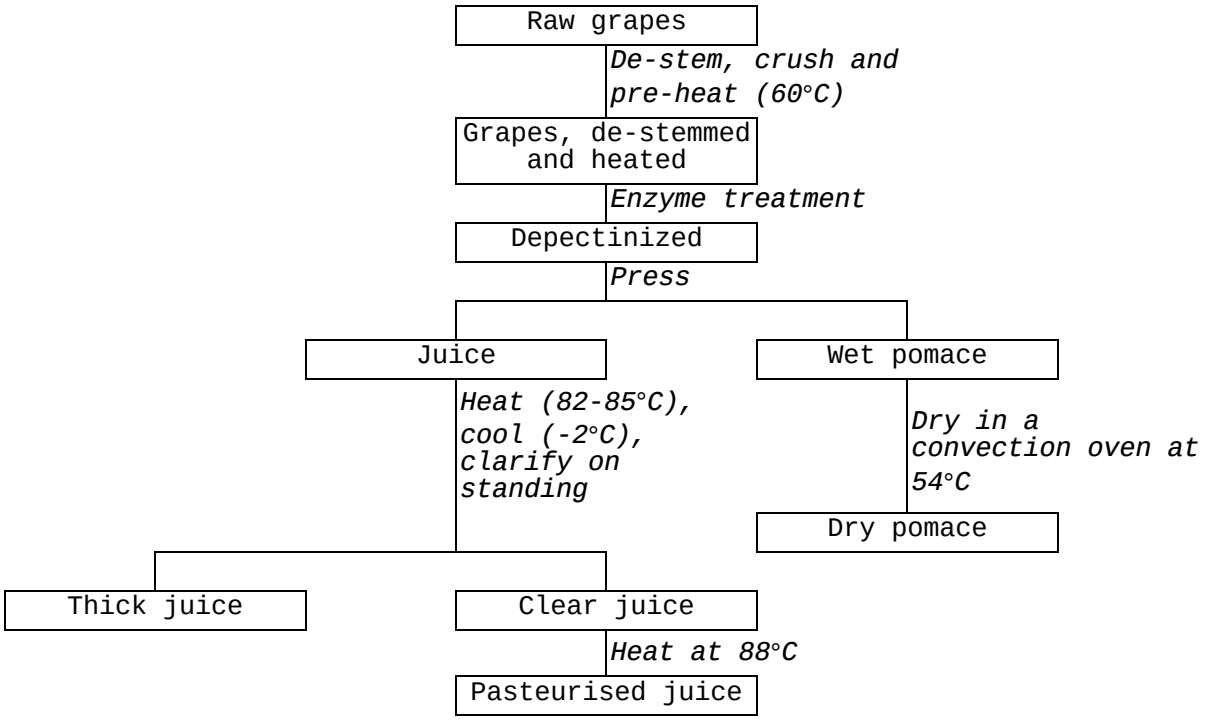


Table 29. Dithiocarbamate and ETU residue in grapes and grape products from a US (CA) 1987 processing study with maneb applied once at 14 kg ai/ha (4 × maximum label rate). Grapes were harvested 8 days after the final treatment (Bookbinder, 1988j).

Commodity	Dithiocarbamate residues, mg/kg as CS_2	ETU residues, mg/kg
Fresh grapes	10.5	0.067
Raw grapes at processor	6.6	0.026
Wet pomace	3.7	0.16
Dry pomace	3.9	0.50
Thick grape juice	0.46	5.0
Raisins	1.9	0.66
Raisin waste	10.6	1.3

In a US (CA) 1987 study maneb (SC formulation) was applied 7 times at 18 kg ai/ha (10 × maximum label rate) at a spray concentration of 9.5 kg ai/hl to a sugar beet crop, which was harvested 14 days after the final treatment (Bookbinder, 1988q). Sugar beet roots (220 kg) were sent for simulated commercial processing. Dithiocarbamate and ETU residues in the roots and the processed commodities are shown in Table 30.

Table 30. Dithiocarbamate and ETU residues in processed commodities from sugar beet treated 7 times with maneb at 18 kg ai/ha (10 × maximum label rate) and harvested 14 days after the final treatment (Bookbinder, 1988q).

Commodity	Dithiocarbamate residues, mg/kg as CS_2	ETU residues, mg/kg
Sugar beet roots	0.069	<0.01
Sugar beet molasses	<0.03	2.1[1]
White sugar	<0.03	<0.01
Dried beet pulp	0.088	0.028

[1] The authors suspected contamination of the molasses, but were not able to locate the source of contamination.

In a US (CA) 1988 processing study maneb (SC formulation) was applied 5 times at 9 kg ai/ha (5 × maximum label rate) at a spray concentration of 2.4 kg ai/hl to sweet corn, which was harvested 4 days after the final treatment (Bookbinder, 1989). Whole sweet corn ears (45 kg from each plot) were sent for simulated commercial processing. Whole sweet corn ears (4-5 kg) were also sent directly to the analytical laboratory. The report did not provide detailed information on the nature and duration of the washing and cleaning processes, or the times and temperatures of heating. Dithiocarbamate and ETU residues in the sweet corn and the processed commodities are shown in Table 31.

Table 31. Dithiocarbamate and ETU residues in processed commodities from sweet corn treated 5 times with maneb at 9 kg ai/ha (5 × maximum label rate) and harvested 4 days after the final treatment (Bookbinder, 1989).

Commodity	Dithiocarbamate residues, mg/kg as CS_2	ETU residues, mg/kg
Kernels (laboratory prepared)	0.21	0.01
Cobs + husks (laboratory prepared)	4.2	0.05
Kernels (commercial)	<0.03	<0.01
Husks (commercial)	5.4	0.11
Cobs (commercial)	0.76	<0.01
Blended husks and cobs (commercial)	3.4	0.04
Corn: cut, washed, blanched (commercial)	<0.03	<0.01

In a US (CA) 1987 processing study maneb (WP formulation) was applied 7 times at 5.4 kg ai/ha (2 × maximum label rate) in a spray concentration of 1.4 kg ai/hl to a tomato crop, which was harvested 3 days after the final treatment (Bookbinder, 1988r). Tomatoes (220 kg) were sent for simulated commercial processing (Figure 4). Dithiocarbamate and ETU residue data for the tomatoes and processed commodities are summarized in Table 32.

Tomatoes were peeled and filled into cans with fresh juice as the packing medium, then the cans were sealed and heated at 115°C for 50 minutes to produce canned whole tomatoes.

Figure 4. Processing of tomatoes field-sprayed with maneb (Bookbinder, 1988r).

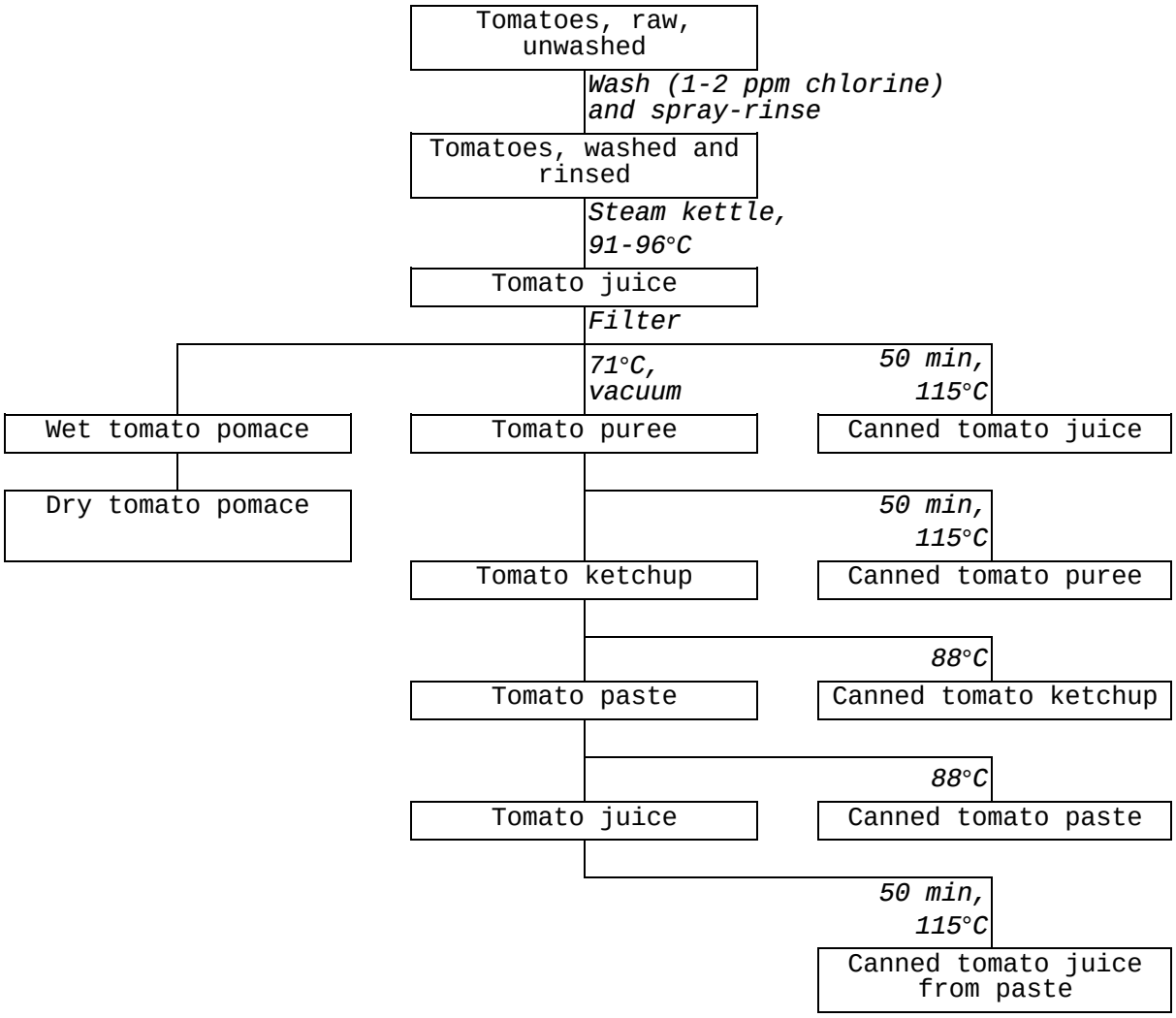


Table 32. Analysis of processed tomatoes for dithiocarbamates and ETU (Bookbinder, 1988r). The tomato crop had been treated 7 times with maneb at 5.4 kg ai/ha (2 × maximum label rate) and harvested 3 days after the final treatment. Residues are the means from analysis of 3 or 4 replicate samples.

Commodity	Dithiocarbamate residues, mg/kg as CS_2	ETU residues, mg/kg
Raw tomatoes (unwashed)	0.087	<0.01
Wet tomato pomace	0.07	<0.01
Dry tomato pomace	<0.03	0.031
Canned whole tomatoes	<0.03	<0.01
Tomato puree	<0.03	0.01
Tomato ketchup	<0.03	<0.01
Tomato paste	0.03	0.02
Tomato juice (from paste)	<0.03	0.02

Residues in the edible portion of food commodities

Removal of the wrapper leaves from cabbages reduced maneb residue levels by an average of 30%.

Removal of the wrapper leaves from lettuce reduced maneb residue levels by an average of 87%.

Maneb residue levels in washed spinach were about 25% lower than in unwashed spinach.

Washing beans did not significantly affect the maneb residue levels.

Maneb residue levels in apple juice were approximately 20-50% of the levels in the apples when no washing step was included in the process.

Maneb residue levels in beans were reduced by 99% in producing canned beans and baby food, and by 90% in producing frozen beans. Approximately 12-16% conversion of maneb to ETU took place in the production of canned beans and baby food.

Maneb levels in thick grape juice were approximately 5% of the levels in the fresh grapes, but there was approximately 55% conversion to ETU. Maneb levels in raisins were reduced by 82% compared with levels in fresh grapes, with approximately 14% conversion to ETU.

Dithiocarbamates and ETU were both undetectable in white sugar produced from maneb-treated sugar beet.

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

Information was made available to the Meeting on dithiocarbamate residue surveys on food items; the results are given in the mancozeb monograph.

METHODS OF RESIDUE ANALYSIS

Methods for dithiocarbamates and ETU are also described in the monograph on mancozeb.

Holstege and Westberg (1987) described the method used in the US trials for measuring maneb residues. Maneb residues in the sample were converted to CS_2 by reaction with stannous chloride in acid in a sealed tube at 100°C. An aliquot of the headspace was then analysed by GLC with flame-photometric detection (sulphur mode). The limit of determination was commonly 0.05 mg/kg (as maneb), or 0.03 mg/kg (as CS_2). Recoveries were usually good; instances of lower recoveries are reported in the section on supervised trials.

ETU residues in the US trials were measured by HPLC with UV detection (Rogers *et al.*, 1989). The sample was extracted with a water/ethanol mixture and the extract cleaned up on an alumina column. The limit of determination was 0.01 mg/kg. Difficulties were experienced with recoveries from some substrates; instances are reported in the section on supervised trials.

Samples from trials in The Netherlands were analysed for dithiocarbamates by the colorimetric method of Keppel (1971) and for ETU by an HPLC method (Lawrence *et al.*, 1981). Limits of determination were CS_2 0.01 mg/kg, ETU 0.002 mg/kg.

NATIONAL MAXIMUM RESIDUE LIMITS

The Meeting was aware of MRLs established for dithiocarbamates; a list is included in the mancozeb monograph.

APPRAISAL

Maneb, first evaluated in 1967, was scheduled for periodic re-evaluation at the 1993 JMPR (ALINORM 93/24A, para 133).

The Meeting received extensive information on GAP, supervised residue trials, metabolic fate in farm animals and crops, fate during processing, residues in food in commerce and at consumption, and methods of residue analysis.

When [^{14}C]maneb ([^{14}C]ethylenediamine) was fed to lactating goats for 5 days at the equivalent of 50 ppm maneb in the feed, the total ^{14}C residues in milk were close to a steady-state concentration by days 3 and 4. The levels in the morning milk samples, collected just before the daily dose, were always considerably lower than in the evening samples, which suggested that levels in milk would decrease rapidly when dosing ceased. Total ^{14}C residues were distributed among the tissues and milk, with the highest levels in the liver and kidney. Ethyleneurea, 1-(2-imidazolin-2-yl)-2-imidazolidinethione and ethylenethiourea (ETU) were identified in all the tissues and milk. The main primary metabolite was Jaffe's base (1-(2-imidazolin-2-yl)-2-imidazolidinethione). Much of the ^{14}C had been incorporated into natural products.

ETU was identified in the goat tissues and milk. The levels of ETU by direct chemical analysis were: liver 0.075 mg/kg, kidney 0.050 mg/kg, muscle 0.035 mg/kg, fat <0.01 mg/kg, milk 0.037 mg/kg.

When [^{14}C]maneb ([^{14}C]ethylenediamine) was fed to laying hens for 7 days at the equivalent of 51 ppm maneb in the feed, the total ^{14}C residues in egg whites had reached a plateau by days 5-6 while the total ^{14}C in egg yolk was still increasing at the end of the study. Total ^{14}C was distributed among the tissues, but the highest levels were in the liver and kidney. Ethyleneurea was the main metabolite. Ethyleneurea, 1-(2-imidazolin-2-yl)-2-imidazolidinethione and ethylenediamine were identified in all of the tissues, egg white and egg yolk. Much of the ^{14}C had been incorporated into natural products.

ETU was identified in all of the tissues (except skin) and eggs. ETU levels in tissues and eggs by direct chemical analysis were: liver 0.14 mg/kg, breast muscle 0.044 mg/kg, egg white 0.098 mg/kg, and egg yolk 0.039 mg/kg.

Lettuce plants treated with foliar sprays of [^{14}C]maneb were harvested and surface-rinsed with an EDTA solution to identify the components of the dislodgable residue. Surface residues included mainly maneb and the primary metabolites ethylenebisisothiocyanate sulphide, ethyleneurea and ethylenethiourea. The identified metabolites in the lettuce tissue included ethylenebisisothiocyanate sulphide, ethyleneurea, ethylenethiourea, ethylenediamine and *N*-acetylethylenediamine. Amino acids and protein were found to contain ^{14}C, which demonstrated that metabolites had been incorporated into the natural carbon pool. ETU accounted for 7% of the total ^{14}C residues in lettuce + rinsings, or 2.8% of the total ^{14}C in the rinsed lettuce.

Most of the ^{14}C residues were in the foliage, with less than 0.3% in the tubers, of potatoes harvested 17 days after the final foliar application of [^{14}C]maneb. The primary metabolites constituted only a minor part, less than 9%, of the residues in the tuber. ETU identified in the potato peel, but not in the body of the tuber, (0.02 mg/kg tuber, by direct chemical analysis) was thought to be the result of contamination rather than of metabolism. The metabolism may be interpreted in terms of a relatively rapid conversion of the primary metabolites to a common plant

metabolite such as glycine, which provides the mechanism for the ^{14}C to be incorporated widely into natural products.

In a metabolism study tomato plants were treated with foliar sprays of [^{14}C]maneb and harvested 24 days after the final application for metabolite identification and analysis. Much of the ^{14}C residue (49-63%) was dislodgable and was removed from harvested tomatoes when they were washed with a 1% EDTA solution. Maneb and EBIS (ethylenebisisothiocyanate sulphide) constituted the major part of the dislodgable residue; most of the ETU residue in the whole tomatoes was dislodgable. EBIS was the major metabolite identified in the whole tomato. The processes in tomato metabolism are similar to those in the other crops studied. The ^{14}C enters the metabolic carbon pool probably via glycine, from which it is incorporated into natural products.

Maneb is registered as a protective fungicide for use on pome fruits, stone fruits, berries and other small fruits, tropical and subtropical fruits, bulb vegetables, root and tuber vegetables, Brassica vegetables, leafy vegetables, stalk and stem vegetables, fruiting vegetables, legume vegetables, cereals, and tree nuts in many countries.

Typical spray concentrations for high-volume application are 0.15-0.3 kg ai/hl, and typical application rates for a wide range of crops are 1.3-3 kg ai/ha.

The Meeting received residue data from supervised trials on the following crops and commodities:

apples (*Netherlands, USA*), peaches (*USA*), grapes (*USA*);

onions (*USA, Netherlands*), broccoli (*USA*), cabbage (*USA*), cucumbers (*USA*), watermelons (*USA*), endive (*Canada*), kale (*USA*), lettuce (*Canada, USA*), beans (*USA*), peppers (*USA*), sweet corn (*USA*), tomatoes (*Netherlands, USA*), potatoes (*Netherlands, UK, USA*), sugar beet (*USA*), celery (*USA*);

barley (*Netherlands*), wheat (*Netherlands, UK, USA*), almonds (*USA*);

barley straw (*Netherlands*), maize forage (*USA*), wheat straw (*Netherlands, UK*) bean vines (*USA*), almond hulls (*USA*), sugar beet tops (*USA*).

Dithiocarbamate residues are expressed as mg CS_2/kg throughout.

The residue data on apples from The Netherlands could not be evaluated because the recommended use pattern was expressed in terms of spray concentration while the trial use pattern was expressed in terms of application rate. The US apple trials did not meet GAP conditions because the longest treatment-to-sampling interval in the trials was 30 days, but the US recommended use pattern requires a 77-day PHI.

Peach trials in the USA could not be evaluated because GAP information was not available.

The highest residues in grapes were 1.8 and 1.9 mg/kg in US trials where maneb was used within GAP conditions. The Meeting estimated a maximum residue level of 2 mg/kg for maneb uses on grapes.

Maneb use on green onions according to US GAP resulted in residues up to 7.4 mg/kg. The Meeting noted that analysis of a control sample of green onions produced 0.5 mg/kg as CS_2, probably resulting from endogenous CS_2. The Meeting estimated a maximum residue level of 10 mg/kg for spring onions.

The maneb application rate to bulb onions in Netherlands trials was 2.4 kg ai/ha, which is higher than the Netherlands recommended rate of 1.6 kg ai/ha. Residues in bulb onions are likely to arise from inadvertent spraying of exposed onions; the application rate will not be so influential on the residues. Dithiocarbamate residues in onions from the Netherlands

trials were low (0.1 mg/kg and lower).

The recommended PHI in the USA for bulb onions is 7 days, but in the trials onions were harvested on the same day as the final spray and the results could not be evaluated against the recommended use pattern. The Meeting noted the repeated detection of CS_2 in control samples at levels up to 0.13 mg/kg.

Broccoli in US trials was sampled 3 and 4 days after the final maneb application, but US GAP specifies a PHI of 7 days. The Meeting noted the detection of CS_2 in control samples at levels up to 0.55 mg/kg, which was consistent with other analyses on control broccoli (up to 0.79 mg/kg as CS_2). The Meeting did not estimate a maximum residue level for broccoli because of the limited number of trials. It drew attention to the endogenous CS_2 levels in broccoli and possible endogenous CS_2 in related crops.

The highest residue in untrimmed cabbage from US maneb trials in 1987 was 10 mg/kg, but residues in that trial seemed much higher than the others. These trials and four others in 1989 and 1990 also included analyses of trimmed cabbages; removal of the wrapper leaves reduced maneb residue levels by an average of 30%. The highest residue in a control sample was 0.59 mg/kg, suggesting that endogenous CS_2 levels could be similar to those reported for broccoli. The Meeting estimated a maximum residue level of 5 mg/kg for maneb uses on cabbage and noted that the correct portion of the sample for analysis included the wrapper leaves unless obviously withered or decayed.

Dithiocarbamate residues in cucumbers exceeded 1 mg/kg in one trial when maneb was used according to GAP in a series of trials in the USA in 1987 and 1989. The Meeting estimated a maximum residue level of 2 mg/kg for maneb uses on cucumber.

The highest dithiocarbamate residue in watermelons was 0.57 mg/kg when maneb was used according to US GAP. An experiment in one trial demonstrated that residues existed exclusively on the peel and not in the pulp. The Meeting estimated a maximum residue level of 1 mg/kg for maneb uses on watermelon.

Residues in kale from a series of supervised trials in the USA during 1987 were typically in the 4-8 mg/kg range, but the highest were 14 and 28 mg/kg. The questionable aspect of this trial was that residues on day 10 after the final application were somewhat higher than on day 7. The distribution of the results suggested that residues up to 15 mg/kg would be possible. The Meeting estimated a maximum residue level of 15 mg/kg for maneb uses on kale.

Supervised trials data from Canada and the USA were made available for uses of maneb on lettuce, leaf lettuce and cos lettuce. The commodity described as lettuce was taken to be head lettuce. The highest residues in the US trials on lettuce were in the 5-7 mg/kg range. The highest residues in cos lettuce from the Canadian trials were in the 6-9 mg/kg range. Only one trial was specified as leaf lettuce and residues were just under 1 mg/kg. The Meeting estimated maximum residue levels of 10 mg/kg for uses of maneb on cos lettuce and head lettuce.

Samples from the US trials on lettuce were analysed with and without wrapper leaves. Removal of the wrapper leaves reduced residue levels by an average of 87%.

US maneb trials on spinach could not be evaluated because no US GAP was available. The trials demonstrated that maneb residue levels in washed spinach were about 25% lower than in unwashed spinach.

The official PHI for maneb use on beans in the USA is 30 days; the PHI in the trials was 4 days so no MRL could be recommended. Washing the beans did not significantly affect the dithiocarbamate residue levels.

The use of maneb on sweet peppers in US trials in 1987-89 typically

produced residues in the 0.2-1 mg/kg range. The Meeting estimated a maximum residue level of 1 mg/kg for maneb uses on sweet peppers.

For sweet corn in the USA the registered application rate and PHI are 1.3 kg ai/ha and 7 days. The use pattern in the trials was 1.8 kg ai/ha with 4- and 5-day PHIs; consequently, a maximum residue level could not be estimated.

The highest dithiocarbamate residue in tomatoes arising from maneb use within US GAP was 2.0 mg/kg. Most commonly, residues were in the 0.1-0.5 mg/kg range. The Meeting estimated a maximum residue level of 2 mg/kg for the use of maneb on tomatoes.

Potato trials from The Netherlands could not be evaluated because application rates were double the official rate, 1.6 kg ai/ha, and because Netherlands GAP did not specify a PHI. Residues in potatoes in UK trials were undetectable (<0.01 mg/kg) with application at recommended rates and double recommended rates.

In 8 of the 9 US maneb trials on potatoes residues were not detected (<0.03 mg/kg), and in the other trial residues of 0.23 mg/kg were recorded for one plot. Maneb residues are generally immobile in the plant and residues on the tuber are only likely to arise if tubers are exposed above the soil during spraying. The Meeting estimated a maximum residue level of 0.2 mg/kg for maneb uses on potatoes.

In one sugar beet trial in the USA residues were much higher than in the remaining trials. Residues in the sugar beet tops up to 76 and 88 mg/kg seemed excessive for an application rate of 1.8 kg ai/ha. The Meeting was unable to estimate maximum residue levels for sugar beet or sugar beet leaves and tops because the number of trials was too small.

Supervised maneb trials on celery in the USA and barley in The Netherlands could not be evaluated because no relevant GAP was available.

When maneb was used within GAP on wheat in The Netherlands and the UK dithiocarbamate residues were mostly undetectable or in the 0.01-0.05 mg/kg range. The highest residue (0.65 mg/kg) from one plot of a trial in The Netherlands appeared to be anomalous; residues in wheat from the other plot in the same trial were undetectable (<0.01 mg/kg). The Meeting estimated a maximum residue level of 0.2 mg/kg for the use of maneb on wheat.

Residues in almonds from maneb trials in the USA were mostly undetected (<0.03 mg/kg). The Meeting estimated a maximum residue level of 0.05 mg/kg for almonds from the use of maneb.

Residues in wheat straw from The Netherlands and the UK ranged up to 2.1 mg/kg for registered uses of maneb. The Meeting estimated a maximum residue level of 5 mg/kg for wheat straw and fodder, resulting from maneb uses.

GAP information was not available for maize forage or bean vines, so trials data could not be evaluated for MRL purposes. Barley straw data from Netherlands trials evaluated against registered wheat uses supported the estimated maximum residue level in wheat straw and fodder resulting from maneb uses.

Many of the residues in almond hulls were in the 3-10 mg/kg range in US maneb trials on almonds, but the distribution of results suggested that residues in the 10 to 20 mg/kg would be likely from use according to GAP. The Meeting estimated a maximum residue level of 20 mg/kg for almond hulls.

Processing studies were made available to the Meeting on apples, beans, grapes, sugar beet, sweet corn and tomatoes.

Maneb residues in apple juice were approximately 20-50% of the levels in apples when no washing step was included in the process. Maneb residues were retained in the pomace fraction. ETU residue levels in the juice were lower than in the raw commodity.

Beans field-treated with maneb were passed through a simulated commercial process to produce canned beans, frozen beans and pureed beans (baby food). Dithiocarbamate residue levels were much reduced in frozen beans and were at very low levels in canned beans and not detectable in baby food. Heat was used in the production of these commodities; consequently ETU was produced in all of them.

In the processing of maneb-treated grapes dithiocarbamate residue levels in wet pomace and thick grape juice were approximately 60% and 7% respectively of the level in the raw grapes. Juice was heated at 82-85°C before being separated into thick juice and clear juice. The heating caused substantial conversion to ETU, the level in the thick juice being 5 mg/kg.

Dithiocarbamate and ETU residues were not detectable (<0.03, <0.01 mg/kg respectively) in white sugar produced from sugar beet field treated with exaggerated application rates (tenfold) of maneb in the USA.

Dithiocarbamate and ETU residues were not detectable (<0.03, <0.01 mg/kg respectively) in sweet corn (cut, washed and blanched) produced in a commercial process from sweet corn field-treated with a fivefold application rate of maneb in the USA.

Dithiocarbamate and ETU residues were at or about limits of quantification (0.03 and 0.01 mg/kg respectively) in canned whole tomatoes, tomato puree, tomato ketchup and tomato juice commercially produced from tomatoes field-sprayed with maneb at twice the recommended application rate in the USA. It is likely that the first step, commercial washing, reduced residue levels substantially.

No freezer storage stability studies for maneb were available. Because of the nature of the residue the Meeting agreed that the results of the storage stability studies for mancozeb would also apply to maneb.

Information on dithiocarbamate surveys of food items is included in the monograph on mancozeb.

Analytical methods for maneb residues rely on conversion by acid hydrolysis to CS_2, which is then measured colorimetrically or by GLC. Information on methods for dithiocarbamates and ETU is included in the monograph on mancozeb.

RECOMMENDATIONS

The recommendations for maneb are included in the monograph on dithiocarbamates.

REFERENCES

1. Ballantyne, L.G. 1992. Maneb: nature of the residue in lettuce. Elf Atochem BR-88-29, HLA 6120-137. Hazleton Laboratories America, Inc., USA. Unpublished.

2. Bookbinder, M.G. 1988a. Maneb and ethylene thiourea: magnitude of the residue in cucumber treated by ground equipment in California, Michigan, North Carolina, South Carolina and Texas, 1987. File 28-MAN/88015, includes studies 25287, 22687, 22787, 22887, 25387. Orius Associates Inc., USA. Unpublished.

3. Bookbinder, M.G. 1988b. Maneb and ethylene thiourea: magnitude of the residue in watermelons treated by ground equipment in Georgia and Texas, 1987. File 28-MAN/88016, includes studies 22987, 23087. Orius Associates Inc., USA. Unpublished.

4. Bookbinder, M.G. 1988c. Maneb and ethylene thiourea: magnitude of the residue in peppers treated by ground equipment in California, Florida and Texas, 1987. File 28-MAN/88017, includes studies 25687, 25887, 26387. Orius Associates Inc., USA. Unpublished.

5. Bookbinder, M.G. 1988d. Maneb and ethylene thiourea: magnitude of the residue in dry beans and vines treated by ground equipment in California, Colorado, Michigan, North Dakota, and Nebraska, 1987. File 28-MAN/88019, includes studies 25387, 20787, 20887, 21087, 20987. Orius Associates Inc., USA. Unpublished.

6. Bookbinder, M.G. 1988e. Maneb and ETU: residues in commodities processed from snap beans treated by ground equipment in New York, 1987. File 28-MET/88020. Orius Associates Inc., USA. Unpublished.

7. Bookbinder, M.G. 1988f. Maneb and ethylene thiourea: magnitude of the residue in succulent beans and vines treated by ground equipment in California, Delaware, Michigan, New York, Oregon and Wisconsin, 1987. File 28-MAN/88021, includes studies 21187, 21287, 21387, 21487, 21687, 25087. Orius Associates Inc., USA. Unpublished.

8. Bookbinder, M.G. 1988g. Maneb and ethylene thiourea: magnitude of the residue in cabbage treated by ground equipment in California, Michigan, New York, and Texas, 1987. File 28-MAN/88022, includes studies 21587, 22587, 23287, 21987. Orius Associates Inc., USA. Unpublished.

9. Bookbinder, M.G. 1988h. Maneb and ethylene thiourea: magnitude of the residue in broccoli treated by ground equipment in California, 1987. File 28-MAN/88023, includes study 25187. Orius Associates Inc., USA. Unpublished.

10. Bookbinder, M.G. 1988i. Maneb and ethylene thiourea: magnitude of the residue in sweet corn and sweet corn forage treated by ground equipment in Georgia, Illinois, Minnesota, New York, Wisconsin, and Oregon, 1987. File 28-MAN/88024, includes studies 22487, 22187, 22287, 22387, 24887. Orius Associates Inc., USA. Unpublished.

11. Bookbinder, M.G. 1988j. Maneb and ETU: residues in commodities processed from grapes treated by ground equipment in California, 1987. File 28-MAN/88025. Orius Associates Inc., USA. Unpublished.

12. Bookbinder, M.G. 1988k. Maneb and ethylene thiourea: magnitude of the residue in grapes treated by ground equipment in California, 1987. File 28-MAN/88027, includes studies 024, 028. Orius Associates Inc., USA. Unpublished.

13. Bookbinder, M.G. 1988l. Maneb and ethylene thiourea: magnitude of the residue in kale treated by ground equipment in California, New Jersey, and Texas, 1987. File 28-MAN/88028, includes studies 26687, 26987, 27187. Orius Associates Inc., USA. Unpublished.

14. Bookbinder, M.G. 1988m. Maneb and ethylene thiourea: magnitude of the residue in lettuce treated by ground equipment in California, 1987. File 28-MAN/88029, includes studies 23187, 26587. Orius Associates Inc., USA. Unpublished.

15. Bookbinder, M.G. 1988n. Maneb and ethylene thiourea: magnitude of the residue in potatoes treated by ground equipment in California, Idaho, Maine, North Dakota, and Oregon, 1987. File 28-MAN/88030, includes studies 23587, 23687, 23787, 23887, 23987. Orius Associates Inc., USA. Unpublished.

16. Bookbinder, M.G. 1988o. Maneb and ethylene thiourea: magnitude of the residue in spinach treated by ground equipment in New Jersey, Tennesee, and Texas, 1987. File 28-MAN/88031, includes studies 25987, 26187, 26287. Orius Associates Inc., USA. Unpublished.

17. Bookbinder, M.G. 1988p. Maneb and ethylene thiourea: magnitude of the residue in sugar beets treated by ground equipment in California, Idaho, Minnesota, and North Dakota, 1987. File 28-MAN/88032, includes studies

24087, 24187, 24287, 24387. Orius Associates Inc., USA. Unpublished.

18. Bookbinder, M.G. 1988q. Maneb and ETU: residues in commodities processed from sugar beet roots treated by ground equipment in California, 1987. File 28-MET/88034. Orius Associates Inc., USA. Unpublished.

19. Bookbinder, M.G. 1988r. Maneb and ETU: residues in commodities processed from tomatoes treated by ground equipment in California, 1987. File 28-MET/88035. Orius Associates Inc., USA. Unpublished.

20. Bookbinder, M.G. 1988s. Maneb and ethylene thiourea: magnitude of the residue in tomatoes treated by ground equipment in California, Florida, Michigan and Texas, 1987. File 28-MAN/88036, includes studies 24487, 24587, 24687, 24787. Orius Associates Inc., USA. Unpublished.

21. Bookbinder, M.G. 1988t. Maneb and ethylene thiourea: magnitude of the residue in green onions treated by ground equipment in Texas, 1987. File 28-MAN/88037, includes study 25487. Orius Associates Inc., USA. Unpublished.

22. Bookbinder, M.G. 1988u. Maneb and ethylene thiourea: magnitude of the residue in or on watermelons treated by ground equipment in California, 1988. File 4-MAN/88172, includes study 23487. Orius Associates Inc., USA. Unpublished.

23. Bookbinder, M.G. 1988v. Maneb and ethylene thiourea: magnitude of the residue in or on cucumbers treated by ground equipment in Florida, 1988. File 4-MAN/88173, includes study 21787. Orius Associates Inc., USA. Unpublished.

24. Bookbinder, M.G. 1988w. Maneb and ethylene thiourea: magnitude of the residue in or on cabbage treated by ground equipment in Florida, 1988. File 4-MAN/88173, includes study 21887. Orius Associates Inc., USA. Unpublished.

25. Bookbinder, M.G. 1988x. Maneb and ethylene thiourea: magnitude of the residue in or on peppers treated by ground equipment in North Carolina in 1988. File 4-MAN/88174, includes study 10688. Orius Associates Inc., USA. Unpublished.

26. Bookbinder, M.G. 1988y. Maneb and ethylene thiourea: magnitude of the residue in spinach treated by ground equipment in California, 1988. File 4-MAN/88175, includes study 25587. Orius Associates Inc., USA. Unpublished.

27. Bookbinder, M.G. 1988z. Maneb and ethylene thiourea: magnitude of the residue in or on kale treated by ground equipment in Florida, 1988. File 4-MAN/88176, includes study 26787. Orius Associates Inc., USA. Unpublished.

28. Bookbinder, M.G. 1988aa. Maneb and ethylene thiourea: magnitude of the residue in or on green onions treated by ground equipment in California and Florida, 1987-88. File 4-MAN/88177, includes studies 23387, 23487. Orius Associates Inc., USA. Unpublished.

29. Bookbinder, M.G. 1988bb. Maneb and ethylene thiourea: magnitude of the residue in potatoes treated at an exaggerated rate by ground equipment in California, 1987. File 28-MAN/88185, includes study 23587. Orius Associates Inc., USA. Unpublished.

30. Bookbinder, M.G. 1988cc. Maneb and ethylene thiourea: magnitude of the residue in or on green onions treated by ground equipment in Arizona, 1988-89. File 40-MAN/89050, includes study 88137. Orius Associates Inc., USA. Unpublished.

31. Bookbinder, M.G. 1989. Maneb and ETU: residues in sweet corn commodities processed from sweet corn treated by ground equipment in California, 1988. File 28-MAN/89060, includes 13588. Orius Associates Inc., USA. Unpublished.

32. Byast, T.H. 1992. Determination of maneb, mancozeb and ethylene thiourea (ETU) residues in samples of potato and winter wheat, grain and

straw. Study OA00011/R52628. Oxford Analytical, UK. Unpublished.

33. Holstege, D. and Westberg, G.L. 1987. Determination of maneb in crops. Method MTF-88AM-005. Morse Laboratories, Inc., USA. Unpublished.

34. Hubert, T.D. 1991. Maneb: nature of the residue in tomatoes (analytical phase). Project HLA6120-135. Hazleton Laboratories America, Inc., USA. Unpublished.

35. Kallio, H. and Salorinne, L. 1990. Comparison of onion varieties by headspace gas chromatography-mass spectrometry. J. Agric. Fd Chem., 38, 1560-1564.

36. Keppel, G.E. 1971. Collaborative study of the determination of dithiocarbamate residues by a modified carbon disulfide method. J. Assoc. Off. Analyt. Chem., 54, 528-532.

37. Lawrence, J.F., Iverson, F., Hanekamp, H.B., Bos, P. and Frei, R.W. 1981. Liquid chromatography with UV absorbance and polarographic detection of ethylenethiourea and related sulfur compounds. J. Chromatog. 212, 245-250.

38. Pitt, J.L. 1989a. Maneb and its metabolite ethylene thiourea: magnitude of the residue in apple. Study 10-88. Project BR-88-16,includes 10A-88, 10B-88, 10C-88. Morse Laboratories, Inc., USA. Unpublished.

39. Pitt, J.L. 1989b. Maneb and its metabolite ethylene thiourea: magnitude of the residue in processed apple fractions. Study 37-88, includes 37A-88, 37B-88. Project BR-88-17. Pennwalt Corporation, USA. Unpublished.

40. Pitt, J.L. 1989c. Maneb and its metabolite ethylene thiourea: magnitude of the residue in peach. Study 39-88, includes 39A-88, 39B-88, 39C-88, 39F-88. Pennwalt Corp., USA. Unpublished.

41. Pitt, J.L. 1990a. Maneb and its metabolite ethylene thiourea: magnitude of the residue in onion. Study BR-88-15, includes 38A-88, 38B-88, 38C-88, 38D-88, 38E-88, 38F-88, 38G-88, 38H-88, 38I-88, 38J-88, 38K-88, 38L-88. Pennwalt Corporation, USA. Unpublished.

42. Pitt, J.L. 1990b. Maneb and its metabolite ethylene thiourea: magnitude of the residue in broccoli. Study BR-89-09, includes 53A-89, 53B-89, 53C-89. Pennwalt Corporation, USA. Unpublished.

43. Pitt, J.L. 1991a. Maneb and its metabolite ethylene thiourea: magnitude of the residue in almond. Study BR-88-13. Project ML88-0040-PEN includes 36A-88, 36B-88, 36C-88, 36D-88. Morse Laboratories, Inc., USA. Unpublished.

44. Pitt, J.L. 1991b. Maneb and its metabolite ethylene thiourea: magnitude of the residue in dry bean. Study BR-89-05, includes 54A-89, 54B-89, 54C-89. Atochem North America, USA. Unpublished.

45. Pitt, J.L. 1991c. Maneb and its metabolite ethylene thiourea: magnitude of the residue in bell pepper. Study BR-89-08, includes 47A-89, 47B-89, 47C-89. Atochem North America, USA. Unpublished.

46. Pitt, J.L. 1991d. Maneb and its metabolite ethylene thiourea: magnitude of the residue in cabbage. Study BR-89-10, includes 43B-89, 43E-89, 43J-89, 43L-89. Atochem North America, USA. Unpublished.

47. Pitt, J.L. 1991e. Maneb and its metabolite ethylene thiourea: magnitude of the residue in cucumber. Study BR-89-12, includes 51A-89, 51B-89, 51C-89. Atochem North America, USA. Unpublished.

48. Pitt, J.L. 1991f. Maneb and its metabolite ethylene thiourea: magnitude of the residue in lettuce. Study BR-89-13, includes 46B-89, 46J-89, 46L-89. Atochem North America, USA. Unpublished.

49. Pitt, J.L. 1991g. Maneb and its metabolite ethylene thiourea: magnitude of the residue in potato. Study BR-89-14, includes 38A-89, 38B-89, 38C-89,

38D-89. Atochem North America, USA. Unpublished.

50. Pitt, J.L. 1991h. Maneb and its metabolite ethylene thiourea: magnitude of the residue in sweet corn. Study BR-89-15, includes 49A-89, 49B-89, 49C-89. Atochem North America, USA. Unpublished.

51. Pitt, J.L. 1991i. Maneb and its metabolite ethylene thiourea: magnitude of the residue in tomato. Study BR-89-16, includes 48A-89, 48B-89, 48C-89, 48D-89. Atochem North America, USA. Unpublished.

52. Pitt, J.L. 1991j. Maneb and its metabolite ethylene thiourea: magnitude of the residue in watermelon. Study BR-89-17, includes 50A-89, 50B-89, 50C-89. Atochem North America, USA. Unpublished.

53. Pitt, J.L. 1991k. Maneb and mancozeb and their metabolite ethylene thiourea: magnitude of the residue in tomato. Study BR-89-23, includes 61A-89, 61B-89, 61C-89, 61D-89, 61E-89, 61F-89, 61G-89, 61H-89. Atochem North America, USA. Unpublished.

54. Pitt, J.L. 1991l. Maneb and mancozeb and their metabolite ethylene thiourea: magnitude of the residue in celery. Study BR-89-24, includes 62A-89, 62B-89, 62C-89, 62D-89, 62E-89, 62F-89. Atochem North America, USA. Unpublished.

55. Pitt, J.L. 1992. Maneb and its metabolite ethylene thiourea: magnitude of the residue in snap bean. Study BR-89-07, includes 52A-89, 52B-89, 52C-89. Atochem North America, USA. Unpublished.

56. Pitt, J.L. 1993. Maneb and mancozeb and their metabolite ethylene thiourea: magnitude of the residue in grape. Study BR-90-30, includes 24A-90, 24B-90, 24C-90, 24D-90, 24E-90, 24F-90. Elf Atochem North America, USA. Unpublished.

57. Ripley, B., Frank, R., Braun, H.E., Ritcey, G., McEwen, F.L., Valk, M., Knibbe, E., Burchat, C.S., and Harris, C.R. 1989. Pesticide research report. ICAR #84100761, Canada.

58. Rogers, E., Tufts, K. and Westberg, G.L. 1989. Determination of ethylene thiourea in crops. Method MTF-88AM-004. Morse Laboratories, Inc., USA. Unpublished.

59. Wright, M.C. and Malik, N.S.A. 1992. Maneb: nature of the residue in potatoes. Study BR-88-30, includes HLA6120-136 and XBL89007. Hazleton Laboratories America, Inc., and Xenobiotic Laboratories, Inc., USA. Elf Atochem, North America, USA. Unpublished.

60. Wright, M.C. and Ussary, J.P. 1993. Maneb: nature of the residue in tomatoes. Study BR-88-28, includes HLA6120-135 and R098901. Hazleton Laboratories America, Inc., and Research for Hire, USA. Elf Atochem, North America, USA. Unpublished.

61. Wu, J. 1990a. Metabolism of ^{14}C-maneb in lactating goats - analysis and quantitation of metabolites and/or the corresponding natural products in milk and tissues. Project XBL 88003, RPT0019. XenoBiotic Laboratories, Inc., USA. Unpublished.

62. Wu, J. 1990b. Metabolism of ^{14}C-maneb in laying hens - analysis and quantitation of metabolites and/or the corresponding natural products in eggs and tissues. Project XBL 88004, RPT0016. XenoBiotic Laboratories, Inc., USA. Unpublished.

63. Wu, J. 1990c. Metabolism of ^{14}C-maneb in potatoes - nature of the residue in potatoes: analysis and quantitation of metabolites. Report XBL89007. XenoBiotic Laboratories, Inc., USA. Unpublished.

Cross-index of report numbers, study numbers and references.

Reports and studies are listed in numerical and alphabetical order and each is linked to a reference. Reference numbers are signified by # numbers. Report numbers in the data tables where there were only summary data or reports without authors or report titles are not included in this cross-index.

[Blank page]

[Blank page]

PHORATE (112)

EXPLANATION

Residue and analytical aspects of phorate were evaluated by the JMPR in 1977, 1984, 1990, 1991 and 1992. At the 21st Session of the CCPR (1989) several delegations considered the MRL for phorate on carrots (0.5 mg/kg) to be too high and it was held at Step 7B. Information on current use patterns in the United Kingdom and Australia was provided to the 1990 Joint Meeting which concluded that as they were essentially the same as those recorded in 1977 the MRL for carrots (0.5 mg/kg) was still valid. The MRL was maintained at Step 7B by the 1991 CCPR pending further information from the United Kingdom and evaluation by the JMPR. The United Kingdom informed the 1992 CCPR that it had changed its GAP. The MRL was moved to Step 7C at the 1993 CCPR awaiting further information from the United Kingdom and Australia.

Residue data and updated information on GAP in the United Kingdom were made available to the Meeting and the Australian GAP available to the 1990 JMPR was confirmed.

USE PATTERN

Updated information on authorized uses of phorate on carrots in the United Kingdom is given in Table 1.

Table 1. Registered uses of phorate on carrots

Country	Application		PHI (weeks)
	No.	Rate, g ai/100 m row	
Australia	1 per crop either as a band at sowing or to established plants	1 kg ai/ha	10
UK	1 per crop	6 mineral soil 10 organic soil	21

RESIDUES RESULTING FROM SUPERVISED TRIALS

The MRL for carrot was originally proposed by the 1977 JMPR on the basis of residue trials

conducted in the United Kingdom with 2 or 3 applications of 1.5 or 3 kg ai/h. It was based on a PHI of 120 days (17 weeks).

Information was received from the United Kingdom on supervised trials conducted in 1983 and 1984 on both mineral and organic soils involving single applications by soil incorporation at planting. Total phorate residues in carrots treated in mineral soils at 4 to 8 g ai/100 m row after PHIs of 16 to 24 weeks ranged up to 0.25 mg/kg. The corresponding residues in carrots treated at 8 to 10 g ai/100 m row in organic soils after PHIs of 20 to 21 weeks ranged from 0.02 to 0.06 mg/kg. Further trials at higher rates (16-22 g ai/100 m row) and PHIs of 20-27 weeks showed a maximum residue of 0.22 mg/kg although most were <0.1 mg/kg. Results are shown in Table 2.

The data to support the application to established plantings (10 weeks pre-harvest) recommended in the Australian use pattern are limited.

Table 2. Phorate residues in carrots from supervised trials in the United Kingdom. Underlined residues are from treatments according to GAP.

Year	Application		Weeks after sowing	Soil type	Residue, mg/kg	Ref.
	g ai/ 100 m row	No.				
1983 (June)	2	1	31	Mineral	0.064, 0.047	1
	5	1	31		0.148, 0.167	
	22	1	27		0.181	
	2.8		27	Organic	0.005	
	17		27		0.053	
1984 (June)	2	1	24	Mineral	0.028, 0.020, 0.039	2*
					0.014, 0.005, 0.008	
	4	1	24		<u>0.059</u>, <u>0.060</u>, <u>0.036</u>	
					<u>0.016</u>, <u>0.016</u>, <u>0.016</u>	
	8	1	24		<u>0.159</u>, <u>0.115</u>, <u>0.253</u>	
					<u>0.028</u>, <u>0.017</u>, <u>0.049</u>	
	4	1	20	Organic	0.021, 0.031, 0.062	
					0.036, 0.027, 0.025	

Year	Application		Weeks after sowing	Soil type	Residue, mg/kg	Ref.
	g ai/ 100 m row	No.				
	8	1	20		**0.024, 0.042, 0.062**	
					0.027, 0.025, 0.032	
	16	1	20		0.056, 0.025, 0.050	
					0.071, 0.059, 0.220	

Residues are expressed as total phorate
* Residues >90% phorate sulphone

Table 2 (contd.)

Year	Application	Weeks after sowing	Soil type	Residue, mg/kg	Ref.

	g ai/ 100 m row	No.			P	PSO	PSO_2	Total	
1984	6	1	16	Sandy loam	-	0.006	0.110	0.12	3
					-	0.009	0.136	0.15	
					-	0.006	0.052	0.06	
		1	24		-	0.002	0.016	0.02	
					-	<0.001	0.019	0.02	
					-	<0.001	0.030	0.03	
	10	1	13	Organic	0.009	0.122	0.074	0.21	
					0.036	0.211	0.028	0.28	
					0.003	0.037	0.029	0.07	
		1	21		0.003	0.032	0.029	0.06	
					0.001	0.008	0.030	0.04	

P = parent phorate
PSO = phorate sulphoxide
PSO_2 = phorate sulphone
All residues calculated as phorate equivalent

APPRAISAL

New information on use patterns and data on residues in carrots resulting from supervised trials were evaluated. The MRL proposed for carrots (0.5 mg/kg) by the 1977 Joint Meeting has been the subject of much discussion at the CCPR as it would appear that the use is limited to the United Kingdom and Australia. GAP in the United Kingdom will result in residues that are unlikely to exceed 0.2 mg/kg. The residue data available were inadequate to permit an effective assessment of the residues likely to result from Australian GAP.

RECOMMENDATIONS

On the basis of the new GAP information and residue data from the United Kingdom the meeting concluded that the residue level listed below is suitable for establishing a maximum residue limit.

Definition of the residue: Sum of phorate, its oxygen analogue and their sulphoxides and sulphones, expressed as phorate.

Commodity	Recommended MRL (mg/kg)

CCN	Name	New	Previous	PHI on which based (weeks)
VR 0577	Carrot	0.2	0.5	21

PROCYMIDONE (136)

EXPLANATION

Procymidone was reviewed by the Joint Meeting in 1981, 1989 and 1990. The 1992 CCPR held all MRLs at step 7B in view of the need to ensure that the available residue data, most of which were reviewed in 1981, reflected current GAP.

The manufacturer indicated that residue data on common beans, cucumbers, grapes, head lettuce, bulb onions and tomatoes, together with information on current GAP, would be submitted to the JMPR.

Extensive information was provided by the manufacturer and some member countries on use patterns, with some residue data from supervised field trials and the monitoring of food commodities. The new information is evaluated in this monograph.

USE PATTERNS

Procymidone is a preventive and curative fungicide which is moderately systemic. It is especially effective for the control of Botrytis, Cocliobolus, Helmintosporium, Sclerotinia and Monilia species in fruits, vegetables, field crops and ornamental plants.

The current recommended or registered use patterns are summarized in Table 1.

RESIDUES RESULTING FROM SUPERVISED TRIALS

Additional residue data were provided from several countries (Tables 2-8). Most of the trials were conducted in accordance with the national use patterns or at about double rates. In the Tables the countries are indicated by the following codes: ARG-Argentina, AUL-Australia, AUS-Austria, BEL-Belgium, FRA-France, GER-Germany, HUN-Hungary, ITA-Italy, JPN-Japan, LUX-Luxembourg, NET-Netherlands, NZE-New Zealand, POR-Portugal, SAF-South Africa,SPA-Spain, SWI-Switzerland, URU-Uruguay, VEN-Venezuela. Underlined residues are from treatments according to GAP.

Cherries, sour. A cherry orchard was treated with Sumilex 50 WP at the maximum rate according to registered uses in Hungary. Samples were taken from days 1 to 21 after application. The residues found are shown in Table 2.

Grapes. Residue trials were conducted by Landis Europe SA at sites in seven regions in Europe where wine grapes are grown. Two of the trials were located in France, two in Italy, two in Spain, and one in Hungary. Six separate trials at each of the sites (a total of 42 trials) provided data on the residues of procymidone in or on mature, whole wine grapes and in wine prepared from them. In each test, four applications of Sumisclex 50 WP or Sumisclex 50 L were made using ground equipment. The first three applications were at growth stages of petal fall, cluster closing and colour change. The last application was at a prescribed time before harvest (28, 21, 15, and 5 days) in order to test the effect of the PHI on the residue level. Application was at the maximum rate found on any labels (1x) for four tests per trial site and at twice the maximum rate (2x) for two tests per site for the respective countries. These application rates were equivalent to 250 and 500 g ai/ha for Spain, and 750 and 1500 g ai/ha for France, Italy and Hungary.

Table 1. Registered or approved uses of procymidone

Crop	Country	Application				PHI, days
		Formulation	No.	g ai/hl	g ai/ha	
Almond	Lebanon	50 WP	*	25-50		14
Apple	Lebanon	50 WP	*	25-50		14
Apple	Japan	50 WP	4	25		90
Apricot	Bulgaria	50 WP	3-4	75		14
Apricot	France	50 WP, 50 FL	*	75		15
Apricot	Japan	50 WP	3	37.5		14
Apricot	Jordan	50 WP	*	25-50		14
Apricot	Lebanon	50 WP	*	25-50		14
Apricot	Turkey	50 WP	*	100		15
Barley	Czechoslovakia	50 WP	1[1]		1500	
Beans, Adzuki	Japan	50 WP	4	25-50		21
Beans	Chile	50 WP	*		375-600	
Beans	France	50 WP, 50 FL	1-2		375	15
Beans	Jordan	50 WP	*	25-50		3
Beans	Lebanon	50 WP	*	25-50		3
Beans	New Zealand	25 FL	2		1000	3
Beans	Poland	50 WP	2-3		500	
Beans	South Africa	25 SC	2		375	14
Beans	Thailand	50 WP	At 7-10 d intervals	15-30		
Beans (seed)	Czechoslovakia	50 WP	2	50		
Beans, Dwarf	Austria	WG 50 %	*	50		7
Beans, Dwarf	Germany	WG 50 %	3		380	7
Beans, Dwarf	Netherlands	50 WP/FL	2		500	14
Beans, Faba	Australia	50 WP	4-5		250	
Beans, Faba	Australia	27.5 FL	4-5		275	2
Beans, Green	Australia	50 WP	2[6]	50	500-750	
Beans, Green	Australia	27.5 FL	2		550-825	2
Beans, Haricot	Belgium	50 WP			500	14
Beans, Kidney	Japan	50 WP	4	25-50		21
Beans, Navy	Australia	50 WP	2		500-750	
	Australia	27.5 FL	2		550-825	2
Beans, Runner	Netherlands	50 WP/FL	1-2		1000	14
Blackberries	Switzerland	50 WG	2		750-1000	14
Cabbage	Greece	WG 50 %	8-10	62.5		3
Cabbage	Japan	50 WP	4	50-75		14
Cabbage	Jordan	50 WP	*	25-50		3
Cacao	Venezuela	50 WP	*		500-750	3
Carrot	Hungary	50 WP	2-3		500	14
Carrot	Yugoslavia	50 FL				

Crop	Country	Application				PHI, days
		Formulation	No.	g ai/hl	g ai/ha	
Cauliflower	Greece	WG 50 %	8-10	62.5		3
Celery	Japan	50 WP	5	25-50		14
Celery	Jordan	50 WP	*	25-50		3
Celery	Venezuela	50 WP	*		500-750	3
Cherries	Hungary	50 WP	3		600-750	14
Cherries	Japan	50 WP	3	25-37.5		14
Cherries	Jordan	50 WP	*	25-50		14
Cherries	Lebanon	50 WP	*	25-50		14
Cherries, sour	Yugoslavia	50 WP		50		28
Cherries, sour	Yugoslavia	50 FL		75		28
Chicory	France	50 WP/(50 FL)	3-4		750	21
Citrus	Peru	50 WP	1	25-50		
Clover	Czechoslovakia	50 WP	1-2		500	
Coffee	Thailand	50 WP	At 7-10 d intervals	15-30		
Cotton	Thailand	50 WP	At 7-10 d intervals	15-30		
Cucumbers	China	50 WP	*		300-375	
Cucumbers	CIS	50 WP	*	30-37.5		
Cucumbers	Greece	WG 50 %	8-10	50		1
Cucumbers	Italy	12.3/49 WP[2]	1-2	12.3-24.6		
Cucumbers	Japan	25 D	6		750	1
	Japan	30 SA	6		1800	1
	Japan	50 WP	6	25		1
Cucumbers	Jordan	50 WP	*	25-50		3
Cucumbers	Lebanon	50 WP	*	25-50		3
Cucumbers	Netherlands	50 WP, 50 FL	*	25		3
Cucumbers	Poland	50 WP	*	50-75		3
Cucumbers	Portugal	50 WP	*	75		7
Cucumbers	Rep. of Korea	50 WP	3		800	5
Cucumbers	Turkey	50 WP	*	37.5		15
Currant	Czechoslovakia	50 WP	1	75[3]		
Durian	Thailand	50 WP	*[1]	25-50		
Egg plant	Greece	WG 50 %	8-10	50		
Egg plant	Japan	25 D	6		750	1
	Japan	30 SA	6		1800	1
	Japan	50 WP	6	25-50		1
Egg plant	Jordan	50 WP	*	25-50		3
Egg plant	Lebanon	50 WP	*	25-50		3
Egg plant	Netherlands	50 WP, 50 FL	*	25		3
Egg plant	Poland	50 WP	*	50-75		3
Endive	France	50 WP, 50 FL	1[3]	30		
	France	50 WP, 50 FL	1[4]		1500	

Crop	Country	Application				PHI, days
		Formulation	No.	g ai/hl	g ai/ha	
	France	50 WP, 50 FL	1-2[5]		1500	
Endive	Netherlands	50 WP, 50 FL	2		750	
Fruits	Thailand	50 WP	1[6]	40-80		
Garlic	Australia	27.5 FL	1		5.5 g/kg	
	Australia	50 WP	1		5 g/kg[7]	
Garlic	France	50 WP, 50 FL	1[8]		1.5 g/kg[8]	
Garlic	Poland	50 WP	1[8]		2 g/kg	
	Poland	50 WP	1	150		
Garlic	Thailand	50 WP	At 10-14 d intervals	25-50		
Garlic	Uruguay	50 WP	1		500	
Garlic	Venezuela	50 WP	1		500-1000	1
Gherkins	Netherlands	50 WP, 50 FL	*	25		3
Grapes	Argentina	50 WP	2	37.5-50		28
Grapes	Australia	27.5 FL	4		550	5
	Australia	50 WP	2		500	5
	Austria	WG 50 %	2	37.5-50		21
Grapes	Bulgaria	50 WP	4	50		14
Grapes	Chile	50 WP	*	25-37.5		
Grapes	CIS	50 WP	4		500-750	30
Grapes	Czechoslovakia	50 WP	1-2	50		21
Grapes	France	50 WP, 50 FL	1-2		750	14
Grapes	Germany	WG 50 %	3	37.5	380-680	28
Grapes	Greece	WG 50 %	*	50		28[9]
	Greece	WG 50 %	*	62.5		14[10]
Grapes	Hungary	50 WP	2		500-750	14
Grapes	Italy	12.3/49 WP[2]	3-4	24.6		21
Grapes	Italy	22.5/45 WP[11]	*	45-56.2		21
	Italy	25 SC	3-4	50-75		21
	Italy	50 FL	3-4	50-75		21
	Italy	50 WP	3-4	50-75	250-500[12]	21
	Italy	5 D	3-4		1000-1250	21
	Italy	75 DFL	3-4	52.5-75		21
Grapes	Luxembourg	50 WP	3	37.5		28
Grapes	New Zealand	25 FL	3		1000	1
Grapes	Peru	50 WP	2		250-500	
Grapes	Portugal	50 WP	4	75		21
Grapes	Rep. of Korea	50 WP	*		1250	3
Grapes	Romania	50 WP	*		500-750	
Grapes	South Africa	25 SC	1-2	50		7[9], 28[10]
Grapes	Spain	3 E 3 D)	*		600-900	15
	Spain	50 WP	*	75		5

Crop	Country	Application				PHI, days
		Formulation	No.	g ai/hl	g ai/ha	
Grapes	Switzerland	50 WG	4	50	1000	
Grapes	Thailand	50 WP	At 7-10 d intervals	10-15		
Grapes	Turkey	50 WP	*	37.5		21
Grapes	Uruguay	50 WP	1-2	37.5-50	500-750	28
Grapes	Venezuela	50 WP	*		500-1000	3
Grapes	Yugoslavia	50 FL	4		500	28
Grapes	Yugoslavia	50 WP			500	28
	Zimbabwe	50 WP	4		500	21
Kiwifruit	Italy	25 SC	5	75-100		14
Lemon	Thailand	50 WP	At 10-14 d intervals	25-50		
Lettuce	Argentina	50 WP	*	50		7
Lettuce	Australia	27.5 FL	3-4		310-660	2
	Australia	27.5 FL	1	275	1000	
	Australia	50 WP	3-4		281-600	2
	Australia	50 WP	1	250	1000	2
Lettuce	Austria	WG 51 %	*	50		7
Lettuce	Belgium	50 WP			5-7.5 g/acre	21
Lettuce	France	50 WP, 50 FL	3-4		750	21
Lettuce	Greece	WG 50 %	8-10	62.5		
Lettuce	Japan	50 WP	5	25-50		7
Lettuce	Jordan	50 WP	*			3
Lettuce	Lebanon	50 WP	*			3
Lettuce	Netherlands	50 WP, 50 FL	1		2000	
Lettuce	Poland	50 WP	2		500-750	21
Lettuce	Rep. of Korea	50 WP	*		700	3
Lettuce	Taiwan	50 WP	4		375-500	3
Lettuce	Uruguay	50 WP	*	50		
Lettuce	Venezuela	50 WP	*		500-750	3
Lettuce	Yugoslavia	50 FL	3		300	35
	Yugoslavia	50 WP			300-750	
Litchi	Thailand	50 WP	At 10-14 d intervals	25-50		
Longan	Thailand	50 WP	At 10-14 d intervals	25-50		
Lupins	Australia	50 WP			1 g/kg[7]	
	Australia	27.5 FL			1.1 ml/kg	
Mango	Thailand	50 WP	At 10-14 d intervals	25-50		
Melon	France	50 WP, 50 FL	*		750	7
Melon	Netherlands	50 WP, 50 FL	*	25		3
Melon	Poland	50 WP	*	50-75		3
Melon	Thailand	50 WP	At 7-10 d	15-30		

Crop	Country	Application				PHI, days
		Formulation	No.	g ai/hl	g ai/ha	
			intervals			
Nectarine	Uruguay	50 WP	*		500-750	7
Onions	Argentina	50 WP	*	50		7
Onions	Australia	27.5 FL	1		11 g/kg	28
	Australia	27.5 FL	2		1000	28
	Australia	27.5 FL	1	55[7]		28
	Australia	50 WP	1		10 g/kg red	28
	Australia	50 WP	1		2000	28
	Australia	50 WP	1		1000	28
	Australia	50 WP	1	500[7]		28
Onions	Austria	WG 50 %	*	50		7
Onions	Egypt	50 WP			5 g/kg[8]	
Onions	Greece	WG 50 %	8-10	62.5		1
Onions	Japan	50 WP	5	25		1
Onions	Jordan	50 WP	*	25-50		3
Onions	Netherlands	50 WP, 50 FL	1		250	28
Onions	New Zealand	10 % granule	1		2000	1
	New Zealand	25 FL	*		1000	21
Onions	Poland	50 WP	1[1]		2.5 g/kg	14
	Poland	50 WP	1		5-15 g/kg	14
	Poland	50 WP	1[7]	150		14
Onions	Spain	50 WP		75		5
Onions	Thailand	50 WP	At 10-14 d intervals	25-50		
Onions	Uruguay	50 WP	*		750	
Onion	Venezuela	50 WP	*		500-1000	1
Orange	Japan	25 D	3		2000	
	Japan	30 SA	3		3000	
	Japan	50 WP	3	37.5-75		
Papaya	Thailand	50 WP	At 10-14 d intervals	25-50		
Parsley	Hungary	50 WP	2-3		500	14
Peaches	Chile	50 WP	*		375-600	
Peaches	France	50 WP, 50 FL	2	75		8
Peaches	Italy	12.3/49 WP[2]	3-5	24.6-30.75		14
	Italy	25 SC	3	75-100		14
	Italy	25 SC	4-5	50-75		14
	Italy	50 WP	3	75-100		14
	Italy	50 WP	4-5	50-75		14
	Italy	50 FL	3	75-100		14
	Italy	50 FL	4-5	50-75		14
	Italy	75 DFL	3	70-97.5		14
	Italy	75 DFL	4-5	52.5-75		14

Crop	Country	Application				PHI, days
		Formulation	No.	g ai/hl	g ai/ha	
Peaches	Japan	50 WP	3	25-37.5		3
Peaches	Jordan	50 WP	*	25-50		3
Peaches	Lebanon	50 WP	*	25-50		14
Peaches	Morocco	50 WP	*	250		3
Peaches	Romania	50 WP	*	50-75		
Peaches	South Africa	25 SC	2	37.5		7
Peaches	Uruguay	50 WP	*	25-37.5	500-750	7
Peaches	Venezuela	50 WP	*		500-750	3
Peanut	Japan	50 WP	4	25-50		21
Peanut	South Africa	25 SC	2-3		375	14
Peanut	Venezuela	50 WP	*	50-100		21
Pear	Italy	50 WP	*	50-75		
	Italy	50 FL	*	50-75		14
	Italy	25 SC	*	50-75		14
	Italy	75 DFL	*	52.5-75		14
Peas	France	50 WP, 50 FL	1-2		375	15
Peas	Germany	WG 50 %	1		500	14
Peas	South Africa	25 SC	2-3		375	14
Peppers	Austria	WG 50 %	*	50		7
Peppers	Greece	WG 50 %	8-10	62.5		3
Peppers	Jordan	50 WP	*	25-50		3
Peppers	Lebanon	50 WP	*	25-50		3
Peppers	Portugal	50 WP	*	75		7
Peppers	Thailand	50 WP	At 7-10 d intervals	15-30		
Peppers, Chilli	Thailand	50 WP	At 7-10 d intervals	15-30		
Peppers, Green	Hungary	50 WP	*		500	3
Peppers, Green	Japan	30 SA	5		1800	1
	Japan	50 WP	5	25-50		7
Peppers, Green	Netherlands	50 WP, 50 FL	*	25		3
Peppers, Red	Poland	50 WP	*	50-75		3
Peppers, Red	Rep. of Korea	50 WP	5		800	7
Plum	France	50 WP, 50 FL	1	75		8
Plum	Lebanon	50 WP	*	25-50		14
Prune, dried	France	50 WP, 50 FL	2-3	75		8
Pome fruits	Switzerland	50 WG	2		1000	
Potatoes	Japan	50 WP	4	25-37.5		21
Potatoes	Jordan	50 WP	*	25-50		3
Potatoes	Lebanon	50 WP	*	25-50		3
Potatoes	South Africa	25 SC	1-2		125-250	35
Potatoes	Thailand	50 WP	At 10-14 d intervals	25-50		

Crop	Country	Application				PHI, days
		Formulation	No.	g ai/hl	g ai/ha	
Quince	Turkey	50 WP	*	100		15
Rambutan	Thailand	50 WP	At 10-14 d intervals	25-50		
Rape seed	China	50 WP	*		225-450	
Rape seed	CIS	50 WP	*		225-450	
Rape seed	France	50 WP, 50 FL	1-2		375-750	
Rape seed	Germany	WG 50 %	1		500	56
Rape seed	Poland	50 WP	*		750	56
Rape seed	Yugoslavia	50 WP			300-750	42
Raspberries	Hungary	50 WP	3		500-600	14
Raspberries	Poland	50 WP	2		1250	7
Raspberries	Switzerland	50 WG	2		750-1000	14
Rice	Thailand	50 WP	At 7-10 d intervals	15-30		
Shallot	Netherlands	50 WP, 50 FL	1[7]		1000	14
Soya bean	Japan	50 WP	4	25-50		21
Soya bean	Venezuela	50 WP	*		500-1000	21
Squash, Summer (Courgettes)	Netherlands	50 WP, 50 FL	*	25		3
Stone fruits	Australia	27.5 FL	1	27.5-55[6]		1
	Australia	50 WP	4-5	25-37.5		1
	Australia	50 WP	1	50[6]		
Stone fruits	Chile	50 WP	*	25-50		
Stone fruits	Greece	WG 50 %	*	50-75		
Stone fruits	New Zealand	25 FL	2-3		750-1000	1
Stone fruits	Switzerland	50 WG	2	50		
Strawberries	Austria	WG 50 %	*	37.5		10
Strawberries	Belgium	50 WP			750	3
Strawberries	Bulgaria	50 WP	3	50		14
Strawberries	Chile	50 WP	*	25-37.5		
Strawberries	CIS	50 WP	2		500	
Strawberries	Czechoslovakia	50 WP	2	50	1000	
Strawberries	France	50 WP, 50 FL	3-4		750	7
Strawberries	Germany	WG 50 %	3	37.5	750	7
Strawberries	Greece	WG 50 %	*	50-62.5		3
Strawberries	Hungary	50 WP	3		500-600	14
Strawberries	Italy	12.3/49 WP[2]	3	24.6-30.7		14
	Italy	22.5/45 WP	*	45-56.2		14
	Italy	25 SC	3	40-50		14
	Italy	50 FL	3	40-50		14
	Italy	50 WP	3	40-50		14
	Italy	5 D	3		1000	14
	Italy	75 DFL	3	37.5-52.5		14

Crop	Country	Application				PHI, days
		Formulation	No.	g ai/hl	g ai/ha	
Strawberries	Japan	25 D	3		750	7
	Japan	30 SA	3		1800	1
	Japan	50 WP	3	50		3
Strawberries	Jordan	50 WP	*	25-50		3
Strawberries	Netherlands	50 WP, 50 FL	*		225-375	14
Strawberries	New Zealand	25 FL	3		500-1000	1
Strawberries	Peru	50 WP	1-3		250-500	3
Strawberries	Poland	50 WP	1-2		750-1250	7
Strawberries	Portugal	50 WP	*	75		7
Strawberries	Romania	50 WP	*	50		
Strawberries	Rep. of Korea	50 WP	*		700	2
	Rep. of Korea	FW Smoke pellet	3		360	2
Strawberries	Spain	3 E (3 D)	*		600-900	5
	Spain	50 WP		75		5
Strawberries	Switzerland	50 WG	2	50		14
Strawberries	Taiwan	50 WP	1-2		250	5
Strawberries	Thailand	50 WP		10-15[7]		
Strawberries	Uruguay	50 WP	1-3		500-750	
Strawberries	Venezuela	50 WP	*		500-1000	1
Strawberries	Yugoslavia	50 FL	3		750	21
	Yugoslavia	50 WP			300-750	21
Sugar beet	Czechoslovakia	50 WP		150		
Sugar beet	Poland	50 WP	1[6]	0.5		
	Poland	50 WP	1[1]	0.75-1		
Sunflower	Bulgaria	50 WP	*		500	14
Sunflower	CIS	50 WP	1[11]		2 g/kg	
Sunflower	Czechoslovakia	50 WP	2		500	
Sunflower	Hungary	50 WP	*		500	21
Sunflower	Romania	50 WP			500 g/ton[1]	
Sunflower	Yugoslavia	50 WP			300-750	42
Tea	Thailand	50 WP	At 7-10 d intervals	15-30		
Tomatoes	Australia	27.5 FL	2-3		310-660	2
	Australia	50 WP	2-3		281-600	2
Tomatoes	Bulgaria	50 WP	*	50		14
Tomatoes	Chile	50 WP	*	25-37.5		
Tomatoes	China	50 WP	*		375-750	
Tomatoes	CIS	50 WP	*			
Tomatoes	France	50 WP, 50 FL	As needed		750	7
Tomatoes	Greece	WG 50%	8-10	50		1
Tomatoes	Hungary	50 WP	*		500	3
Tomatoes	Italy	12.3/49 WP[2]	*	24.6-30.7		14

Crop	Country	Application				PHI, days
		Formulation	No.	g ai/hl	g ai/ha	
	Italy	22.5/45 WP	8-10	45-56.2		14
	Italy	25 SC	*	40-50		14
	Italy	50 FL	*	40-50		14
Tomatoes	Italy	50 WP	At 15 d intervals	40-50		14
	Italy	5 D	*		750-1000	14
	Italy	75 DFL	*	37.5-52.5		14
Tomatoes	Japan	25 D	3		750	3
	Japan	30 SA	3		1800	1
	Japan	50 WP	3	25-50		3
Tomatoes	Jordan	50 WP	*	25-50		3
Tomatoes	Lebanon	50 WP	*	25-50		3
Tomatoes	Netherlands	50 WP, 50 FL	*		370-750	3
Tomatoes	Morocco	50 WP	3-4		375-500	7
Tomatoes	New Zealand	25 FL	3		1000	3
Tomatoes	Poland	50 WP	*	50-75		3
Tomatoes	Portugal	50 WP	*	75		7
Tomatoes	Rep. of Korea	50 WP	5		800	3
Tomatoes	Romania	50 WP	*	50		
Tomatoes	South Africa	25 SC	1-3	25		3
Tomatoes	Thailand	50 WP	At 7-10 d intervals	15-30		
Tomatoes	Turkey	50 WP	*	37.5		15
Tomatoes	Venezuela	50 WP	*		500-750	3
Tomatoes	Yugoslavia	50 FL			500	21
	Yugoslavia	50 WP			300-750	
Tomatoes	Zimbabwe	50 WP	1-2		250	7
Vegetables	Algeria	50 WP	*	25-50		21
Vegetables	Chile	50 WP	*		375-600	
Vegetables	Spain	3 E (3 D)	*		600-900	5
	Spain	50 WP	*	75		5
Vegetables	Thailand	50 WP	*	25-50		
Watermelon	Japan	25 D	5		750	21
Watermelon	Jordan	50 WP	*	25-50		3
Watermelon	Lebanon	50 WP	*	25-50		3

Notes: see next page.

Notes to Table 1:

* Not specified
[1] Seed treatment
[2] Procymidone/thiram 12.3/49 WP
[3] Conservation in cold store
[4] Forcing
[5] Forcing in a forcing bed
[6] Stored product protection
[7] Oil treatment before sowing/planting
[8] Bulb treatment against white rot before planting
[9] Wine grapes
[10] Table grapes
[11] Procymidone/chlorothalonil 22.5/45 WP
[12] Dry treatment

For tests using 2x rates, grapes were harvested 15 and 5 days after the last application. Grapes were treated at the higher rates in order to ensure sufficient residues for wine-processing trials. All analyses were performed at the same laboratory. The results are summarized in Table 3.

Examination of the results indicates that average procymidone levels from treatments at the 1x application rate varied considerably between sites. In all, 84 separate analyses of grapes were completed for 1x applications at PHIs of 15 days or longer. None of the 84 results exceeded the 5 mg/kg TMRL for procymidone in grapes. An additional 28 analyses were completed on grapes after 1x application and a 5-day PHI. Only one residue exceeded 5 mg/kg, with a level of 5.39 mg/kg.

Raspberry. Raspberry fields were treated with Sumilex 50 WP at maximum rates according to registered uses in Hungary and Poland. Samples were taken from 1 to 14 days after application. The residues found are shown in Table 2.

Table 2. Residues of procymidone in various crops from supervised trials.

Commodity, Country, Year	Application			Residues (mg/kg) at days after last application					Ref.
	Form.	kg ai/ha	No.	0-1	3	7	14	21	
Cherries, Sour, HUN, 1992	WP	0.7	1	0.8	0.63	0.52	0.77[2] 0.52	0.32	3
Egg plant, POL, 1992	WP	0.6	1	0.6	0.93	0.82	0.53[2] 0.42		9
Potato[6], JPN, 1977	WP	0.5	4				0.02[3]	0.05 0.03[4] 0.02[5]	22
Potato, JPN, 1977	WP	0.5	4				0.08[3]	0.05[4] 0.02 0.03[5]	23
Raspberry, HUN, 1992	WP	0.6	1	4.38	3.98	0.97	0.56[2] 0.52[7] 0.21[8]		1
Raspberry, POL, 1992	WP	1.25	2	3.1	2	1.4	0.73[2] 0.51		2
Green beans, SPA, 1990	WP	0.75	1	1.28	0.73[1]	0.59	0.36[2] 0.51		21
	WP	0.75	1	1.0	0.81[1]	0.46	0.43[2] 0.27		21
	WP	0.75	1	1.56	0.65[1]	0.56	1.15[2] 0.39		21

Commodity, Country, Year	Application			Residues (mg/kg) at days after last application					Ref.
	Form.	kg ai/ha	No.	0-1	3	7	14	21	
	WP	0.75	1	1.31	0.69[1]	0.67	0.89[2] 0.17		21
Green beans, SPA, 1992	WP	0.45	1	0.98	0.67	0.38	0.30	0.08	21
	WP	0.45	1	0.99	0.78	0.28	0.26	0.07	21
	WP	0.45	1	0.95	0.86	0.36	0.32	0.08	21
Haricot beans, FRA, 1991	L	0.75	2			0.39			14
	L	0.75	1			0.54			14
	L	0.75	1			0.57			14
Sunflower	WP	0.5	3 1					0.12[4] 0.11[4] 0.04	6

[1] Samples were taken 2 days after application
[2] Samples were taken 10 days after application
[3] Samples were taken 19 days after application
[4] Samples were taken 28 days after application
[5] Samples were taken 30 days after application
[6] Peeled potatoes
[7] Unripe fruits
[8] Ripe fruits

Strawberry. Supervised trials were reported from Hungary and Spain. Sumilex 50 WP was applied according to registered uses at rates of 0.6-1 kg ai/ha. Residues detected in samples taken between days 0 and 21 following application are shown in Table 4.

Shallots. Procymidone was used for seed treatment in France at a rate of 150 g ai/100 kg seed. At harvest the crop contained 0.05 mg/kg residue 116 days after sowing (Macdonald *et al.*, 1992e).

Table 3. Residues of procymidone in grapes from supervised trials

Country, location	Application			Residues (mg/kg) at days after last application			
	Form.	kg ai/ha	Sray l/ha	5	15	21	28
FRA, Tours	50L	0.79	198	2.16 2.63 2.68 3.05	2.59 2.24 2.64 2.65	2.45 2.32 2.51 1.69	2.12 2.16 1.93 2.40
	50L	1.57	198	9.62 9.66 5.45 5.47	5.72 7.09 5.51 8.87		
FRA, Avignon	50L	0.75	194	1.12 1.31 1.20 1.38	1.06 1.07 0.86 0.86	1.07 0.92 1.09 1.50	0.55 0.92 1.08 0.98
	50L	1.50	194	4.21 3.88 3.49 5.03	2.16 3.11 2.90 2.85		
SPA, Toledo	50 WP	0.25	200	0.74 1.72 1.53 1.98	1.47 0.55 0.95 1.43	0.59 0.56 3.10 1.47	0.99 0.84 0.61 0.41
	50 WP	0.48	200	4.82 3.75 4.03 2.60	2.24 2.94 3.29 2.39		
SPA, Cordoba	50 WP	0.24	197	0.42 0.42 0.34	0.56 0.36 0.40	0.28 0.37 0.40	0.18 0.30 0.29

Country, location	Application			Residues (mg/kg) at days after last application			
	Form.	kg ai/ha	Sray l/ha	5	15	21	28
				0.35	0.55	0.32	0.26
	50 WP	0.50	197	1.04 0.69 1.08 0.96	1.06 0.70 0.80 1.27		
SPA (Ref. 21)	WP	0.6		0.40	0.24	0.22	
ITA, Asti	50 WP	0.74	324	2.93 2.53 2.18 1.78	2.14 2.46 2.13 2.50	1.83 2.93 1.99 2.61	2.72 3.62 2.28 2.69
	50 WP	1.42	324	4.62 4.09 3.28 3.74	5.64 4.83 4.84 6.55		
ITA, Bologna	50 WP	0.74	1200	2.83 2.72 2.11 3.19	2.94 2.85 3.53 3.15	2.56 2.62 3.17 2.56	2.22 2.72 2.06 2.13
	50 WP	1.49	1200	7.24 5.29 7.44 5.80	6.29 5.23 5.05 4.97		
HUN, Balaton	50 WP	0.75	168	2.30 5.39 1.33 4.16	4.60 3.63 1.59 1.89	2.11 2.04 2.56 2.70	1.81 1.35 2.12 2.19
	50 WP	1.52	168	3.95 6.79 4.80 3.84	4.36 6.25 5.26 4.47		

Table 4. Residues of procymidone in strawberries from supervised trials. All single applications of WP.

Country Year	Kg ai/ha	Residues (mg/kg) at days after last application					Ref.
		0	3	7	14	21	
HUN, 1987	0.6	0.39 0.59 0.42	0.3 0.44 0.42	0.17* 0.4* 0.22*			10
SPA, 1985	1	2.77	2.20	1.30	0.50		21
SPA, 1986	1	1.70	1.06	3.71	1.60	0.80	21
		1.90	1.30	3.50	2.10	0.80	
		1.74	0.92	4.24	1.50	0.96	
SPA, 1987	0.75	4.20	2.55	1.33	0.80	0.52	21
		4.90	4.30	1.65	1.25	0.46	
		4.80	4	1.80	1.50	0.52	
SPA, 1988	1.25	2.80	1.50	1.06	0.60	0.50	21

		1.75	1.35	0.95	0.56	0.40	
		1.80	1.30	0.87	0.40	0.28	

* Samples taken 5 days after application.

Cucumber. Field trials were conducted in Spain in 1990 and 1991, applying procymidone one to five times at rates of 0.5-0.91 kg ai/ha. The residues found in samples taken between 2 and 16 days after application are shown in Table 5.

Egg plant. Residues from a supervised field trial in Poland, applying procymidone at a rate of 0.6 kg ai/ha, are shown in Table 2.

Peppers, Sweet. The results of supervised trials in glasshouses in Spain are summarized in Table 6.

Tomatoes. Supervised trials were conducted in France and Italy following recommended use patterns. Procymidone was applied 4 or 6 times at rates of 0.5 and 0.75 kg ai/ha respectively. Residues at 0 to 21 days after the lastp application are presented in Table 7.

Table 5. Residues of procymidone in cucumbers from supervised trials in Spain in 1990 and 1991. All WP. Reference 13.

Year	Application		Residues (mg/kg) at days after last application				
	kg ai/ha	No.	2	5	8	12-13	16
1991	0.5	5	0.06	0.38	0.11	0.12	0.06
	0.56	5	0.51	0.41	0.20	0.08	0.09
	0.56	5	0.73	0.29	0.16	0.08	0.08
1990	0.75	1	0.28	0.09[1]	0.11[2]	0.07[3]	
1991	0.91	5	0.90	0.30	0.21	0.07	0.06

[1] Samples were taken 7 days after last application.
[2] Samples were taken 10 days after last application.
[3] Samples were taken 15 days after last application.

Table 6. Residues of procymidone in peppers, grown indoors, from supervised trials in Spain. All single applications of WP, 0.75 kg ai/ha. Reference 21.

Year	Residues (mg/kg) at days after last application				
	0	2-3	7	10 -11	15
1989	2.61	0.42	0.73	0.85	0.32
	2.51	0.52	0.57	0.72	0.48
	1.17	0.71	1.30	0.51	0.52
	0.88	0.44	0.72	0.42	0.52
1988		0.91	1.2	0.51	0.27
		0.33	1.07	0.42	0.34
		0.9	0.17	0.22	0.16
		0.93	0.34	0.33	0.18

Lettuce. Supervised field trials were performed in France and Spain applying Sumilex 50 WP and L formulations one to four times at rates of 0.75 or 0.85 kg ai/ha. Residues measured in samples taken between days 0 and 21 are summarized in Table 8.

Beans. Green beans and haricot beans were treated according to registered uses with powder and liquid formulations of procymidone in France and Spain. Residues found after 0-21 days are shown in Table 2.

Potato. Trials were reported from Japan in which Sumilex 50 WP was applied four times at 0.5 kg ai/ha. Whole and peeled potatoes were analyzed for procymidone residues. The results are shown in Table 2.

Table 7. Residues of procymidone in tomatoes from supervised trials in France and Italy, 1991.

Country	Application			Residues (mg/kg) at days after last application					Ref.
	Form.	kg ai/ha	No.	0-1	3	7	14	21	
FRA	L	0.75	6		0.96				11
	L	0.75	6		0.41				11
ITA	WP	0.5	4	0.48	0.45	0.51	0.43	0.27	12
	WP	0.5	4	0.93	1.08	0.32	0.46	1.58	12
	WP	0.5	4	0.56	0.40	0.19	0.14	0.06	12
	WP	0.5	4	0.78	0.67	0.28	0.12	0.08	12
	WP	0.5	4	0.39 0.37	0.11[1]	0.15	0.12[2]		12
	WP	0.5	4	0.35	0.46	0.32	0.17[2]		12

[1] Sample was taken 4 days after last application.
[2] Samples were taken 10 days after last application.

Sunflower. Supervised field trials were conducted in Hungary, applying Sumilex 50 WP one and three times at the maximum registered rate. Samples were taken 21 and 28 days after the last application. The residues detected in the seeds are shown in Table 2.

Table 8. Residues of procymidone in lettuce from supervised trials.

Country, Year	Application			Residues (mg/kg) at days after last application					Ref.
	Form.	kg ai/ha	No.	0	3	7	14	21	
SPA, 1990	WP	0.75	1	3.8 4.05 9.6	1.3 0.9 2.1	0.98 0.85 1.05	0.38 0.4 0.38	0.2 0.42 0.4	20
FRA, 1991	L	0.75	3					0.17	15
	L	0.75	4					0.15	15
	L	0.75	3					0.17	15
SPA, 1989	WP	0.85	1	4.85 4.5 4.25		0.65 0.55 0.53		0.07* 0.05* 0.07*	20

* Samples were taken 22 days after last application

FATE OF RESIDUES

In processing

Grapes were harvested from the 42 separate treated test plots detailed above ("Residues resulting from supervised trials", where other details are given) and fermented into wine. At each site at least 50 kg of grapes were collected from the control plot. For the treated plots, at least 14 kg of grapes were collected from each of the sub-plots (replicates), and subsequently combined at the laboratory into a 50-kg processing sample for vinification. The processing samples were shipped to the processing laboratory in Grabels, France. All shipments were completed within 24 hours of harvest.

Processing of the grapes began shortly after receipt. The vinification procedure used for grapes from each site was chosen to match the procedure used locally in the country or region of origin. The use of different procedures, each typical of the locale in which the grapes originated, allowed a realistic estimate of residues to be expected in commercially-produced wine.

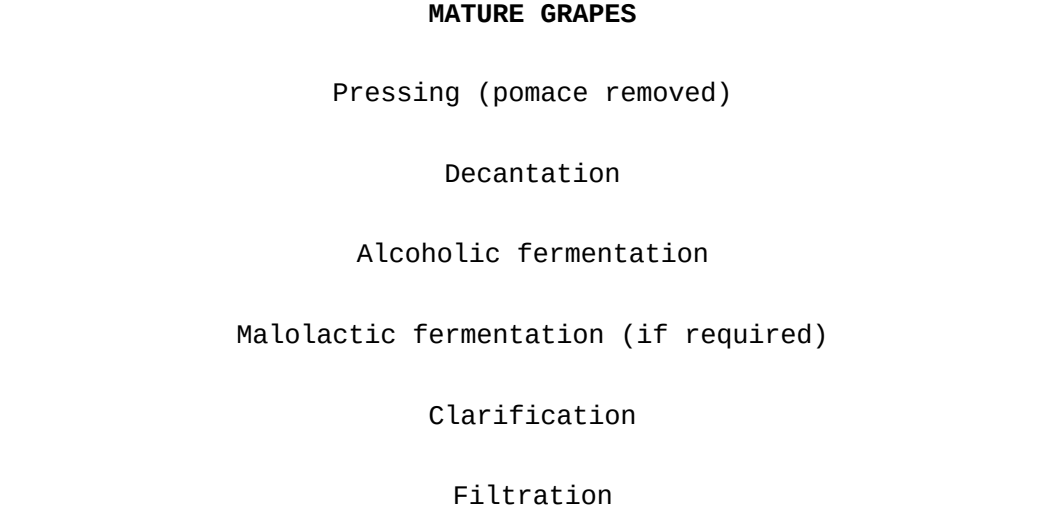
The process for making white wine is shown in Figure 1. The basic steps followed in producing all white wines are pressing to remove pomace, decantation, alcoholic fermentation, clarification and filtration before bottling. The principal differences between the local practices were in the amount and point of addition of potassium bisulphite, the amount of added rehydrated yeast, the addition of sugar to the fermentation media, the addition of clarification aids, and the use of a pectolytic enzyme.

The process for making red wine is showed in Figure 2. The basic steps followed in producing all red wines are crushing, alcoholic fermentation, pressing to remove pomace, malolactic fermentation, clarification, and filtration before bottling. The principal differences between local practices were in the amount, if any, of added rehydrated yeast and the use of natural or added malolactic bacteria.

Each grape lot was sampled and analyzed for procymidone. Fresh wine was sampled at the time of bottling, which varied with the vinification process, and analyzed for procymidone and 3,5-dichloroaniline (DCA) which may be formed during or just after vinification.

The results are summarized in Table 9. Apparent residues of procymidone in or on control samples of whole grapes from the 7 locations were <0.01 mg/kg (undetectable) or 0.01 mg/kg (just detectable). Average levels of procymidone in the grapes used for vinification ranged between 0.20 mg/kg and 2.68 mg/kg in or on the 84 samples from tests at 1x application rates which were harvested after 5, 15, 21 or 28 days. Residues of procymidone ranged between 0.81 mg/kg and 7.48 mg/kg in or on the corresponding 42 samples from application at the 2x rate harvested after PHIs of 5 and 15 days. Procymidone residues in 56 samples of fresh wine from test plots treated at the 1x rate ranged between 0.01 and 0.43 mg/kg. Residues of procymidone in 26 samples of fresh wine from treatments at the 2x rate ranged between 0.13 and 1.35 mg/kg.

Figure 1. The basic process for making white wine.



WINE

Bottling

Residues of DCA were below the limit of detection (0.01 mg/kg) in untreated control samples of fresh wine, representing wine from all seven locations. Residue levels of DCA in fresh wine from each location at the 1x application rates ranged between <0.01 mg/kg and 0.08 mg/kg. DCA levels in wine produced from the grapes treated at the 2x rate ranged between 0.01 and 0.20 mg/kg.

Table 9. Residues of procymidone and 3,5-dichloroaniline (DCA) in fresh wine prepared from grapes treated wtih Sumilex 50WP or 50L.

Country, Location	Rate kg ai/ha	Residues (mg/kg) at days after last application					
		Commodity	Compound	5	15	21	28
FRA, Tours	0.79	grape wine	parent parent DCA	2.4 0.32 0.07	2.26 0.31 0.07	2.46 0.34 0.07	1.14 0.30 0.06
	1.57	grape wine	parent parent DCA	7.42 1.01 0.20	5.53 0.98 0.15		
FRA, Avignon	0.75	grape wine	parent parent DCA	0.90 0.19 0.05	1.45 0.24 0.03	0.87 0.22 0.03	1.16 0.20 0.03
	1.5	grape wine	parent parent DCA	2.99 0.54 0.09	3.39 0.48 0.09		
SPA, Toledo	0.25	grape wine	parent parent DCA	1.22 0.28 0.03	1.07 0.19 0.04	0.42 0.15 0.04	0.67 0.20 0.04
	0.48	grape wine	parent parent DCA	4.85 * *	2.28 0.59 0.10		
SPA, Cordoba	0.24	grape wine	parent parent DCA	0.33 0.04 <0.01	0.34 0.03 <0.01	0.35 0.03 <0.01	0.20 0.01 <0.01
	0.50	grape wine	parent parent DCA	0.81 0.19 0.02	1.16 0.13 0.01		
ITA, Asti	0.74	grape wine	parent parent DCA	2.08 0.36 0.06	1.72 0.41 0.03	2.24 0.38 0.07	2.37 0.39 0.08
	1.42	grape wine	parent parent DCA	4.52 0.81 0.16	5.15 0.78 0.15		
ITA, Bologna	0.74	grape wine	parent parent DCA	2.26 0.25 0.05	1.89 0.14 0.03	2.68 0.11 0.02	1.90 0.12 0.02
	1.49	grape wine	parent parent DCA	5.32 0.48 0.11	3.74 0.48 0.08		
HUN, Balaton	0.75	grape wine	parent parent DCA	2.11 0.43 0.04	1.76 0.40 0.03	2.70 0.35 0.03	1.74 0.21 0.02
	1.52	grape wine	parent parent DCA	7.48 1.35 0.10	4.65 0.74 0.06		

* Samples were not received

Figure 2. The basic process for making red wine

MATURE GRAPES

Crushing

Alcoholic fermentation

Pressing (pomace removed)

Malolactic fermentation

Clarification

Filtration

WINE

Bottling

Sunflower seeds treated one or three times at rates of 0.5 kg ai/ha were processed with small-scale laboratory equipment. However, the processing technology very closely resembled the industrial process. The scheme of processing, the yield and the loss of oil at various steps are shown in Figure 3. The residues in the seed and crude and refined oil are summarized in Table 10 (Ambrus, 1992c). The results indicate concentration factors ranging between about 2 and 3 from seed to crude oil, and about 1 and 2 from seed to refined oil.

Figure 3. Production of oil from sunflower seed.

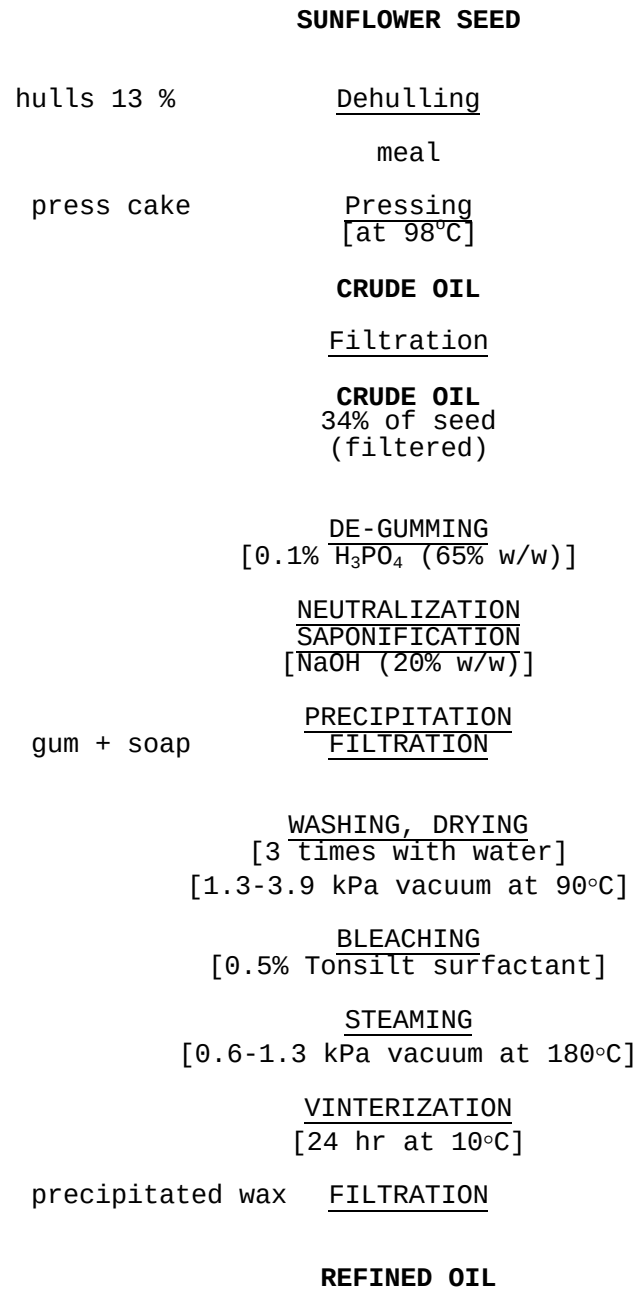


Table 10. Residues (mg/kg) of procymidone in sunflower seed and oil.

Seed	Crude oil	Refined oil
0.12	0.23	0.13
0.11	0.34	0.14
0.04	0.09	0.08
0.04	0.10	0.08

Stability of residues in stored analytical samples

Fresh grapes were fortified at levels of 0.05, 0.50 and 5.0 mg/kg, and the samples were stored at about -20°C. Over a period of 12 months, no loss of procymidone was observed at any level. Procymidone residues have been demonstrated to be stable in wine at -20°C for at least one month, in an on-going investigation. The interim results show that at the level of 0.5 mg/kg, DCA recovery was approximately 75% after storage for 30 days (Roberts *et al.*, 1992).

Strawberry samples were fortified at 0.05 and 0.5 mg/kg and stored at about -26°C. Residues determined after 6 months and one year did not show any significant changes (Halasz-Laky, 1992).

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

In 1987 and 1988, 37 samples of grape juice, 144 samples of young vines and 9 samples of cognac were examined in The Netherlands. Residues above the limit of determination (0.02 mg/kg) were found in 2 samples of grape juice (both 0.03 mg/kg), and in 41 samples of French vines, ranging from 0.02 to 0.32 mg/kg. No residues of procymidone were detected in vines from Spain, Italy or Germany, or in the French cognac (Netherlands, 1993).

In Finland about 3000 consignments of imported fruits and vegetables were analyzed during 1990-1991 (Finland, 1993). Table 11 shows the results.

METHODS OF RESIDUE ANALYSIS

Procymidone residues reported in this monograph were determined by similar methods to those used for the earlier trials. Samples with a high water content were extracted with either acetone or methanol and extraction was followed by partition into dichloromethane, clean-up on Silica gel, Florisil or alumina, if necessary, and GLC with an ECD or nitrogen/phosphorus thermionic detector. Capillary and packed columns were equally suitable. The limit of determination ranged from 0.01 to 0.02 mg/kg with recoveries between 70 and 120%.

Analytical methods suitable for the determination of procymidone residues in various plant commodities have been reviewed recently (Ambrus *et al.*, 1991).

For the determination of DCA in wine a sample aliquot was transferred to a Merck Extelut 20 solid-phase extraction column, rinsed in with methanol-water and eluted with hexane. The hexane was evaporated, the residue was taken up in methanol and determined directly on a LiChrosorb RP-18 reversed-phase HPLC column, eluting with acetonitrile-water (1:1 v/v) and detecting by absorption at 250 nm (Roberts *et al.*, 1992).

Table 11. Residues of procymidone detected in imported commodities in Finland.

Commodity	Number of		Highest residue, mg/kg
	Consignments	Positives.	
Broccoli	18	5	0.42
Celery	82	6	0.8
Chinese cabbage	44	3	0.04
Cucumber	196	93	0.43
Dill	144	11	6.2
Lettuce	39	3	0.11
Lollo Rosso	28	14	6.2
Nectarine	52	3	0.23
Plum	89	3	0.13
Parsley	93	9	2.3
Peach	53	4	0.23
Pear	210	136	1.6
Pepper, Sweet	331	87	0.32
Squash, Summer	28	7	0.16
Strawberry	32	10	0.75
Tomato	213	32	0.92

NATIONAL MAXIMUM RESIDUE LIMITS

National MRLs reported to the Meeting are shown in the Tables below.

Country codes: ARG-Argentina, AUL-Australia, AUS-Austria, BEL-Belgium, FRA-France, GER-Germany, HUN-Hungary, ITA-Italy, JPN-Japan, LUX-Luxembourg, NET-Netherlands, NZE-New Zealand, PER-Peru, POR-Portugal, ROK-Republic of Korea, SAF-South Africa,SPA-Spain, SWI-Switzerland, URU-Uruguay, VEN-Venezuela.

Commodity/Country	MRL, mg/kg									
	AUL	AUS	ARG	BEL	FRA	GER	HUN	ITA	LUX	NET
apricot					2					
beans										1
beans, dwarf, fresh dry		2				1 0.1				
beans, faba	2									
beans, green	2									
beans, haricot				0.2						
beans, navy	2									
beans, runner										1

Commodity/Country	MRL, mg/kg									
carrot							3			
cherry, sour							3			
chicory					5					
cucumbers										1
egg plant										1
endive					5					1
garlic	5				2					
gherkins										1
grapes grapes, wine		2	1.5		5	5 8	3	1.5	5	
kiwifruit								1.5		
lettuce	2	2	1	0.02	5					1
melon					2					1
okra										1
onions	0.2	2	1							0.5
parsley							3			
patisson										1
peaches					2			1.5		
pear								1.5		
peas, fresh peas, dry						1 0.1				
pepper		2					1	1.5		
pepper, green, sweet										1
plum					2					
prune, dried					2					
rape seed					1.5	1				
rape seed oil					5					
raspberries							3			
shallot										0.2
squash, summer (courgettes)										1
stone fruits	10									
strawberries		10		5	2	10	3	1.5		3
sunflower							3			
tomatoes	2				2		1	1.5		1
watermelon										1

Commodity/Country	NEZ	PER	POR	ROK	SAF	SPA	SWI	URU	VEN
beans	2				1.0				
blackberries							1.5		
cacao									3
celery									2
citrus		3							
cucumbers			2	2					

Commodity/Country	NEZ	PER	POR	ROK	SAF	SPA	SWI	URU	VEN
garlic								5	2
grapes	5		5	5	5	5	5	5	3
grape juice							2		
lettuce				5					2
nectarine								5	
onions	0.1					0.2		5	2
peaches					10			5	3
peanut					0.5				2
peas					0.1				
pepper			5						
pepper, red				5					
pome fruits							0.05		
potato					0.2				
raspberries							1.5		
soya bean									2
stone fruits	3						0.05		
strawberries	0.5	3	10	10		5	1.5	5	2
tomatoes	1		5	5	3				2
vegetables						2			
watermelon				1					

APPRAISAL

Procymidone was reviewed by the Joint Meeting in 1981, 1989 and 1990. The 1992 CCPR retained all MRLs at step 7B in view of the need to ensure that the residue data which were reviewed in 1981 reflected current GAP.

Residue data for common beans, cucumbers, grapes, lettuce, bulb onions and tomatoes were required together with information on current GAP.

In response to the request of the CCPR extensive information was provided by the manufacturer and some member countries on use patterns, together with some residue data from supervised field trials and monitoring.

In order to estimate maximum residue levels, the residues resulting from supervised trials published in the previous evaluations which accorded with current use patterns (GAP) were also taken into consideration.

Apples. GAP was reported from Japan and the Lebanon. Two trials reported from Japan in 1981 do not reflect GAP in either country. The previous recommendation (5 mg/kg) is withdrawn.

Cherries. GAP was reported from 6 countries. The maximum dosage rate is 0.75 kg ai/ha with a PHI of 14-28 days. A single trial in Hungary corresponds to current GAP. However the initial residues (0.8 mg/kg) are much lower than in the Australian trials (2.1-6.2 mg/kg) reflecting GAP which were reported in 1990. The previous recommendation (5 mg/kg) is replaced by 10 mg/kg.

Beans. GAP was reported from 6 countries with PHIs ranging between 2 and 21 days. Residues, reported in 1981, 1989 and 1993, deriving from corresponding national GAP (7-14 days, maximum 0.75 kg ai/ha) range from 0.1 to 0.8 mg/kg. The current GAP leads to lower residues, consequently the recommended limit is 1 mg/kg.

Cucumbers and gherkins. GAP was reported from 12 countries with PHIs ranging between 1 and 15 days. Trials reported in 1981 from Japan which reflect current GAP showed residues of 0.33-1.2 mg/kg at day 1. The previous recommendation (2 mg/kg) is maintained.

Currants. GAP was reported from one country where procymidone is used for stock treatment. Two trials reported in 1981 involved foliar applications to black currants. The residue limit established previously is not supported by current GAP, so the recommendation (10 mg/kg) is withdrawn.

Egg plants. GAP was reported from 6 countries with PHIs of 1-3 days. Residues from a trial in Poland ranged from 0.6 to 0.93 mg/kg at days 1 and 3. A trial reported from France in 1981 showed a residue level of 1.5 mg/kg at 14 days. The data are insufficient to estimate a maximum residue level, so the recommendation (2 mg/kg) is withdrawn.

Grapes. GAP was reported from 27 countries with PHIs of 1-28 days, 1-4 applications at 0.25-1.0 kg ai/ha. An extensive trial programme was conducted in seven wine-growing regions of Europe. Dosage and pre-harvest intervals were selected according to the relevant national GAP which cover the world-wide uses. Residues deriving from recommended uses ranged from 0.34 mg/kg to 4.6 mg/kg. They are in the same range as those obtained in earlier trials. The present limit (5 mg/kg) is reaffirmed and it should no longer be temporary.

Kiwifruit. GAP was reported from Italy. The trial conditions reported from New Zealand in 1981 reflect the current Italian GAP, but there was no information on the comparability of climatic conditions and cultural practices. Consequently the previous recommendation (7 mg/kg) is withdrawn.

Lettuce. GAP was reported from 16 countries with PHIs of 2-35 days. The number of applications is from 1 to 10, and the rates are between 0.28 and 2 kg ai/ha. Residues reported from France (indoor and outdoor) and Spain were in the range of 0.07 to 3.4 mg/kg 21-22 days after the last application. The residues derived from glass-house applications were about 2 to 4 times those from trials conducted outdoors. The previous recommendation (5 mg/kg) is reaffirmed.

Melons. GAP was reported from 4 countries. Trials reported in 1981 reflect the current use patterns, but no residues were reported in the whole commodity. The data base is considered inadequate for estimating a maximum residue level. The previous recommendation (1 mg/kg) is withdrawn.

Onions. GAP was reported from 14 countries with PHIs of 1-28 days. Trial conditions reported in 1981 are within the current recommended uses and lead to residues in the range of 0.01-0.14 mg/kg which support the present limit (0.2 mg/kg).

Peaches and nectarines. GAP for foliar and post-harvest applications was reported from 15 countries with PHIs of 1-14 days and maximum rates of 0.37-1.0 kg ai/ha applied 2-5 times. The trial conditions reported from Australia and New Zealand in 1981 and 1990 are in line with present use recommendations. The previous recommendation (10 mg/kg) is reaffirmed.

Peppers. GAP was reported from 11 countries on sweet, green and chilli peppers with PHIs of 1-7 days. The trials reported from Japan in 1981 reflect the current use and lead to residues up to 3.8 mg/kg one day after the last application. The present limit (5 mg/kg) is reaffirmed.

Potatoes. GAP was reported from 5 countries with PHIs of 3-35 days. In two Japanese trials, carried out in 1977 but reported in 1981 and again in 1993, the residues were 0.02, 0.03, 0.05 and 0.08 mg/kg in potatoes 19-28 days after the last application. The data base was considered inadequate to estimate a maximum residue level. The previous recommendation (0.1 mg/kg) is withdrawn.

Raspberries. GAP was reported from 3 countries with PHIs of 7-14 days. In German trials reported in 1989 residues ranged from 0.59 to 6.9 mg/kg at 14 days after applications according to current GAP. Trials in France in 1989 and in Hungary and Poland in 1992 resulted in lower residues, but the combined data support the present limit (10 mg/kg).

Rice. GAP was reported from Thailand where the application is repeated every 7-10 days. Results reported in 1981 were from samples taken 19-22 days after the last application with about 2.5 times the rate registered in Thailand. The trial conditions cannot be related to GAP, so the recommendations (rice, husked: 3 mg/kg and rice, polished: 1 mg/kg) are withdrawn.

Strawberries. GAP was reported from 27 countries with PHIs of 2-21 days and application rates of 0.23-1.0 kg ai/ha. Residues from field trials in France, Germany, The Netherlands and Poland in 1981 ranged from 0.4 to 5.1 mg/kg. Following glasshouse application in Japan, the residues were between 0.9 and 8.0 mg/kg. Residues reported from Spain in 1993 were in the range of 1.3-4.24 mg/kg. The previous recommendation (10 mg/kg) is maintained.

Sunflower seed. GAP was reported from 6 countries with PHIs of 14-42 days. Residues deriving from treatments with recommended and double rates were in the range of 0.02 to 0.12 mg/kg 14-28 days after the last application. The Meeting considered the results of a single trial leading to high residues atypical and estimated a maximum residue level of 0.2 mg/kg which replaces the previous recommendation (2 mg/kg).

Tomatoes. GAP was reported from 25 countries with PHIs of 1-21 days and maximum rates of 0.5-1.8 kg ai/ha. Residues reported from France, Japan and New Zealand in 1981, from New Zealand in 1990 and from Italy in 1993 ranged from 0.1 to 2.1 mg/kg with a residue of 2.5 mg/kg at day 1 from a glasshouse trial in Japan. The previous recommendation (5 mg/kg) is maintained.

The fate of residues in wine processing was extensively studied. Grapes were harvested from 42 separately treated test plots and fermented into wine. The vinification procedure used for grapes from a given site was chosen to match the procedure used locally in the country or region of origin. The use of different procedures, each typical of the locale in which the grapes originated, allows a realistic estimation of residues expected in commercially-produced wine.

In addition to procymidone, 3,5-dichloroaniline (DCA), which may be formed during or just after vinification, was also determined in the wine.

When grapes were treated according to GAP, the wine contained procymidone residues between 0.04 and 0.59 mg/kg. The level of DCA ranged from <0.01 to 0.07 mg/kg in the same samples.

The results indicate that procymidone residues remaining in or on grapes after treatment show no tendency to concentrate in the wine. The average wine/grape ratio for procymidone ranged between 0.07 and 0.27, with an overall average of 0.16. DCA amounted to a maximum of 20% of the procymidone concentration in wine.

Sunflower seeds, containing residues of 0.04-0.12 mg/kg, were processed to oil. The

crude and refined oil samples contained residues of 0.1-0.34 mg/kg and 0.08-0.14 mg/kg respectively. The concentration factors were between 2 and 3 for seed to crude oil, and between 1 and 2 for seed to refined oil.

A survey of procymidone residues in fresh fruits and vegetables imported by Finland gave positive results in 16 commodities. The maximum values were below the recommended limits in all cases. The commodities in which the positive results exceeded 10% were the following: broccoli 28%, cucumber 47%, pear 65%, sweet pepper 26%, strawberry 32%, tomato 15%. It is to be noted that maximum residue levels have not been estimated by previous Meetings for broccoli or pears. Furthermore, information on current GAP indicates that the compound is registered for pears only in Italy and not at all for broccoli.

RECOMMENDATIONS

On the basis of data on residues from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits.

Definition of the residue: procymidone

Commodity		Recommended MRL (mg/kg)		PHI on which based, days
CCN	Name	New	Previous	
FP 0226	Apple	W	5	
FS 0013	Cherries	10	5	1
VP 0526	Common bean (pods and/or immature seeds)	1	2	7-14
VC 0424	Cucumber	2	2	1-3
FB 0021	Currants, Black, Red, White	W	10	
VO 0440	Egg plant	W	2	
VC 0425	Gherkin	2	2	1-3
FB 0269	Grapes	5	5 T	5-21
FI 0341	Kiwifruit	W	7	
VL 0482	Lettuce, Head	5	5	21[1]
VC 0046	Melons, except Watermelon	W	1	
FS 0245	Nectarine	10	10	1
VA 0385	Onion, Bulb	0.2	0.2	1-14
FS 0247	Peach	10	10	1
VO 0051	Peppers	5	5	1
VR 0589	Potato	W	0.1	
FB 0272	Raspberries, Red, Black	10	10	14

Commodity		Recommended MRL (mg/kg)		PHI on which based, days
CCN	Name	New	Previous	
CM 0649	Rice, husked	W	3	
CM 1205	Rice, polished	W	1	
FB 0275	Strawberry	10	10	1-3
SO 0702	Sunflower seed	0.2	2	14-28
OR 0702	Sunflower seed-oil, edible	0.5		
VO 0448	Tomato	5	5	1-3

[1] In glasshouse
W: the limit is withdrawn

REFERENCES

1. Ambrus, A., Buys, M., Miyamoto, J.,Otto, S. and Smart, N.A., 1991. Analysis of residues of dicarboximide fungicides in food. Pure & Appl. Chem., Vol 63, No. 5, pp. 747-762.

2. Ambrus, A., 1992. Determination of procymidone residues in raspberry. Sumitomo Chemical Co. Ltd. Osaka , Unpublished Report No.: BR-21-0277

3. Ambrus, A., 1992a. Determination of procymidone residues in sour cherry. Sumitomo Chemical Co. Ltd. Osaka, Unpublished Report No.: BR-21-0279

4. Ambrus, A., 1992b. Determination of procymidone residues in sunflower seed. Sumitomo Chemical Co. Ltd. Osaka, Unpublished Report No.: BR-21-0281

5. Ambrus, A., 1992c. Determination of procymidone residues in crude and refined sunflower seed oil. Sumitomo Chemical Co. Osaka, Unpublished Report No.: BR-21-0282

6. Finland, 1993. Information on compounds on the CCPR priority list.

7. Halasz-Laky, V., 1992a. Final report on determination of procymidone storage stability in strawberry samples. Sumitomo Chemical Co. Osaka, Unpublished Report No.: BR-21-02838.

8. Halasz-Laky, V., 1992b. Final report on determination of procymidone residues in raspeberry samples. Sumitomo Chemical Co. Osaka, Unpublished Report No.: BR-21-0284

9. Halasz-Laky, V., 1992c. Determination of procymidone residue in egg plant samples. Sumitomo Chemical Co. Osaka, Unpublished Report No.: BR-31-0294

10. Hungary, 1990. Information on compounds on the CCPR priority list.

11. Macdonald, I. A., Gillis, N. A. and Howie D., 1992a. Determination of residual concentrations in tomatoes from field trials in France. Sumitomo Chemical Co. Osaka, Unpublished Report No.: BR-21-0260.

12. Macdonald, I. A., Gillis, N. A. and Howie, D. 1992b. Determination of residual concentrations in tomatoes from field trials in Italy. Sumitomo Chemical Co. Osaka, Unpublished Report No.: BR-21-0261

13. Macdonald, I. A., Gillis, N. A. and Howie, D. 1992c. Determination of residual concentrations in cucumbers from field trials in Spain. Sumitomo

Chemical Co. Osaka, Unpublished Report No.: BR-21-0264

14. Macdonald, I. A., Gillis, N. A. and Howie, D. 1992d. Determination residual procymidone concentrations in haricot beans from field trials in France. Sumitomo Chemical Co. Osaka, Unpublished Report No.: BR-21-0265

15. Macdonald, I. A., Gillis, N. A. and Howie, D. 1992e. Determination of residual concentrations in lettuce from field trials in France. Sumitomo Chemical Co. Osaka, Unpublished Report No.: BR-21-0266

16. Macdonald, I. A., Gillis, N. A. and Howie, D. 1992f. Determination of residual concentrations in shallots from field trials in France. Sumitomo Chemical Co. Osaka, Unpublished Report No.: BR-21-0268

17. Netherlands, 1993. Information on compounds on the CCPR priorites list.

18. New Zealand, 1993. Information on compounds on the CCPR priority list.

19. Roberts, N. L., Macdonald, I. A. and Gillis, N. A. 1992. Procymidone - Magnitude of residues in grapes and processed commodities of grapes - 1991 European trials. Sumitomo Chemical Co. Osaka, Japan, Unpublished Report No.: BR-21-0258

20. Spain, 1992. Information on compounds on the CCPR priority list.

21. Spain, 1993. Information on compounds on the CCPR priority list.

22. Sumitomo Chemical Co. Ltd. 1992. Residue report of procymidone on potatoes. Unpublished, Report No.: BR-70-0256

23. Sumitomo Chemical Co. Ltd. 1992. Residue report of procymidone on potatoes. Unpublished, Report No.: BR-70-0257

PROPINEB

EXPLANATION

Propineb was evaluated in 1977, 1984 and 1985. The temporary ADI was withdrawn by the 1985 JMPR, but the CCPR maintained the guideline levels for propylenethiourea.

USE PATTERN

Propineb is a protectant foliar-applied fungicide with long residual activity and belongs to the dithiocarbamate group of compounds. It is used as a protective treatment on several crops for the control of various fungi, especially Oomycetes, Ascomycetes, Basidiomycetes and Fungi imperfecti. Propineb controls blight on potatoes and tomatoes, downy mildew on hops and vines, apple scab, blue mould on tobacco and Sigatoka disease of bananas. It can also be used on gooseberries, black currants, celery and cereals.

Propineb is applied as a WG or WP formulation mainly as a spray. It is also applied, especially in southern Europe, in combination with oxadixyl, carbendazim, copper oxychloride, triadimefon or cymoxanil.

Table 1 shows the registered uses of propineb reported to the Meeting. However, the principal manufacturer intends to recommend the use of propineb only on the following crops: grapes, tomatoes, potatoes, pome fruit, onions, melons and bell peppers. In this monograph residue data are reviewed only for these crops.

Table 1. Registered or approved uses of propineb

Crop	Country	Application				PHI, days
		Form.	No.	g ai/hl	kg ai/ha	
Grapes	Austria	70 WP	1-4	0.21	up to 2.10	7
	France	70 WP	up to 2		up to 2.80	
	Germany	70 WG 70 WG	up to 2 up to 6	0.14	up to 1.12 up to 2.52	56 56
	Greece	70 WP 65 WP	3-4 3-4	0.16	1.40-1.75 1.30-1.63	7 7
	Italy	70 WP 65 WP	2-5 2-5	0.14 0.13	1.05-1.40 0.98-1.30	28 28
	Portugal	70 WP	up to 7		1.45-2.10	7
	Spain	70 WP	up to 3	0.14-0.21	0.7-1.05	15,28
	Thailand	70 WP	1-2	0.1	1.64-1.97	21
	Turkey	70 WP	2-3		up to 1.40	21
Melons	Australia	70 WP	up to 7		up to 1.40	7
	Guatemala	70 WP	2-3		1.75-2.10	7
	Korea	70 WP	up to 3		up to 2.10	7
	Japan	70 WP	up to 3	0.17	2.34-3.50	7
	Turkey	70 WP	1-2		up to 0.84	7
Onions	Australia	70 WP	up to 4	0.14	up to 2.80	14
	Israel	70 WP	up to 2		up to 1.75	3

Crop	Country	Application				PHI, days
		Form.	No.	g ai/hl	kg ai/ha	
	Japan	70 WP	up to 5	0.17	1.75-2.63	7
	Spain	70 WP	up to 2	0.14-0.21	1.12-1.68	15
	Venezuela	70 WP	4-8		1.40-2.10	7
Pome fruit	Germany	70 WG	up to 12		up to 1.58	28
	Italy	70 WP 65 WP	3-5 up to 2		1.58-2.10 1.46-1.95	28 28
	Japan	70 WP	up to 4		up to 8.40	14
	Korea	70 WP	up to 3		up to 5.60	10
	Portugal	70 WP	up to 2		up to 2.10	7
	Taiwan	70 WP	7-10		up to 3.50	10
	Turkey	70 WP	4-5		up to 4.20	14
Potatoes	Australia	70 WP	up to 7	0.14	up to 2.10	7
	Colombia	70 WP	up to 6		1.05-1.75	3
	Germany	70 WG	1-6		1.05-1.26	7
	Greece	70 WP 65 WP	2-3 2-3	0.17 0.16	1.40-1.75 1.30-1.63	7 7
	Guatemala	70 WP	4-8		1.75-2.10	7
	Indonesia	70 WP	up to 10		1.05-1.75	14
	Peru	70 WP	3-4		1.05-1.75	7
	Portugal	70 WP	up to 8	0.17	up to 2.10	7
	Spain	70 WP	2-3	0.14-0.21	1.40-2.10	15
	Venezuela	70 WP	4 -6		1.40-2.10	7
Tomatoes	Australia	70 WP	up to 7		up to 1.40	3
	Germany	70 WP	up to 4		0,84-1.68	7
	Greece	70 WP 65 WP	3-4 3-4		1.40-1.75 1.30-1.63	3 3
	Indonesia	70 WP	up to 10		1.05-1.75	14
	Italy	70 WP 65 WP	1-2 1-2	0.14 0.13	up to 1.12 up to 1.04	28 28
	Morocco	70 WP	8-14		1.40-2.10	7
	Spain	70 WP	2-3	0.14	2.8 -4.35	15

RESIDUES RESULTING FROM SUPERVISED TRIALS

A number of trials were carried out with several crops in various countries. The residues determined were propineb, measured and calculated as CS_2, as well as the major metabolite propylenethiourea (PTU) and in some cases propylenediamine (PDA). The results are discussed in relation to the current registered uses.

The following country codes are used in the Tables: ARG-Argentina, AUL-Australia, AUS-Austria, BEL-Belgium, CHI-Chile, CYP-Cyprus, DEN-Denmark, FIN-Finland, FRA-France, GER-Germany, HUN-Hungary, ISR-Israel, ITA-Italy, JPN-Japan, LUX-Luxembourg, MAL-Malaysia, MOZ-Mozambique, NET-Netherlands, NZE-New Zealand, POR-Portugal, SAF-South Africa, SPA-Spain, SWE-Sweden, SWI-Switzerland, TAW-Taiwan, TUR-Turkey, YUG-Yugoslavia.

Underlined residues are from treatments according to GAP.

Pome fruits. A total of 13 supervised trials were conducted in Germany with Antracol 70 WG/WP (10 on apples and 3 on pears), where up to 12 sprays

were applied at rates from 0.45 to 2.1 kg ai/ha. The dosage rates and pre-harvest intervals accord with those in effect in several countries. The results are summarized in Tables 2 and 3.

Residues of propineb in fruit 14-21 days after the last treatment ranged from <0.05 mg/kg to 0.96 mg/kg. For the main metabolite, PTU, the results were in the range <0.02 to 0.08 mg/kg.

Table 2. Residues of propineb in apples from supervised trials in Germany with Antracol 70 WP.

Sample, Year	Application			PHI, days	Residue, mg/kg		Ref.
	No.	kg ai/ha	kg ai/hl		CS_2	PTU	
Fruit, 1982	10	2.1	0.14	21	0.96	0.03	8009-82
Juice					<0.05	0.03	
Puree					<0.05	0.02	
Fruit, 1982				28	0.8	0.03	
Fruit, 1982	10	2.1	0.56	21	<0.05	<0.01	8010-82
Juice					<0.05	<0.05	
Puree					<0.05	<0.05	
Fruit				28	<0.05	<0.01	
Fruit, 1987	12	1.6	0.105	0 5 7 14 21	1.5 1.4 0.85 0.31 0.17	 0.02 <0.02	8008-87
Fruit, 1987	12	1.6	0.105	0 5 7 14 21	2.3 1.0 1.0 0.34 0.48	 <0.02 <0.02	8058-87
Fruit, 1988	12	1.6	0.105	0 5 7 14 21	0.46 0.26 0.18 <0.05 <0.05	<0.02 <0.02 <0.02 <0.02 <0.02	0023-88
Fruit, 1988	12	1.6	0.105	0 5 7 14 21	0.70 0.21 0.21 0.09 0.09	<0.02 <0.02 <0.02 <0.02 <0.02	0024-88
Fruit, 1991	12	1.6	0.105	0 3 7 13 21	0.81 0.59 0.68 0.34 0.31	<0.01 <0.01 <0.01 <0.01 <0.01	0038-91
Fruit, 1991	12	1.6	0.105	0 4 7 14 21	1.1 0.90 0.72 0.59 1.0	0.02 0.02 0.02 0.03 0.02	0039-91
Fruit, 1991	12	0.78	0.315	0 4 7 14 21	0.73 0.66 0.44 0.29 0.24	0.01 0.03 0.03 0.01 0.01	0040-91
Fruit, 1991	12	0.94	0.315	0 4 7 14 21	0.41 0.24 0.21 0.17 0.11	<0.01 <0.01 <0.01 <0.01 <0.01	0041-91
Fruit, 1991		1.58	0.14	0 3 7 13 21	0.81 0.59 0.68 0.34 0.31	<0.01 <0.01 <0.01 <0.01 <0.01	RA 2004/91
Fruit, 1991		1.58	0.14	0 4 7 14 21	1.1 0.90 0.72 0.59 1.0	0.02 0.02 0.02 0.03 0.02	RA 2004/91
Fruit, 1991		0.78	0.32	0 4 7	0.73 0.66 0.44	0.01 0.03 0.03	RA 2004/91

Sample, Year	Application			PHI, days	Residue, mg/kg		Ref.
	No.	kg ai/ha	kg ai/hl		CS_2	PTU	
				14 21	0.29 0.24	0.01 0.01	
Fruit, 1991		0.45	0.32	0 4 7 14 21	0.41 0.24 0.21 0.17 0.11	<0.01 <0.01 <0.01 <0.01 <0.01	RA 2004/91

Table 3. Residues of propineb in pears from supervised trials in Germany.

Sample Year	Application			PHI, days	Residue, mg/kg		Ref.
	No.	kg ai/ha	kg ai/hl		CS_2	PTU	
Fruit, 1982	10	2.1	0.14	21	0.52	0.06	8011-82
Puree					<0.05	<0.01	
Fruit				28	0.55	0.05	
Fruit, 1987	12	1.58	0.105	0 5 7 14 21	1.9 1.2 1.3 0.49 0.40	 0.05 0.05	8010-87
Fruit, 1987	12	1.58	0.105	0 5 7 14 21	2.0 1.9 1.1 0.82 0.82	 0.07 0.08	8060-87

Grapes. Many trials have been conducted in Germany and Turkey since 1981 with Antracol 70 WG and WP using a range of grape varieties and use patterns. At pre-harvest intervals of 49 to 69 days, residues of propineb and PTU were between <0.05 and 1.2 mg/kg and between <0.01 and 0.08 mg/kg respectively, except in one trial where 2.1 mg/kg propineb and 0.15 mg/kg PTU were measured (Table 4). With different application schedules, the results for propineb are more or less in the same range, although there is a slight tendency for the residue to increase with increasing dosage or number of applications. The variety of grape did not influence the residue levels.

Table 4. Residues of propineb in grapes, must and wine from supervised trials in Germany and Turkey.

Crop, product Year	Application			PHI, days	Residues (mg/kg) at days after application		Ref.
	No.	kg ai/ha	kg ai/hl		CS_2	PTU	
Germany							
must, 1981 grape wine	6	2x1.4 4x2.8	0.14	69	0.07 0.13 <0.05	0.01 0.01 0.03	8000-81
must, 1981 grape wine	6	2x1.4 4x2.8	0.14	69	<0.05 <0.05 <0.05	<0.01 <0.01 0.02	8001-81
must, 1981 grape wine	6	2x1.4 4x2.8	0.14	69	0.41 0.75 0.05	0.07 0.05 0.10	8002-81
must, 1981 grape wine	8	2x1.4 6x2.8	0.14	40	0.10 0.49 <0.05	0.03 0.03 0.05	8003-81
must, 1981 grape wine	8	2x1.4 6x2.8	0.14	40	<0.05 0.19 <0.05	<0.01 0.02 0.01	8004-81
must, 1982 grape wine	6	2x1.4 4x2.8	0.28	41	0.1 <0.05 <0.05	0.03 <0.01 0.02	8025-82
must, 1982 grape wine	6	2x1.4 4x2.8	0.56	43	<0.05 <0.05 <0.05	0.03 0.01 0.04	8026-82
must, 1982 grape wine	6	2x1.4 4x2.8	0.56	41	0.1 0.2 0.1	0.2 0.05 0.2	8027-82

Crop, product Year	Application			PHI, days	Residues (mg/kg) at days after application		Ref.
	No.	kg ai/ha	kg ai/hl		CS_2	PTU	
must, 1982 grape wine	6	2x1.4 4x2.8	0.56	35	0.1 0.5 0.1	0.2 0.03 0.1	8028-82
must, 1982 grape wine	6	2x1.4 4x2.8	0.28	42	<0.05 0.07 <0.05	0.1 0.01 0.06	8029-82
must, 1982 grape wine	6	2x1.4 4x2.8	0.28	42	0.08 0.07 <0.05	0.04 0.02 0.05	8030-82
must, 1982 grape wine	5	2x1.4 3x2.8	0.28	55	<0.05 <0.05 <0.05	<0.01 <0.01 <0.01	8034-82
must, 1982 grape wine	5	2x1.4 3x2.8	0.56	58	<0.05 <0.05 <0.05	<0.01 0.01 0.02	8035-82
must, 1982 grape wine	5	2x1.4 3x2.8	0.56	56	<0.05 <0.05 <0.05	0.05 0.03 0.05	8036-82
must, 1982 grape wine	5	2x1.4 3x2.8	0.56	49	0.05 0.2 0.05	0.02 0.08 0.07	8037-82
must, 1982 grape wine	5	2x1.4 3x2.8	0.28	56	<0.05 <0.05 <0.05	0.08 <0.01 0.03	8038-82
must, 1982 grape wine	5	2x1.4 3x2.8	0.28	56	<0.05 <0.05 <0.05	0.05 0.02 0.05	8039-82
must, cold, 1984 must,heated grape wine, cold wine,heated	3	1.4;1.68; 1.96	0.14	42 49 56 42 28 42 49 56 42 49 56 42	1.2 0.24 1.5 0.57 1.1 1.6 0.74 0.52 <0.05 <0.05 <0.05 <0.05	<0.02 <0.02 <0.02 <0.02 <0.02 <0.02 <0.02 <0.02 0.10 0.13 0.07 0.14	8000-84
must, 1984 grape wine	4	1.4;1.68 2x1.96	0.14	42 49 56 0 28 42 49 56 42 49 56	0.45 0.75 1.3 4.8 1.1 0.30 1.7 0.37 <0.05 <0.05 <0.05	<0.02 <0.02 <0.02 0.03 <0.02 <0.02 <0.02 <0.02 <0.02 <0.02 <0.02	8001-84
must, cold, 1984 must,heated grape wine, cold wine, heated	5	1.4;1.68; 2x1.96; 2.24	0.14	42 49 56 56 0 28 42 49 56 42 49 56 56	1.5 1.8 2.5 0.21 3.7 2.1 0.22 1.5 1.1 <0.05 <0.05 <0.05 <0.05	<0.02 <0.02 <0.02 <0.02 0.08 <0.02 <0.02 <0.02 <0.02 <0.02 0.17 0.24 0.14	8002-84
must, cold, 1984 must, heated grape wine, cold wine, heated	3	1.4;1.68; 1.96	0.14	42 42 49 56 28 42 49 56 42 42 49 56	<0.05 0.89 <0.05 0.12 0.74 1.3 0.50 0.68 <0.05 <0.05 <0.05 <0.05	<0.02 <0.02 <0.02 <0.02 <0.02 <0.02 <0.02 <0.02 0.10 0.10 0.04 0.05	8003-84
must, heated, 1984 grape	4	1.4;1.68; 2x1.96	0.14	42 49 56 28 42 49 56 42	0.34 0.73 0.80 3.2 1.4 0.61 0.51 <0.05	<0.02 <0.02 <0.02 <0.02 <0.02 <0.02 <0.02 0.17	8004-84

Crop, product Year	Application			PHI, days	Residues (mg/kg) at days after application		Ref.
	No.	kg ai/ha	kg ai/hl		CS_2	PTU	
wine, heated				49 56	<0.05 <0.05	0.09 0.09	
must, cold, 1984 must, heated grape wine, cold wine, heated	5	1.4;1.68; 2x1.96;2.24	0.14	49 56 42 49 56 0 28 42 49 56 49 56 42 49 56	0.95 0.46 0.70 0.83 0.40 2.4 <0.05 0.35 1.5 0.99 <0.05 <0.05 <0.05 <0.05 <0.05	<0.02 <0.02 <0.02 <0.02 <0.02 0.06 <0.02 <0.02 <0.02 <0.02 0.08 <0.02 0.18 0.09 0.04	8005-84
mash, 1986 must grape wine	6	2x1.13;2x 1.68; 2.1; 2.52	0.42	56 56 0 56 56	0.17 <0.05 1.9 0.32 <0.05	<0.02 <0.02 0.02 <0.02 0.03	8000-86 G
must, 1986 grape wine	7	0,56;1.05; 1.68;1.96; 2.1;2.38; 2.52	0.42	56 0 56 56	0.11 2.9 0.27 <0.05	0.02 0.01 0.04	8000-86 N
must, 1986 grape wine	7	0.56;1.05; 1.68;1.96; 2.1;2.38; 2.52	0.42	56 0 56 56	<0.05 4.8 1.1 <0.05	0.03 0.11 0.02 0.04	8001-86 N
must, 1986 grape wine	6	2x0.84; 4x2.52	0.42	56 0 56 56	<0.05 4.6 1.2 <0.05	0.02 0.11 0.02 0.04	8001-86 Z
mash, 1986 must grape wine	6	2x1.3; 2x1.68; 2.1;2.52	0.42	56 56 0 56 56	0.18 <0.05 3.1 0.35 <0.05	<0.02 <0.02 0.06 <0.02 0.03	8002-86 G
must, 1986 grape wine	7	0.56;1.68; 1.68;1.96; 2.1;2.38; 2.52	0.42	56 0 56 56	0.15 3.0 0.28 <0.05	0.02 0.03 0.01 0.04	8002-86 N
must, 1986 grape wine	7	0.56;1.05; 1.68;1.96; 2.1;2.38; 2.52	0.42	56 0 56 56	0.25 4.7 0.78 <0.05	0.03 0.08 0.01 <0.05	8003-86 N
must, 1986 grape wine	6	2x0.84; 4x2.52	0.42	56 0 56 56	0.10 5.9 0.46 <0.05	0.03 0.12 0.02 0.04	8003-86 Z
mash, 1986 must grape wine	6	2x1.13; 2x1.68; 2.1;2.52	0.42	49 49 0 49 49	0.46 0.05 1.7 0.38 <0.05	0.02 <0.02 0.04 <0.02 0.04	8004-86 G
must, 1986 grape wine	7	0.56;1.05; 1.68;1.96; 2.1;2.38; 2.52	0.42	49 0 49 49	0.05 4.5 0.59 <0.05	0.02 0.06 0.01 0.05	8004-86 N
must, 1986 grape wine	7	0.56;1.05; 1.68;1.96; 2.1;2.38; 2.52	0.42	49 0 49 49	<0.05 3.3 0.71 <0.05	0.04 0.07 0.01 0.06	8005-86 N
must, 1986 grape wine	6	2x0.84 4x2.52	0.42	49 0 49 49	0.10 5.7 0.60 <0.05	0.02 0.07 0.01 0.04	8005-86 Z
mash, 1986 must grape wine	6	2x1.13; 2x1.68 2.1;2.52	0.42	49 49 0 49 49	0.24 <0.05 2.4 0.30 <0.05	0.03 <0.02 0.04 <0.02 0.03	8006-86 G
must, 1986 grape wine	7	0.56;1.05; 1.68;1.96; 2.1; 2.38; 2.52	0.42	49 0 49 49	0.10 3.3 0.99 <0.05	0.03 0.07 0.01 0.04	8006-86 N
must, 1986 grape	7	0.56;1.05; 1.68;1.96;	0.42	49 0	<0.05 3.4	0.03 0.03	8007-86 N

Crop, product Year	Application			PHI, days	Residues (mg/kg) at days after application		Ref.
	No.	kg ai/ha	kg ai/hl		CS_2	PTU	
wine		2.1; 2.38; 2.52		49 49	0.83 <0.05	<0.01 0.05	
must, 1986 grape wine	6	2x0.84 4x2.52	0.42	49 0 49 49	0.05 5.6 0.60 <0.05	0.05 0.08 <0.02 0.06	8007-86 Z
mash, 1986 must grape wine	6	2x1.13; 2x1.68; 2.1; 2.52	0.42	56 56 0 56 56	0.13 0.06 2.6 0.30 <0.05	<0.02 <0.02 0.06 <0.02 <0.02	8008-86 G
must, 1986 grape wine	7	0.56;1.05; 1.68;1.96; 2.1; 2.38; 2.52	0.42	56 0 56 56	0.10 3.1 0.69 <0.05	0.20 0.06 0.01 0.28	8008-86 N
must, 1986 grape wine	7	0.56;1.05; 1.68;1.69; 2.1;2.38; 2.52	0.42	56 0 56 56	0.20 3.5 1.1 <0.05	0.50 0.14 0.03 0.67	8009-86 N
must, 1986 grape wine	6	2x0.84; 4x2.52	0.42	56 0 56 56	0.08 6.8 1.0 <0.05	0.25 0.16 0.02 0.26	8009-86 Z
must, 1987 grape wine	6	1.12;2x1.68 3.8;2x3.5	0.14	56 0 28 35 56 56	<0.05 20 1.6 1.3 0.9 <0.05	<0.02 0.6 0.03 0.03	8000-87
must, 1987 grape wine	6	0.84;1.05; 1.68;2x2.1 2.52	0.42	56 0 28 35 56 56	0.05 4.6 1.1 0.38 0.72 <0.05	<0.02 <0.02 0.02 <0.02	8001-87
must, 1987 grape wine	6	2x0.84; 4x2.52	0.42	56 0 28 35 56 56	0.05 7.2 1.5 0.62 0.46 <0.05	0.04 0.07 0.04 0.04	8002-87
must, 1987 grape wine	6	0.7;1.12; 2.1;2.52; 2.8;3.5	0.14	56 0 28 35 56 56	<0.05 4.3 1.2 0.50 0.42 <0.05	<0.02 0.03 <0.02 <0.02	8050-87
must, 1987 grape wine	6	0.84;1.05; 1.68;2x2.1; 2.52	0.42	56 0 28 35 56 56	0.05 4.7 1.0 0.44 0.75 <0.05	<0.02 <0.02 <0.02 <0.02	8051-87
must, 1987 grape wine	6	2x0.84 4x2.52	0.42	56 0 28 35 56 56	0.11 13 2.1 2.0 2.1 <0.05	0.06 0.12 0.15 0.04	8052-87
bunch, 1988 must wine	4	2x0.84 2x1.68	0.42	0 70 70 70	6.9 0.06 <0.05 <0.05	0.27 <0.02 <0.02 <0.02	0406-88
bunch, 1988 must wine	5	2x0.84 3x2.1	0.42	0 70 70 70	0.50 0.66 <0.05 <0.05	<0.02 0.03 0.07 0.10	0408-88
bunch, 1988 must wine	5	2x0.84 3x2.1	0.42	0 70 70 70	5.8 0.53 <0.05 <0.05	0.16 0.04 0.10 0.11	0409-88
Turkey							
grape, 1990	4	1.4	0.14	0 76	2.6 <0.1		0610-90
grape, 1990 raisin	4	1.4	0.14	0 62 62	1.2 0.15 <0.1	<0.01	0611-90

Crop, product Year	Application			PHI, days	Residues (mg/kg) at days after application		Ref.
	No.	kg ai/ha	kg ai/hl		CS_2	PTU	
grape, 1990	4	1.4	0.14	0 48	1.5 0.15		0612-90
grape, 1990 raisin	4	1.4	0.14	0 76 76	3.2 0.22 <0.1	0.29 <0.01 <0.01	0613-90
grape, 1990	4	1.4	0.14	0 62	2.2 <0.1	0.16 <0.01	0614-90
grape, 1990 raisin	4	1.4	0.14	0 48 48	0.99 0.29 <0.1	0.01 0.01 <0.01	0615-90
grape, 1990 raisin	4	1.4	0.14	0 76 76	4.2 0.11 0.20	0.12 <0.01 <0.01	0616-90
grape, 1990	4	1.4	0.14	0 62	1.8 0.11	0.12 <0.01	0617-90
grape, 1990 raisin	4	1.4	0.14	0 48 48	3.2 0.39 0.12	0.08 0.02 0.01	0618-90
grape, 1990	4	1.4	0.14	0 76	3.8 0.15	0.10 <0.01	0620-90
grape, 1990 raisin	4	1.4	0.14	0 62 62	2.1 0.67 <0.1	0.09 0.02 <0.01	0621-90
grape, 1990	4	1.4	0.14	0 48	2.2 0.18	0.07 0.01	0623-90

Onions. In 1987, 2 residue trials were conducted in Australia with rates of 1.4 or 2.8 kg ai/ha applied 5 times per season, according to the current registered use. 14 days after the last treatment no residues of propineb were found above the lower limit of determination (0.2 mg/kg). In these trials PTU was not analyzed.

In Japan 6 residue trials were conducted according to the current use pattern. Applications were made 5 or 7 times at a rate of 1.4 or 1.75 kg ai/ha. Seven days after the last treatment the residues of propylenediamine (PDA) were <0.05 mg/kg in four samples and 0.05 and 0.08 mg/kg in the other two. Residues of PTU were below the lower limit of determination (<0.01 mg/kg). Propineb was not determined in these trials.

Melons. Four residue trials were conducted in Australia, where 7 or 8 sprays at 1.4 or 2.8 kg ai/ha were applied to melons, according to the current use pattern or at double rate. Seven days after the last treatment no measurable residues of propineb were found (<0.1 mg/kg).

In Japan, residue trials were conducted with application rates from 2.63 to 3.5 kg ai/ha, applied 3-5 times per season, according to the current use pattern. Seven to 21 days after the last application residues of propineb were in the range <0.01 to 0.03 mg/kg, and those of PDA 0.06 to 0.72 mg/kg. The results are shown in Table 5. Residues of PTU were below the lower limit of determination (0.01 mg/kg) in every sample.

Table 5. Residues of propineb in melons from supervised trials in Austraia and Japan.

Country, Year	Application		Residues (mg/kg) at days after last application								Ref.
	No.	kg ai/ha	Residue	1	3	5-6	7-8	9-14	21	28	
AUL, 1985	7 7	1.4 2.8	CS_2 CS_2	0.3 1.4	0.4 0.8	0.4 0.4	<0.2 <0.2	<0.2 <0.2			12/85A 12/85B
AUL. 1985	8 8	1.4 2.8	CS_2 CS_2	1.3 0.4	0.3 0.5	0.2 0.5	<0.1 <0.1	<0.1 <0.1			56/85A 56/85B
JPN, 1977	3	2x2.63 3.5	CS_2 PDA					<0.03 0.06- 0.15*		<0.03 0.18- 0.42*	N559 A N560 A
	5	3x2.63 2x3.5	CS_2 PDA				<0.03 0.18- 0.4*		<0.03 0.38- 0.72*		N559 B N560 B

JPN, 1977	3 5	3.0 3.0	CS_2 PDA CS_2 PDA				<0.03 0.17- 0.32*	<0.03 0.09- 0.3*	<0.03 0.20- 0.38*	<0.03 0.16	N562 A N563 A N562 B N563 B
JPN, 1984	4	3.5	CS_2	0.1			0.02				N1097
JPN, 1984	4	3.5	CS_2	0.03		<0.01					N1098

* Samples were analyzed in two laboratories.

Tomatoes. A total of 14 supervised trials were conducted in Germany in 1978, 1982 and 1987. Antracol 70 WP was applied 4 to 6 times at rates of 0.84 to 2.94 kg ai/ha. When the treatments were according to German GAP, seven days after the last application residues of propineb ranged from 0.08 to 0.55 mg/kg and those of PTU were at or below the lower limit of determination of 0.02 mg/kg (Table 6).

Potatoes. A summary of 9 supervised residue trials in Germany was provided. Three trials were conducted in 1971 in which 3 sprays, each at 1.68 kg ai/ha, were applied. No residues of propineb were found above the lower limit of determination (0.2 mg/kg) after 8, 48 or 69 days. In a further three residue trials in 1973, where 1.26 kg ai/ha was applied 4-5 times, no residues of propineb were measured above the lower limit of determination (0.1 mg/kg) after 22, 51 or 60 days. In 1979, 3 residue trials were carried out in which 8 sprays, each at 1.26 kg ai/ha, were applied. The analyses showed no measurable amounts of either propineb (<0.04 mg/kg) or PTU (<0.01 mg/kg) 7 days after the last treatment.

7 supervised trials were conducted in Australia with rates between 1.1 and 2.8 kg ai/ha applied up to 9 times per season. Although Australian GAP allows a maximum of 7 applications at 2.1 kg ai/ha, no residues of propineb could be detected in the potatoes. The results are comparable to those found in the German trials.

Table 6. Residues of propineb in tomatoes from supervised trials with Antracol 70 WP applied in 0.14 kg ai/hl in Germany.

Sample, Year	Application		Resi-due	Residues (mg/kg) at days after last applicn.						Ref.
	No.	kg ai/ha		0	1	3	4-5	7	10-14	
fruit,1978	6	0.84; 2x1.26; 3x1.68	CS_2 PTU	0.66 0.04	0.22 0.05		0.3 0.05	0.08 0.02	<0.05 0.02	8005-78
fruit,1978	6	0.84;2x1.26; 3x1.68	CS_2 PTU	0.54 0.03	0.43 0.05		0.38 0.04	0.32 0.02		8017-78
fruit,1982 juice ketchup	4	0.84; 1.68; 2.52; 2.94	CS_2 PTU CS_2 PTU CS_2 PTU					0.3 0.04 <0.05 0.02 <0.05 0.02		8019-82
fruit,1982 juice ketchup	4	0.84; 1.68; 2.52; 2.94	CS_2 PTU CS_2 PTU CS_2 PTU					0.2 0.01 <0.05 <0.01 <0.05 <0.01		8020-82
fruit,1982 juice ketchup	4	2x1.68; 2x2.52	CS_2 PTU CS_2 PTU CS_2 PTU					0.7 0.03 <0.05 0.02 <0.05 0.01		8021-82
fruit,1982 juice ketchup	4	0.84; 1.68; 2,52; 2,94	CS_2 PTU CS_2 PTU CS_2 PTU					0.6 0.04 <0.05 0.02 <0.05 0.02		8022-82
fruit,1982 juice ketchup	4	2x1.68; 2x2.52	CS_2 PTU CS_2 PTU CS_2 PTU					0.8 0.03 <0.05 0.02 <0.05 0.02		8023-82
fruit,1982	4	2x1.68; 2x2.52	CS_2 PTU					0.7 0.03		8024-82

Sample, Year	Application		Resi-due	Residues (mg/kg) at days after last applicn.						Ref.
	No.	kg ai/ha		0	1	3	4-5	7	10-14	
juice ketchup			CS_2 PTU CS_2 PTU					<0.05 0.01 <0.05 0.02		
fruit,1987	4	0.84; 1.26; 2x1.68	CS_2 PTU	1.5		0.41	0.44	0.55 <0.02	0.22 <0.02	8005-87
fruit,1987	4	4x1.26;	CS_2 PTU	0.80		0.39	0.21	0.06 <0.02	<0.05 <0.02	8006-87
fruit,1987	4	0.84; 1.26; 2x1.68	CS_2 PTU	0.87		0.38	0.20	0.11 <0.02	<0.05 <0.02	8007-87
fruit,1987	4	0.84; 1.26; 2x1.68	CS_2 PTU	0.83		0.61	0.57	0.29 0.02	0.40 0.02	8055-87
fruit,1987	4	4x1.26	CS_2 PTU	0.65		0.24	0.22	0.15 <0.02	0.18 <0.02	8056-87
fruit,1987	4	0.84; 1.26; 2x1.68	CS_2 PTU	0.45		0.29	0.21	0.14 <0.02	0.06 <0.02	8057-87

FATE OF RESIDUES

In processing

The effects of processing on propineb residues were studied with various crops. Transfer factors were calculated which indicate the relation between the residue concentrations in the processed product and the initial commodity (Walz-Tylla, 1992). The average ratio of PTU to propineb in raw commodities (P), and transfer factors (F), number of trials (n) and standard deviation of the factors (s_{n-1}) are summarized in Table 7.

Table 7. Transfer factors for propineb and PTU from raw to processed products (propineb/propineb, PTU/propineb, and PTU/PTU).

Commodity/compound		P[1]	F	n	S_{n-1}
Raw	Processed				
Apple/propineb	Apple/PTU	0.06		7	0.03
Apple/propineb	Puree/PTU		0.04	6	0.03
Apple/PTU	Puree/PTU		0.7	6	0.29
Cherry/propineb	Cherry/PTU	0.2		5	0.2
Cherry/propineb	Jam/propineb		0.4	4	0.16
Cherry/propineb	Juice/propineb		0.4	3	0.25
Cherry/propineb	Jam/PTU		0.1	4	0.13
Cherry/propineb	Juice/PTU		0.2	4	0.25
Cherry/PTU	Jam/PTU		1.2	4	1.2
Cherry/PTU	Juice/PTU		1.9	4	2.27
Grape/propineb	Grape/PTU	0.08		45	0.087
Grape/propineb	Must/propineb		0.5	38	0.51
Grape/propineb	Wine/propineb		*	52	
Grape/propineb	Must/PTU		0.2	40	0.28
Grape/propineb	Wine/PTU		0.2	47	0.21
Grape/PTU	Must/PTU		3.1	37	4.49
Grape/PTU	Wine/PTU		3.6	43	5.23
Hop/propineb	Hop/PTU	0.03		5	0.013
Hop/propineb	Beer/PTU		0.003	10	0.005
Hop/PTU	Beer/PTU		0.08	4	0.049
Tomato/propineb	Tomato/PTU	0.09		13	0.06
Tomato/propineb	Ketchup/PTU		0.2	13	0.34
Tomato/propineb	Juice/PTU		0.1	12	0.15
Tomato/PTU	Ketchup/PTU		1.5	12	1.52
Tomato/PTU	Juice/PTU		0.9	10	0.75

* No residue was detected in the wine.
[1] PTU/propineb in raw commodity.

Apples were processed to juice and puree according to household procedures. After sorting, washing and cutting the apples they were crushed in a punched disc mill produce puree, which was separated in a high-pressure press into juice and pomace. The juice was then pasteurized at 85°C for 15 to 150 seconds.

It was found that propineb residues in fruit (up to 1.0 mg/kg) were reduced below the lower limit of determination (0.05 mg/kg) during processing to apple juice and puree. The residues of PTU in apple juice and puree ranged from <0.01 mg/kg to 0.025 mg/kg and were similar to or marginally less than the residues found in fruit 21 days after the last treatment.

Grapes were processed to must and wine according to the BBA-Guideline part IV, 3-3.4. Propineb residues were significantly reduced by processing, while PTU residues were generally increased in the must and wine.

Grapes were also processed to raisins by drying grapes in the air in Turkey. The results show that during the production of raisins the concentration of propineb residues was markedly reduced, while PTU residues remained at or about the lower limit of determination (<0.01 mg/kg) in all analyzed commodities.

Tomatoes were processed to juice and puree according to industrial procedures. After sorting, washing, cutting and blanching the tomato pulp

water was added and the mixture was heated for 2 to 5 minutes at 70°C.

To obtain ketchup the tomato pulp was strained and concentrated, and other ingredients were added. After canning, the tomato ketchup was pasteurized at 93°C for 5 to 10 minutes.

To produce juice the tomato pulp was strained and sodium chloride was subsequently added. After canning, the juice was pasteurized at 93°C for 5 to 10 minutes.

No residues of propineb could be detected in the juice or ketchup, showing a substantial reduction from the residue found in the fruit (0.11 to 0.70 mg/kg).

In all 6 trials the residues of PTU in the juice and ketchup were similar to the levels found in the fruit (0.01-0.04 mg/kg) at a 7-day pre-harvest interval.

METHODS OF RESIDUE ANALYSIS

Propineb

Residues of fungicides belonging to the dimethyldithiocarbamate and ethylene-bis(dithiocarbamate) groups can be determined by colorimetric as well as gas-chromatographic methods.

Several colorimetric methods for the residue analysis of propineb have been developed. Some of these were referred to in the 1977 and 1984 JMPR monographs (Keppel, 1969; Otto *et al.*, 1977; Thier, 1977). The principle of these methods is the determination of propineb by acid decomposition and spectrophotometric measurement of the evolved carbon disulphide (CS_2). The method of Thier (1979) is based on the same principle and is suitable for enforcement purposes.

Nakahara and Aizawa (1978) developed a gas-chromatographic method for the determination of propineb residues in crops. The method is based on the measurement of the carbon disulphide (CS_2) and propylenediamine (PDA) produced by acidic hydrolysis in the presence of stannous chloride. The CS_2 is quantified by GLC in a gas chromatograph equipped with a flame-photometric detector. After derivatization to 1,2-bis(trifluoroacetamido)propane, PDA is determined by GC-MS with selective ion monitoring. A modified version of this method has been validated by Specht (1993) in apples, grapes, grape juice, wine, potatoes and tomatoes. The recoveries ranged from 71 to 113%. The limit of determination was 0.05 mg/kg.

Propylenethiourea (PTU)

Kobayashi *et al.* (1981), Ohs (1988) and Meier (1982) described methods for the determination of ethylenethiourea (ETU) and propylenethiourea (PTU) in plant materials and their processed products, especially beer and wine.

In these methods the residues are determined by HPLC with UV detection after extraction and clean-up. The recoveries ranged from 70 to 110%. The limit of determination was reported as 0.02 mg/kg in plant commodities and 0.004 mg/kg in beer, wine and fruit juices.

NATIONAL MAXIMUM RESIDUE LIMITS

National MRLs reported to the Meeting are summarized below.

The following country codes are used: ARG-Argentina, AUL-Australia, AUS-Austria, BEL-Belgium, CHI-Chile, CYP-Cyprus, DEN-Denmark, FIN-Finland, FRA-France, GER-Germany, HUN-Hungary, ISR-Israel, ITA-Italy, JPN-Japan, LUX-Luxembourg, MAL-Malaysia, MOZ-Mozambique, NET-Netherlands, NZE-New Zealand, POR-Portugal, SAF-South Africa, SPA-Spain, SWE-Sweden, SWI-Switzerland, TAW-Taiwan, TUR-Turkey, YUG-Yugoslavia.

Section 1: Argentina - Japan

Commodity Country	ARG[1]	AUL[1]	AUS[1]	BEL[1]	CHI[1]	CYP[1]	DEN[1]	FIN[1]	FRA[1]	GER[1]	HUN	ISR	ITA	JPN
Almond												1		
Apple					3	3					2			
Banana						1		0.1						
Bean							1		0.5					
Bell pepper											3 5[2]			
Berries + Small fruits							2							
Brussels sprouts							1							
Bulb vegetables				0.5					0.5					
Cabbage							1							
Carrot					0.5	0.5	0.5	0.5				0.5		
Cauliflower							1							
Cereals						0.2		0.1	0.05				2	
Celery		2										5		
Cherry, Sweet					1	1								
Citrus fruits		0.2					2					3		
Coffee										2				
Cucumber						0.5		0.5		1	2	0.5		
Cucurbits		1												
Endive									0.5					
Fruit			2	2						2				0.7
Fruiting vegetables		1					1							
Garlic												0.5		
Grape	2				5				1		2		2	
Hop	0.2		25	25						25	80			
Leafy + Stem vegetables									0.5					
Leafy vegetables							2							
Lettuce														

Commodity Country	ARG[1]	AUL[1]	AUS[1]	BEL[1]	CHI[1]	CYP[1]	DEN[1]	FIN[1]	FRA[1]	GER[1]	HUN	ISR	ITA	JPN
					1	1						5		
Lettuce, Head		4							4					
Melon						1								
Mushroom											2			
Oil plants										2				
Onion		4										0.5		
Other fruits								1	2					
Other plant commodities			0.05	0						0.2	2	0.1	0.2	
Other vegetables				2				1	2	2				
Peach					3	3								
Pear					3	3					2			
Plum					1	1								
Pome fruits							2		1				2	
Potatoes	0.5	0.02			0.1	0.1	0.1	0.1	0.05			0.1		
Root + Tuber Vegetables				0.5										
Root vegetables									0.5					
Root veg. exc. Carrots							0.5							
Spice										2		3		
Spinach									0.5					
Strawberry						3						3		
Stem vegetables							1							
Stone fruit							2		1					
Tea										2				
Tea-like products										2				
Tobacco											25			
Tomato	2	3			3	3			1	1	3 5[2]	3	2	
Tropical fruits							2							
Vegetables			2											0.1
Watermelon						1								
Wheat					0.2									

Section 2: Luxembourg - Yugoslavia

Commodity Country	LUX[1]	MAL[1]	MOZ	NET[1]	NZE	POR[1]	SAF[1]	SPA[1]	SWE[1]	SWI[1]	TAW	TUR	YUG
Apple											1		
Asparagus											0.1		
Aubergine											0.5		
Bamboo											0.1		
Banana											1 0.2		
Bean												2	
Bell pepper											0.5	1	
Berries + Small fruits				3									
Boysenberry							3						
Bulb vegetables				0.5									
Carrot									0.5		0.1		
Cereals		0.5		0.5					0.1[3]	0.1			
Cereals, processed									0.1[3]				
Chick-pea												1	
Cucumber				1								1	
Dewberry							3						
Egg plant											0.5		
Fruit	2	5							1	2			2
Ginger											0.1		
Grape			3				3 2[4]	4					
Grapefruit											1 0.2[5]		
Hop								4					
Leafy vegetables		5											
Leguminosae				0.2									
Lemon											1 0.2[5]		
Lettuce												1	
Lettuce, head				4						2			
Litchi											1 0.2[5]		
Longan											1 0.2[5]		
Loquat											1		
Mango											1 0.2[5]		
Melon	1			1							1 0.2[5]		
Melon, netted (musk)											1 0.2[5]	1	
Melon, water-											1 0.2[5]	1	
Nut, pea-							0.5						

Commodity Country	LUX[1]	MAL[1]	MOZ	NET[1]	NZE	POR[1]	SAF[1]	SPA[1]	SWE[1]	SWI[1]	TAW	TUR	YUG
Onion					0.5						0.1		
Orange											1 0.2[5]		
Other fruits				2				3					
Other plant commodities	0.05			0				0.2					0.05
Other vegetables	2	3		2									
Papaya											1 0.2[5]		
Pea, Chick-												1	
Peach											1		
Peanut							0.5						
Pear											1		
Pepper, Cayenne-											0.5	1	
Pineapple											1 0.2[5]		
Plum											1		
Potato			0.5	0.2		0.05	0.5	0.2	0.1	0.05	0.1		
Pummelo											1 0.2[5]		
Radish, small											0.1		
Root vegetables		1											
Strawberry								4					
Taro (Dasheen)											0.1		
Tobacco		25								50			2
Tomato	1		3				3				0.5	1	
Vegetables										2			2
Vegetables exc. carrots									1				
Vegetables exc. potatoes								3					

[1] Total dithiocarbamates expressed as CS_2
[2] Produced for canning.
[3] Level at or about the limit of determination
[4] In export commodities
[5] Without peel

APPRAISAL

Propineb was evaluated in 1977, 1984 and 1985. The temporary ADI was withdrawn by the 1985 JMPR, but the CCPR maintained the Guideline Levels for propylenethiourea PTU). The compounds are included in the CCPR periodic review programme.

Propineb is currently registered on a large number of crops in several countries around the world, but the Meeting was informed that its actual use is restricted to a few crops. The results of numerous supervised field trials and processing studies were provided by the principal manufacturer only for grapes, tomatoes, potatoes, pome fruits, onions and melons. The use of the compound will still be recommended on these crops and on bell peppers, but the use recommendations for other crops are due to be withdrawn.

The metabolism in plants has been sufficiently presented in the 1984 Evaluations. The laboratory animal metabolism studies are discussed in the Toxicological Evaluations. The

metabolic pathways in plants and animals are essentially the same. Propylenediamine and 4-methylimidazoline identified in animals were present in the form of *N*-formylpropylendiamine and 2-methoxy-4-methylimidazoline, respectively. No information was available on metabolism by farm animals or on animal transfer studies.

In the supervised trials, propineb residues were determined and expressed as mg/kg CS_2, and propylenethiourea (PTU) residues were determined and expressed as mg/kg PTU throughout.

In apples and pears, the residues of propineb ranged from <0.05 mg/kg to 0.96 mg/kg 14-21 days after the last treatment. For the main metabolite, PTU, the results were in the range of <0.02 mg/kg to 0.08 mg/kg. If propineb were used alone the estimated maximum residue levels on apples and pears would be 2 mg/kg propineb and 0.1 mg/kg PTU.

In grapes, at pre-harvest intervals ranging from 49 to 69 days, residues of propineb and PTU were between <0.05 and 1.2 mg/kg, and <0.01 and 0.08 mg/kg respectively, except in one trial where 2.1 mg/kg propineb and 0.15 mg/kg PTU were measured. Following a different application schedule, the residues of propineb were more or less in the same range. The variety of grape did not influence the residue levels. If propineb were used alone the estimated maximum residue levels on grapes would be 2 mg/kg propineb and 0.1 mg/kg PTU.

In onions, 14 days after the last treatment, no residues of propineb were found above the limit of determination (0.2 mg/kg) in Australia. PTU was not determined. In Japanese trials seven days after the last treatment the residues of propylenediamine (PDA) were <0.05 mg/kg in four samples and 0.05 and 0.08 mg/kg in two samples, while the residues of PTU were below the limit of determination (<0.01 mg/kg). Propineb was not determined. The data are not sufficient to estimate a maximum residue level for the use of propineb on onions.

In melons, 7 to 21 days after the last treatment the residues of propineb and PTU were below the limits of determination (0.01-0.2 and 0.01 mg/kg respectively) in all samples, while PDA ranged between 0.06 and 0.72 mg/kg. If propineb were used alone the estimated maximum residue levels in melons would be 0.2 mg/kg propineb and 0.05 mg/kg PTU, both levels being at or about the limit of determination.

In tomatoes treated according to German GAP, seven days after the last application residues of propineb ranged from 0.08 to 0.55 mg/kg and residues of PTU were at or below the limit of determination of 0.02 mg/kg. If propineb were used alone the estimated maximum residue levels in tomatoes would be 1 mg/kg for propineb and 0.05 mg/kg for PTU.

In potatoes no residues of propineb or PTU were found above the limits of determination (0.2 mg/kg and 0.01 mg/kg respectively) within 8 to 69 days after the last treatment. These residue trials do not completely correspond to the current registered uses but they cover present good agricultural practice as the application rate was higher. All trials showed that in spite of the great variations in pre-harvest interval no residues of propineb or the major metabolite PTU could be detected in potatoes. If propineb were used alone the estimated maximum residue levels in potatoes would be 0.2 mg/kg propineb and 0.05 mg/kg PTU (the limits of determination).

The effects of processing on the residues were extensively studied on apples, cherries, grapes, hops and tomatoes. These studies showed that the concentration of propineb residues was reduced to non-detectable (<0.02 mg/kg) in the case of apple juice and puree, wine, beer, and tomato juice and ketchup, while in cherry juice and jam the average propineb residue was 40% of that in the fruits. The residue level of PTU in processed products is primarily influenced by the level of propineb and the mode of processing. The ratio of PTU in the processed product to propineb in the raw commodity was 0.04 for apple puree, 0.003 for beer, 0.2 for cherry juice, 0.1 for cherry jam, 0.2 for must and wine, 0.1 for tomato juice and 0.2 for ketchup. The residue levels of PTU were higher in products where the processing involves extensive contact with the peel of the harvested crop as in red

wine and tomato ketchup.

The freezer storage stability of the residues in samples has not been studied systematically. However the Meeting was informed that the repeated analyses of samples analysed when taken and after prolonged freezer storage did not show any difference in the residue levels. Samples were always frozen whole before storage and homogenized deep-frozen before analysis in order to eliminate decomposition of the residues. The Meeting noted that this information provided on propineb residues was consistent with the results of frozen storage stability studies on mancozeb reported under that heading. It was also considered likely that the results of frozen storage stability studies on ETU would apply to PTU.

No information was reported on PTU levels in food moving in commerce or at consumption.

Residue analytical methods are available to determine propineb residues as CS_2, using colorimetric or GLC detection, and PTU residues by HPLC. These methods are suitable for regulatory purposes with limits of determination of 0.1-0.2 mg/kg for CS_2 and 0.05 mg/kg for PTU. The propineb residues can be qualitatively distinguished from the other dithiocarbamates by converting them to propylenediamine which can be determined by gas chromatography after derivatization.

RECOMMENDATIONS

On the basis of data on residues from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits. Since the origin of CS_2 cannot be identified, the maximum residue levels estimated for the dithiocarbamate group have to be taken into account.

Definition of the residue: propineb: CS_2
PTU: propylenethiourea

Commodity		Recommended MRL (mg/kg)				
		New		Previous		PHI (days)
		CS_2 CS_2[1]	PTU	CS_2[2]	PTU	
FP 0226	Apple	3 (2)	0.1	3	0.1	21
VR 0578	Celeriac		w		0.05	
FS 0243	Cherry, sour		w	1	0.1	
FB 0269	Grapes	5 (2)	0.1	5	0.1	56
VC 0046	Melons	0.1*				7
VA 0385	Onion, bulb	0.2*	0.02*			7
FS 0247	Peach		w	3	0.05	
FP 0230	Pear	3 (2)	0.1	3	0.1	21
FS 0014	Plums (including Prunes)		w	1	0.1	
VR 0589	Potato	0.1*	0.02*	0.1	0.02	7
VO 0448	Tomato	3 (1)	0.05	3	0.1	7

[1] MRLs based on the current residue data from supervised trials.
[2] Codex MRLs for the group of dithiocarbamates (propineb was not included).
* At or about the limit of determination.

FURTHER WORK OR INFORMATION

Desirable

1. Residue data from supervised trials on bell peppers.
2. Freezer storage stability studies on propineb and PTU residues in representative commodities.
3. Metabolism study on farm animals.
4. Residue transfer study on farm animals.
5. Monitoring data on PTU in food in commerce and at consumption.

REFERENCES

1. Bayer AG, Germany, 1982-91. Unpublished reports on supervised trials in apples. Report Nos.:8009-82, 8010-82, 8008-87, 0023-88, 0024-88, RA-2004/91 (includes BBA-reports 0038-91, 0039-91, 0040-91, 0041-91)

2. Bayer AG, Germany, 1981-90. Unpublished reports on supervised trials in grapes. Report

Nos.:8000-81, 8001-81, 8002-81, 8003-81, 8004-81, 8025-82, 8026-82, 8027-82, 8028-82, 8029-82, 8030-82, 8034-82, 8035-82, 8036-82, 8037-82, 8038-82, 8039-82, 8000-84, 8001-84, 8002-84, 8003-84, 8004-84, 8005-84, 8000-86N, 8000-86G, 8001-86Z, 8001-86N, 8002-86N, 8002-86G, 8003-86N, 8003-86Z, 8004-86N, 8004-86G, 8005-86Z, 8005-86N, 8006-86N, 8006-86G, 8007-86Z, 8007-86N, 8008-86N, 8008-86G, 8009-86Z, 8009-86N, 8000-87, 8002-87, 8050-87, 8051-87, 8052-87, 0406-88, 0408-88, 0409-88, 0610-90, 0611-90, 0612-90, 0613-90, 0614-90, 0615-90, 0616-90, 0617-90, 0618-90, 0620-90, 0621-90, 0623-90

3. Bayer AG, Germany, 1982-87. Unpublished reports on supervised trials in pears. Report Nos.:8011-82, 8010-87, 8060-87

4. Bayer AG, Germany, 1971-79. Unpublished reports on supervised trials in potatoes. Report Nos.:83/71, 84/71, 85/71, 428/73, 429/73, 430/73, 8000-79, 8001-79, 8002-79

5. Bayer AG, Germany, 1978-87. Unpublished reports on supervised trials in tomatoes. Report Nos.:8005/78, 8017/78, 8019-82, 8020-82, 8021-82, 8022-82, 8023-82, 8024-82, 8005-87, 8006-87, 8007-87, 8055-87, 8056-87, 8057-87

6. Bayer AG, Germany, 1982. Unpublished processing studies on apples. Report Nos.: 8009-82, 8010-82

7. Bayer AG, Germany, 1981-90. Unpublished processing studies on grapes. Report Nos.: 8000-81, 8001-81, 8002-81, 8003-81, 8004-81, 8025-82, 8026-82, 8027-82, 8029-82, 8030-82, 8034-82, 8035-82, 8036-82, 8037-82, 8038-82, 8039-82, 8000-84, 8001-84, 8002-84, 8003-84, 8004-84, 8005-84, 8000-86N, 8000-86G, 8001-86Z, 8001-86N, 8002-86N, 8002-86G, 8003-86N, 8003-86Z, 8004-86N, 8004-86G, 8005-86Z, 8005-86N, 8006-86N, 8006-86G, 8007-86Z, 8007-86N, 8008-86N, 8008-86G, 8009-86Z, 8009-86N, 8000-87, 8001-87, 8002-87, 8050-87, 8051-87, 8052-87, 0406-88, 0408-88, 0409-88, 0611-90, 0613-90, 0615-90, 0616-90, 0618-90, 0621-90

8. Bayer AG, Germany, 1982. Unpublished processing studies on pears. Report Nos.: 8011-82

9. Bayer AG, Germany, 1982. Unpublished processing studies on tomatoes. Report Nos.:8019-82, 8020-82, 8021-82, 8022-82, 8023-82, 8024-82

10. Bayer Australia, 1985. Unpublished reports on supervised trials in melons. Report Nos.12/85A, 12/85B, 56/85A, 56/85B,

11. Bayer Australia, 1987. Unpublished reports on supervised trials in onions. Report Nos.24/87A, 24/87B

12. Bayer Australia, 1983-86. Unpublished reports on supervised trials in potatoes. Report Nos.38/83A, 38/83B, 39/83A, 39/83B, 39/83C, 38/86A, 38/86B

13. Keppel G.E.1971. Collaborative Study of the Determination of Dithiocarbamate Residues by a modified Carbon Disulfide Evolution Method. J.Ass.off.Anal.Chem 54, (3), 528-532

14. Kobayashi, H.; Matano, O. and Goto S.; 1981. Simultaneous quantitation of thioureas in rat plasma by high-performance liquid chromatography. J. Chromatogr. 207, 281-285

15. Meier, J.; 1982. Methode zur gleichzeitigen Bestimmung von ETU and PTU im Bier und anderen Getranken mittels Hochdruckflussigkeitschromatographie. Brauwissenschaft 35, 229-230

16. Nakahara, T. and Aizawa T.; 1978. Gas-liquid Chromatographic Method for Residues of Propineb in Crops. Unpublished Nihon Bayer Report, no. 1082

17. Nihon Bayer, 1977-84. Unpublished reports on supervised trials in melons. Report Nos.N559 A, N559 B, N560 A, N560 B, N561 A, N561 B, N562 A, N562 B, N563 A, N563 B, N564 A, N564 B, N1097, N1098

18. Nihon Bayer, 1977-78 . Unpublished reports on supervised trials in onions. Report Nos.N565 A, N565 B, N566 A, N566 B, N567 A, N567 B, N568 A, N568 B, N693, N694, N695, N696

19. Ohs, P.; 1988. Method for the liquid chromatographic determination of ethylenethiourea and propylenethiourea residues in plant materials and their processed products after application of dithiocarbamates. Bayer AG, unpublished report (RA-658/87) April 8, 1988

20. Otto, S.; Keller, W. and Drescher, N.; 1977. A New Gas Chromatographic Determination of Ethylenethiourea Residues without Derivatization. J. Environ. Sci. Health 12, 179-191

21. Spain, 1993, Information on pesticides being on the priority list of CCPR.

22. Thier, H.P.; 1977. Arbeitsgruppe "Pestizede", zur Ruckstandsanalytik der Pestizide in Lebensmitteln, 5. Mitteilung: Analytik von Dithiocarbamat-Ruckstanden. Lebensmittelchem. gerichtl. Chem. 31, 25-27 (internal method no. 00028)

23. Thier, H.P.; 1979. Method S 15, Deutsche Forschungsgesellschaft, Manual of Pesticide Residue Analysis, Vol 1 VCH Verlagsgesellschaft Weinheim, 1987

24. Walz-Tylla, B.; 1992. Influence of processing on the formation of PTU from propineb. Bayer AG, unpublished report (RA-409/92) July 10, 1992

PYRAZOPHOS (153)

EXPLANATION

Pyrazophos was reviewed for the first time in 1985 but the data base at that time was inadequate for the estimation of an ADI; some Guideline Levels were recorded. Some additional data were reviewed in 1987 and in 1992, when the toxicological data were deemed sufficient for the estimation of an ADI, the previous Guideline Levels being converted, unchanged, into MRLs. However, the late submission of relevant information on GAP did not allow enough time to complete the evaluation of the residue data. This was therefore postponed until this Meeting.

Although full up-to-date GAP data were provided this year, only limited information was received relating to the previously expressed desiderata on the effects of processing on incurred residues of pyrazophos. No new data were presented on residues in animal products or the identities of plant metabolites.

USE PATTERN

Pyrazophos is a systemic fungicide especially active against powdery mildews and is generally used, both for preventive and curative treatments, as a 30% emulsifiable concentrate, with some use of a 30% wettable powder. Major uses are on cereals, cucurbits and some fruits, with lesser use on vegetables and hops and some applications in the culture of ornamentals and tobacco.

Information was provided on current GAP in 25 countries; this is summarized in Table 1.

Table 1. Pyrazophos - registered use rates and patterns. Nearly all formulations used are 30% EC; a very few are 15% WP mixtures.

Crop	Country	Appl. rate (kg ai/ha)	No. of appl.	PHI (days)	Comments
Apple	Argentina	0.09	-	21	10-14 day intervals
	Costa Rica	0.012-0.03	-	15	7-15 day intervals
	Egypt	0.45	1	-	Interval, 15 days
	France	0.3	3	15	
	Germany	0.221 or 0.331	15 or 12	28	7-14 day intervals
	Greece	0.135-0.48	1-2	14	
	Indonesia	0.1-0.2	4	14	
	Portugal	0.15-0.3	1-2	21	
	Rep. of Korea	1.35	unlim.	14	
	UK	0.165	-	-	7-14 day intervals
Apricot	Egypt	0.45	1	-	Interval, 15 days
	France	0.3	3	15	

Crop	Country	Appl. rate (kg ai/ha)	No. of appl.	PHI (days)	Comments
Artichoke, Globe	Italy	0.04-0.08	2	7	
Barley	France	0.2	3	30	
	Germany	0.3 or 0.588	max. 2	49	
	Ireland	0.6	2	-	
	Rep. of Korea	0.36	unlim.	42	
Broccoli	Netherlands	0.15-0.3	-	-	
Brussels sprouts	Ireland	0.33-0.99	1	14	
	Netherlands	0.15-0.3	-	21 or 35	
	UK	0.33	1-3	14	
Cauliflower	Netherlands	0.15-0.3	-	-	
Cereals	Spain	0.045-0.12	1-2	15	
Chinese cabbage	Netherlands	0.15-0.3	-	21	
Citrus fruits	Indonesia	0.15-0.2	3	7-14	
Cole crops	Costa Rica	0.012-0.03	-	15	7-15 day intervals
	Dominican Republic	0.012-0.03	-	-	7-14 day intervals
Common bean	Spain	0.18-0.45	2-3	15	
Cucumber	Belgium	0.5-0.75	max. 10	3	
	Denmark	0.24	max. 10	3	
	France	0.15	3	3	
	Germany	0.07-0.141	max. 10	3	7 to 10 day intervals
	Greece	0.054-0.336	1-2	3	
	Netherlands	0.15-0.3	-	3	
	Portugal	0.15	2-3	3	
	Republic of Korea	0.45	unlim.	3	
Cucurbits	Argentina	0.09	-	7	10 day intervals
	Costa Rica	0.012-0.03	-	15	7-15 day intervals
	Dominican Republic	?	-	-	7-14 day intervals
	Egypt	0.12	1	-	Interval, 10- 14 days
	Guatemala	0.067	1	-	Local use only
	Italy	0.04-0.08	2-3	7	
	Philippines	Spot treatment	-	3	3-9 g ai/16 l
	Spain	0.18-0.45	2-4	15	
	Venezuela	0.075-0.18	-	3	7-14 day intervals
Egg plant	Spain	0.18-0.45	2-3	15	
Fruit trees	Denmark	0.3	4	14	
Fruits	Colombia	0.435	-	-	
	Ecuador	0.09	2	-	
	Venezuela	0.075-0.18	-	3	7-14 day intervals
Gherkin	Netherlands	0.12-0.15	-	3	
Grapes	Argentina	0.09-0.18	-	28	
	Dominican Republic	?	-	-	7-14 day intervals

Crop	Country	Appl. rate (kg ai/ha)	No. of appl.	PHI (days)	Comments
	Egypt	0.714	1	-	Interval, 15 days
	Greece	0.054-0.192	2-3	21	
	Italy	0.14-0.21	1-2	21	
	Thailand	0.015-0.045	3	21	
Hops	UK	max 0.33	1-3	-	10-14 day intervals
Mango	Egypt	2.14	2	-	Interval, 15 days
	Thailand	0.027-0.09	10	14	
Melons	Belgium	0.5-0.75	max. 10	3	
	Brazil	0.18	2	7	
	France	0.15	3	3	
	Greece	0.054-0.336	1-2	3	
	Netherlands	0.12-0.15	-	3	
	Portugal	0.15	2-3	3	
Mushrooms	Netherlands	1.5	-	3	
Peach	Argentina	0.18	-	21	10 day intervals
	Costa Rica	0.012-0.03	-	15	7-15 day intervals
	Egypt	0.60	1	-	Interval, 15 days
	France	0.3	3	15	
	Italy	0.14-0.21	2	21	
Pear	Argentina	0.09	-	21	10-14 day intervals
	France	0.3	3	15	
Peppers, Sweet	Spain	0.18-0.45	2-3	15	
Pome fruit	Spain	0.15-0.3	1-3	15	
Rambutan	Thailand	0.027-0.09	5	14	
Rye	Germany	0.3 or 0.588	max. 2	49	
Scorzonera	Netherlands	0.15	-	7	
Spring barley	UK	0.6	1-3	-	
Squash, Summer	France	0.15	3	3	
	Netherlands	0.12-0.15	-	3	
Stone fruit	Spain	0.15-0.3	1-3	15	
Strawberry	Belgium	0.5-0.75	max. 7	-	Before flowering & after harvest
	Costa Rica	0.012-0.03	-	15	7-15 day intervals
	Denmark	0.24	2-3	21	Up to 3 weeks before harvest
	Dominican Republic	?	-	-	7-14 day intervals
	Germany	0.235	max. 7	-	After harvest
	Greece	0.036-0.096	1-3	7	
	Italy	0.036-0.06	2	7	
	Netherlands	0.06	5-10	14	
Swede	Netherlands	0.3	-	21	
Tomato	Spain	0.18-0.45	2-3	15	
Vegetables	Brazil	0.18	2	7	

Crop	Country	Appl. rate (kg ai/ha)	No. of appl.	PHI (days)	Comments
	Colombia	0.29-0.435	-	-	
	Ecuador	0.15	2	-	
	Greece	0.054-0.288	1-2	21	
	Philippines	Spot treatment	-	3	3-9 g ai/16 l
	Thailand	0.015-0.045	5	14	
	Venezuela	0.075-0.18	-	3	7-14 day intervals
Watermelon	Indonesia	0.12-0.18	4	7	
	Philippines	Spot treatment	-	3	3-9 g ai/16 l
	Taiwan	0.15	-	-	
Wheat	Germany	0.3 or 0.588	2 or 1	49	
	Netherlands	0.15	-	21	
Winter barley	Denmark	0.6	max. 2	42	
	UK	0.6	1-3	-	

RESIDUES RESULTING FROM SUPERVISED TRIALS

Additional residue data were provided by the manufacturer, Hoechst AG, from supervised trials on fruits, vegetables, and cereals. The earlier data recorded in 1985 (FAO/WHO 1986b) and 1987 (FAO/WHO 1988a) were also reconsidered at this Meeting. Some additional limited residue data were also provided by The Netherlands (Netherlands, 1993) and Spain (Spain, 1993). Summaries of the new data are given in Tables 2, 3 and 4. All reported residues are expressed as mg pyrazophos/kg sample; the analytical method determined only the parent molecule, no relevant levels of metabolites having been detected in plant products.

Fruits - see Table 2.

Apples. In a number of supervised trials carried out in France, Germany and the UK, residues ranged up to about 0.4 mg/kg at the normal PHI of 28 days, consistent with the current MRL of 0.5 mg/kg, although one result of 0.9 mg/kg was observed.

Peaches and nectarines. Four residue trials were carried out in Italy in 1977 at rates of 0.23 and 0.45 kg ai/ha. Residues were up to 0.30 mg/kg, 28 days after treatment at the higher rate. There is no current MRL for peaches.

In one glasshouse trial in Spain in 1988 (Spain, 1993) residues were 0.27 to 0.40 mg/kg, mean 0.33 mg/kg, after 14 days and 0.13 to 0.25 mg/kg, mean 0.19 mg/kg, after 21 days.

Strawberries. Data submitted from three trials in 1967, 1970 and 1976, not previously evaluated, showed that residues were below 0.07 mg/kg after 19-32 days, within the existing MRL of 0.2 mg/kg.

Four trials on strawberries in Spain from 1986 to 1990 (Spain, 1993) showed residues from 0.13 to 0.64 mg/kg after 14 days. Two trials in The Netherlands in 1977 showed residues ranging from 0.04 to 0.08 mg/kg after 7 days, and 0.01 to 0.03 mg/kg after 14/15 days (Netherlands, 1993).

Table 2. Residues of pyrazophos in fruit from supervised trials. All trials were with 30% EC unless otherwise noted.

Crop/ Country/Year	Application		No. of trials	Residues, mg/kg, at days after last appl.	Ref.
	kg/ha	No.			
Apple					
France '73	0.36	9	1	0.03 (32)	A 01354
'75	0.42	9	2	0.06, 0.07 (34)	A 07780,07782
	0.84	9	2	0.07, 0.07 (34)	A 07781,07783
Germany'74	0.23	14	2	<0.05 (21) [30% WP]	A 04072,04074
	0.3	12	2	0.10 (21) [WP]; 0.20 (21)	A 04070,04076
	0.45	9	2	0.40 (14); 0.20 (21) [30% WP]	A 04073,04075
	0.6	10	1	1.0 (10), 0.40 (14), 0.20 (21) [30% WP]	A 04071
	0.6	9	1	6.0 (10), 0.3 (14), 0.2 (21)	A 04077
'80	0.15	12	1	0.40 (17), 0.40 (22), 0.10 (27)	A 21359
'81	0.22 -0.44	12	1	0.90 (14), 0.60 (21), 0.40 (28)	A 27694
	0.44	12	1	1.0 (14), 2.0 (21), 0.90 (28)	A 27693
'82	0.33	3	2	0.30, 0.40 (21), 0.04, 0.20 (28)	A 25641,25642
'87	0.13 -0.33	7	4	0.10-0.39 (21), 0.02-0.06 (70)	A 39422-3, 38797-8
UK '74	0.18	4	2	<0.05 (72)	A 05726,05728
	0.18	7	1	<0.05 (90)	A 05727
Nectarine					
Italy '77	0.23	2	1	0.30 (14), 0.07 (21), 0.05 (28)	A 12478
	0.45	2	1	0.40 (14), 0.10 (21), 0.30 (28)	A 12477
Peach					
Italy '77	0.23	2	1	0.90 (14), 0.30 (21), 0.20 (28)	A 12566
	0.45	2	1	0.40 (14), 0.30 (21), 0.20 (28)	A 12567
Spain '88	1.1	1	1	0.70 (7), 0.33 (14), 0.19 (21) [G'house]	Spain 1993
Strawberry					
Finland'76	0.26	3	1	<0.03 (19)	A 14176
N'lands'67	0.15	4	1	0.06 (26), 0.07 (32)	A 00324
'70	0.12	4	1	0.01 (14), <0.007 (21)	A 01209
Spain '86	0.03	1	1	0.18 (0), 0.95 (3), 0.37 (7), 0.17 (14)	Spain 1993
Spain '87	0.03	1	1	0.72 (0), 0.58 (3), 0.29 (7), 0.17 (14)	Spain 1993
Spain '88	0.03	1	1	1.7 (0). 1.1 (3), 0.92 (7), 0.64 (14)	Spain 1993

Crop/ Country/Year	Application		No. of trials	Residues, mg/kg, at days after last appl.	Ref.
	kg/ha	No.			
Spain '90	0.03	1	1	0.98 (0), 0.95 (3), 0.78 (7), 0.36 (14)	Spain 1993

Vegetables - See Table 3.

Brussels sprouts. Trials data from Germany, The Netherlands and the UK obtained between 1969 and 1984 showed no residues above 0.09 mg/kg after about 21 days, within the current MRL of 0.1 mg/kg.

Cucumbers. Data from field and greenhouse culture studies in Japan showed residues to be generally below 0.01 mg/kg in the whole fruit, 7 or more days after treatment. The current MRL is 0.1 mg/kg.

Egg plant. In two trials in Spain in 1990, residues of 0.05 and <0.01 mg/kg were found 15 days after treatment.

Peppers. Data from 4 trials in Japan in 1970 and 4 in Spain in 1990 were made available to the Meeting. At the usual PHI of 14 days, residues were 0.04 mg/kg or less; at 7 days, a maximum of 0.32 mg/kg was found in Japan.

Tomato. In one trial in Spain in 1989 (Spain, 1993) residues ranged from 0.70 mg/kg at 2 days to 0.25 mg/kg at 15 days.

Beetroot. Residues in beetroot 27 days after application were below 0.02 mg/kg in one trial (Netherlands, 1993).

Carrots. No new data on trials on carrots were supplied by Hoeschst. Data from one trial in The Netherlands in 1984 (Netherlands, 1993) showed no residue above the limit of determination of 0.02 mg/kg. The existing MRL is 0.2 mg/kg.

Witloof chicory. In a trial in 1984 in The Netherlands, residues were below 0.02 mg/kg after 60 days (Netherlands, 1993).

Table 3. Residues in vegetables from supervised trials with 30% EC.

Crop/ Country/Year	Application	No of trials	Residues, mg/kg, at days after last appl.	Ref.

	kg ai/ha	No.			
Brussels sprouts					
Germany '84	0.07	4	1	0.25 (7), 0.13 (14), 0.09 (21)	A 33447
N'lands '69	0.3	1	1	0.05 (12), 0.03 (20)	A 00377
'70	0.12	4	1	<0.02 (7), 0.06 (10), 0.05 (14)	A 01047
	0.12-0.3	4	1	0.05 (7), 0.05(10), <0.02 (14)	A 01046
UK '72	0.34	4	1	0.05 (17)	A 01178
Cucumber					
Japan '73	0.45	3	2	<0.01 (7-21) [Field]	A 15610-1
	0.45	3	2	<0.01 (7-21) [Greenhouse]	A 15615,15620
	0.45	5	2	<0.01 (7-21) [Field]	A 15614,15617
	0.45	5	2	<0.01 (7-21) [Greenhouse]	A 15612,15621
	0.75	3	2	<0.01 (7-21)	A 15613,15618
	0.75	5	2	<0.01 (7-21)	A 15616,15619
Spain '92	0.24	1	1	0.09 (3), <0.01 (7, 15) [G'house]	Spain 1993
Melon					
Spain '92	0.2-0.25	1	4	0.03-0.05 (7), 0.02-0.04 (15) [G'house]	Spain 1993
	0.45	1	2	0.08, 0.22 (7);0.05, 0.12 (15) [G'house]	Spain 1993
Squash, Summer					
Spain '92	0.26	1	1	0.17 (3), <0.01 (7, 15) [G'house]	Spain 1993
	0.30	1	1	0.07 (3), 0.01(7), <0.01 (15) [G'house]	Spain 1993
	0.36	1	1	0.06 (3), 0.02 (7), <0.01 (15) [G'house]	Spain 1993
	0.38	1	1	0.06 (3), <0.01 (7, 15) [G'house]	Spain 1993
	0.39	1	1	0.01 (3), <0.01 (7, 15)	Spain 1993
Watermelon					
Spain '92	0.21	1	1	0.03 (7), 0.04 (15)	Spain 1993
	0.22	1	1	0.01 (7), <0.01 (15)	Spain 1993
	0.23	1	1	<0.01 (7, 15)	Spain 1993
	0.26	1	1	0.01 (7), <0.01 (15)	Spain 1993
Egg plant					
Spain '90	0.34	1	2	0.07,0.10 (7), <0.01,0.05 (15)	Spain 1993
Pepper					
Spain '90	0.22	1	4	0.08-0.17 (7), 0.02-0.04 (14)	Spain 1993
Tomato					
Spain '89	0.45	1	1	0.57 (7), 0.32 (10), 0.25 (15) [G'house]	Spain 1993
Beetroot					
N'lands '84	0.15	2	1	<0.02 (27)	N'lands 1993
Carrot					
N'lands '84	0.15	2	1	<0.02 (27)	N'lands 1993
Witloof chicory					
N'lands'84	0.15	2	1	<0.02 (60)	N'lands 1993

Cereals - See Table 4.

Barley. Data from an additional 15 trials on barley were submitted. In all cases residues in the grain were below the current MRL of 0.05 mg/kg.

Rye. No new residue data for rye were available; the previous data were re-evaluated but again deemed insufficient for any recommendation for an MRL.

Wheat. Details of a total of 54 additional trials were made available to the Meeting, all of which were conducted in Germany in 1975-76. The current MRL is 0.05 mg/kg and the new data only served to confirm that figure.

Table 4. Residues of pyrazophos in cereals from supervised trials, all with 30% EC unless otherwise noted.

Crop/ Country/Year	Application		No. of trials	Residues, mg/kg, at days after last appl.	Ref.
	kg/ha	No.			
Barley (grain)					
Germany '74	0.15	1	1	<0.01 (112)	A 04557
'76	0.3	2	4	<0.01-0.04 (49-56) [30% WP]	A 12789,90,92,95
'83-84	0.59	2	2	<0.05 (66, 76)	A 31247-8
'89	2.25	2	2	<0.01; 0.23 (49)	A 46994-5
Barley (straw)					
'74	0.15	1	3	<0.05, 0.05 (75); <0.01 (112)	A 10177,10179,04557
'76	0.3	2	4	<0.05, 0.10, 0.20, 0.20 (50-56) [30% WP]	A 12788,91,93,94
Wheat (grain)					
Germany '75	0.15	1	4	<0.02 (34-58)	A 05740-3
'76	0.3	1	16	<0.01-0.02 (33-47) [30% WP]	See {1} below
	0.3	2	5	<0.01-<0.02 (55-56) [30% WP]	A 09276-90
	0.6	2	3	<0.02 (55-56) [30% WP]	A 09278,83,84
Wheat (straw)					
Germany '75	0.15	1	4	<0.06-0.20 (34-58)	A 05740-3
'76	0.3	1	16	0.20-4.7 (33-47) [30% WP]	See {2} below
	0.3	2	5	0.07-1.3 (55-56) [30% WP]	A 09277,80,87,98,91
	0.6	2	3	0.20-2.8 (55-56) [30% WP]	A 09279,83,85

{1} References:- A 09194,96,98,9200,38,41,42.44,93,95,96,98,9317,18,20,22

{2} References:- A 09193,95,97,99,9239,40,43,45,92,94,97,99,9316,19,21,66

Dried herbs

Hops. No new data on residues in dry hops were provided. The existing MRL for hops (dry) is 10 mg/kg.

FATE OF RESIDUES

In storage and processing

In two processing studies on apples in Germany in 1987, residues in pressed apple juice and in a cooked mash were below the limit of determination, 0.01 mg/kg (Hoechst, A 38797, 38798).

After a trial on barley, in which an application rate of 4 times the normal was used, residues in the grain were 0.23 mg/kg; conversion of this grain to malt showed a residue of 0.09 mg/kg, while residues in the beer prepared from this were below the limit of determination of 0.01 mg/kg (Hoechst, A 46995).

Processing studies were carried out on wheat in Germany in 1989 in order to investigate the possible residue contamination of flour, bran and bread. Wheat was treated at 5 times the usual application rate (2.9 kg ai/ha) and at harvest, 49 to 51 days after treatment, the residues in the grain ranged from 0.038 to 0.048 mg/kg. After normal processing, the whole wheat flour showed 0.014 mg/kg, bran contained 0.02 to 0.035 mg/kg, while wholemeal bread had residues which were only just above the limit of determination of 0.01 mg/kg. At normal rates of application, residues in all of these products are unlikely to be measurable (Hoechst, A 46996, 46997).

NATIONAL MAXIMUM RESIDUE LIMITS

The Meeting received information on the following MRLs in Spain.

Commodity	MRL, mg/kg
Apple	0.20
Cereals	0.10
Common bean	0.10
Cucurbits	0.10
Peach	0.20
Peppers	0.20
Tomatoes	0.20

APPRAISAL

Owing to the late submission of GAP information, evaluation of the residue data for pyrazophos was postponed from the 1992 Meeting, at which an ADI was estimated and the existing Guideline Levels became MRLs. Full GAP information has now been recorded and some previously unreported data on residues resulting from supervised trials on fruits, vegetables and cereals have been evaluated, together with those included in the 1985 and 1987 reviews. In general, these additional data served only to reinforce the conclusions reached earlier. There were insufficient new data to support any recommendations on new crops, other than on barley and wheat straw.

For apples, the additional data were deemed adequate to

support a recommendation to increase the MRL from 0.5 to 1 mg/kg. Data for strawberries, Brussels sprouts, cucumbers, barley and wheat supported the recommendations previously made for those crops. MRLs could also now be recommended for barley and wheat straw, based mainly on the ample trials data on wheat treatments. No additional data were received for carrots and hops, but a review of the data previously reported led to confirmation of the existing recommendations.

Data on residues in a few crops for which MRLs had not previously been recommended, namely nectarine, peach, summer squash, watermelon, egg plant, peppers, tomato, beetroot and Witloof chicory, were mostly only in summary form and, in any event, they were deemed insufficient as a basis for any MRL recommendation.

Some limited data on residue changes during the processing of barley and wheat were made available in response to previous requests. These showed that residues of pyrazophos were unlikely to be found in beer or bread produced from crops treated according to GAP. Residues in pressed apple juice and in a cooked mash from treated apples were below 0.01 mg/kg. Data on residues in animal products and the identities of plant metabolites were still not available.

RECOMMENDATIONS

On the basis of the data on residues resulting from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits.

Definition of the residue: pyrazophos.

Commodity		Recommended MRL (mg/kg)		PHI on which
CCN	Name	New	Previous	based, days
FP 0226	Apple	1	0.5	28
GC 0640	Barley	0.05	0.05	49-56
AS 0640	Barley straw and fodder, dry	5	-	49-56
VB 0402	Brussels sprouts	0.1	0.1	21
VR 0577	Carrot	0.2	0.2	28
VC 0424	Cucumber	0.1	0.1	3
DH 1100	Hops, dry	10	10	16
VC 0046	Melons, except Watermelon	0.1	0.1	3
FB 0275	Strawberry	0.2	0.2	

14-21			
GC 0654	Wheat	0.05	0.05
33-58			
AS 0654	Wheat straw and fodder, dry	5	-
33-58			

FURTHER WORK OR INFORMATION

Desirable

1. Information on residues in meat and milk from cattle, meat from pigs, and meat and eggs from poultry, fed on a diet containing pyrazophos.

2. Additional information on the identities and quantities of metabolites in plants after treatment with pyrazophos.

3. Full reports of the residue trials supplied in summary form by Spain and The Netherlands.

REFERENCES

(All references are unpublished)

Hoechst AG, 1993. A large number of residue reports in the A..... series, as indicated in the Tables or text as appropriate.

Netherlands, 1993. Data on GAP and residues supplied by The Netherlands for the JMPR.

Spain, 1993. Data on GAP and residues supplied by Spain for the JMPR.

TRIAZOPHOS (143)

EXPLANATION

Triazophos was evaluated in 1982 and has been reviewed several times since, most recently in 1991. At the 23rd and 24th Sessions of the CCPR (1991-92) some countries questioned the interpretation of residue data on citrus fruits and bananas, and undertook to send written comments to the JMPR. It was also suggested that the TMRLs proposed for residues in Brussels sprouts, head cabbages and carrots should be higher, while those for common beans and cauliflower were too high. The question was also raised as to whether 0.01 or 0.05 mg/kg was a realistic limit of determination for cereal grains, bulb onions, potatoes and sugar beet, and the JMPR was asked to reconsider this limit in the light of further data which had been requested. The UK stated that information on GAP and residue data from supervised trials on carrots would be made available to the JMPR.

The Meeting had received written comments on the proposed limits for triazophos in citrus fruits and bananas from France, Germany and The Netherlands and comments on the limits for Brussels sprouts and head cabbage from The Netherlands. Information was also available on GAP and residues from trials on carrots in the UK, including residues after cooking, and the UK provided extensive data from a survey of residues in carrots with a known application history. Residue data were also received from supervised trials on strawberries in Germany and soya beans in Brazil.

USE PATTERN

New registered uses of triazophos in the UK and Spain are listed in Table 1.

Table 1. Registered uses of triazophos.

Crop	Country	Application			PHI
		No.	kg ai/ha	kg ai/hl	days
Citrus fruits	Spain		0.9-1.2	0.06-0.08	40
Apples	Spain		2.4-3.2	0.06-0.08	30
Brassicas	UK	3	0.35		28
Carrots and parsnips	UK	3-6	0.53-1.05		28
Sugar beet	Spain			0.06-0.08	30

RESIDUES RESULTING FROM SUPERVISED TRIALS

Residue data were available from supervised trials on strawberries, carrots and soya beans.

Strawberries. In twelve supervised trials carried out in Germany residues with one exception were below or near the limit of determination (Table 2).

Table 2. Residues of triazophos in strawberries from supervised trials in Germany.

Year	Application			PHI, days	Residues, mg/kg	Report
	No.	kg ai/ha	kg ai/hl			
1969	1	0.96	0.04	34	<0.01 <0.01	A01111 A01112
	1	1.9	0.08	34	<0.01 <0.01	A01113 A01114
1973	1	1.5	0.06	22 28 35 42 49	0.11 0.03 <0.01 <0.01 <0.01	A01074
1974	1	1.6	0.08	43 49 61 68	<0.03 <0.03 0.04 <0.03 <0.03 <0.03 <0.03 <0.03	A01912 A01913
	1	1.6	0.08	46 53 60 67	<0.03 <0.03 <0.03 <0.03	A0914
	1	1.6	0.08	42 49 56 63	<0.03 <0.03 <0.03 <0.03	A0915
	1	1.6	0.08	56 63 73 80	<0.02 <0.01 <0.02 0.01 <0.02 <0.01 <0.02 <0.01	A02279 A02280
1977	2	0.84	0.04	39 57	<0.01 <0.01	A19564

Carrots. Ten supervised trials were carried out in the UK and all except two with applications in accordance with registered UK uses. Residues were 0.06-0.23 mg/kg 28 days after the last aplication. Carrots from six of the trials were cooked for 15 minutes in an open pan in water containing 0.035% sodium chloride. The residues in the cooked carrots ranged from 6% to 73% of the original residues (Table 3).

Table 3. Residues of triazophos in raw and cooked carrots from supervised trials in the UK.

Year	Application No	kg ai/ha	kg ai/hl	PHI, days	Residues, mg/kg	Report
1991	3	1.05	0.11	14 28 44	0.18 0.08 0.08	A47679 0501
	3	2.1	0.21	14 28 44	0.40 0.32 0.36	
	3	1.05	0.11	0 14 28 43	0.19* 0.19* 0.23* 0.34*	A47679 0901
	3	2.1	0.21	0 14 28 43	0.41* 0.42* 0.34* 0.24*	A47679 0901
1992	3	1.05	0.11	0 28 28	0.38 0.38 0.16 0.23 Cooked: 0.01 0.03	0401 0402
	3	1.05	0.11	0 27-28 27-28	0.12 0.30 0.23 0.18 Cooked: 0.04 0.04	0501 0502
	3	1.05	0.11	0 26 26	0.06 0.14 0.12 0.11 Cooked: 0.09 0.06	0901 0902

* Residues in untreated samples were of the same order in these trials.

Soya beans. Residues in seeds and hulls from trials on soya beans were very low, mostly below the limit of determination. Whole plants were also analysed in two trials with residues at 12-13 mg/kg just after application (Table 4).

Table 4. Residues of triazophos in soya beans from supervised trials in Brazil.

Year	Application No	kg ai/ha	kg ai/hl	PHI, days	Residues, mg/kg	Report
1978	2	0.40	0.13	51	Seed: 0.02 0.10 <0.01	A14649 A14650 A15555
1982	1	0.42	0.17	0 43 120 120	Plant: 13 0.10 Seed: <0.001 Hull: <0.001	A30042
	1	0.50	0.20	0 43 120 120	Plant: 12 0.12 Seed: <0.001 Hull: <0.001	A30043
1983	1	0.42	0.17	69 69	Seed: <0.001 Hull: 0.025	A30044
	1	0.50	0.20	69 69	Seed: <0.001 Hull: 0.027	A40045

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

In a survey in the UK 81 samples of carrots were taken in 1991 and 1992 from crops with a known history of treatment with triazophos in accordance with label recommandations. The application rates were 0.53 or 1.05 kg ai/ha, the number of treatments from 1 to 3 and the PHI from 78 to 250 days, mostly between 100 and 200 days. Residues ranged from <0.02 to 0.94 mg/kg with a mean of 0.17 mg/kg, and the 90th percentile was approximately 0.4 mg/kg.

APPRAISAL

Triazophos was evaluated in 1982 and several times since then, most recently in 1991. Maximum residue levels were estimated for a number of commodities and recommended as TMRLs because the ADI was temporary. At the 23rd and 24th Sessions of the CCPR (1991-92) the proposed TMRLs for citrus fruits, bananas, Brussels sprouts, head cabbages, common beans and cauliflower were held at step 7b and referred back to the JMPR. Written comments were received from France, Germany and The Netherlands on citrus fruits, bananas, Brussels sprouts and head cabbages. The manufacturer submitted new residue data from supervised trials on carrots, strawberries and soya beans.

At the same Sessions of the CCPR it was proposed to lower the limit of determination from 0.05 mg/kg to 0.01 mg/kg for residues of triazophos in cereal grains, potatoes, bulb onions and sugar beet.

The Meeting took note of the observations made by France, Germany and The Netherlands, who stated that the data base was insufficient to set residue limits for citrus fruits and bananas. The Meeting examined the data and proposed to withdraw the TMRL of 1 mg/kg for bananas, as the limit is based on residue data from only two trials. It was also difficult to link the summarized data for citrus fruits to the information on GAP in the 1983 Evaluation. The Meeting was informed that new information on GAP for triazophos and residue data from a number of trials on citrus fruits would be available in the near future, and therefore recommended that the limit for triazophos in citrus fruits should be made temporary, irrespective of the status of the ADI, until the new data were evaluated.

In 1986 the JMPR had already re-evaluated and confirmed the proposed limits for residues in Brussels sprouts and head cabbages. No new data were available. The Meeting reaffirmed the recommendation.

The Meeting considered the limit of determination of 0.05 mg/kg for triazophos in cereal grains, potatoes, bulb onions and sugar beets. The residues in these crops reported to the 1983 and 1990 Meetings were below the limit of determination, which ranged from 0.001 to 0.07 mg/kg. Although most residues were below 0.02 mg/kg, the Meeting proposed to maintain the limit of determination of 0.05 mg/kg as a realistic limit of determination for the purpose of enforcement.

Supervised trials on carrots were carried out in the UK, all except two in accordance with recommended use. The highest residue in samples from two trials 28 days after the last treatment was 0.23 mg/kg. In addition to the trials a survey was carried out in the UK in which many samples of carrots with a known pesticide treatment history were analysed, all applications being in accordance with label recommendations. Residues were from <0.02 mg/kg to 0.94 mg/kg with a mean value of 0.17 mg/kg, and the 90th percentile was approximately 0.4 mg/kg. The Meeting recommended a maximum residue limit of 0.5 mg/kg for carrots.

Supervised trials were also carried out on strawberries in Germany. Residues were very low and except for the PHI of 11 days all residues were below 0.05 mg/kg. No information on GAP on in Germany was available, but GAP in The Netherlands would give rise to even lower residues. The Meeting estimated a maximum residue level at the limit of determination for strawberries (0.05 mg/kg).

Residues in seeds from trials on soya beans in Brazil with application in accordance with Brazilian GAP were also below the limit of determination, and a maximum residue limit was proposed at the limit of determination (0.05 mg/kg).

RECOMMENDATIONS

On the basis on the residues from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits.

Definition of the residue: triazpphos.

Commodity		Recommended MRL (mg/kg)		PHI on which
CNN	Name	New	previous[1]	based, days
FI 0327	Banana	W	1	
VR 0577	Carrot	0.5	0.1	28
FC 0001	Citrus fruits	2 T	2	
VD 0541	Soya bean (dry)	0.05*	-	50
FB 0275	Strawberry	0.05*	-	before flower-ing

W: the previous recommendation is withdrawn.
[1] previous recommendations were temporary because the ADI was temporary. As a full ADI has now been allocated, all unchanged limites except that for citrus fruits become full MRLs.

FURTHER WORK OR INFORMATION

Required (by 1994).

Information on GAP for triazophos on citrus fruits and residue data from trials in accordance with GAP.

ANNEX I

ACCEPTABLE DAILY INTAKES AND RESIDUE LIMITS PROPOSED AT THE 1993 MEETING

The table of recommendations includes maximum Acceptable Daily Intakes (ADIs) and Maximum Residue Limits (MRLs). It should be noted that MRLs include draft MRLS and Codex MRLs (CXLs). The MRLs recommended by the JMPR on the basis of its estimates of maximum residue levels enter the Codex procedure as draft MRLs. They become Codex MRLs when they have passed through the procedure and have been adopted by the Codex Alimentarius Commission.

In general, the recommended MRLs listed for compounds which have been reviewed previously are additional to, or amend, those recorded in Annexes to the reports of earlier meetings. For compounds re-evaluated in the CCPR periodic review programme however, both new and previous recommendations are listed because such re-evaluations are regarded as replacing the original evaluation rather than supplementing it.

Limits recommended at meetings from 1965 to 1977 inclusive are summarized in document FAO/WHO 1978c.

Some ADIs are temporary: this is indicated by the letter T and the year in which re-evaluation is scheduled in parenthesis below the ADI. All recommended MRLs for compounds with temporary ADIs are necessarily temporary, but some recommendations are designated as temporary (TMRLs) until required information has been provided and evaluated, irrespective of the status of the ADI. Such recommendations are followed by the letter T in the table. (See also the list of qualifications and abbreviations below.)

The following qualifications and abbreviations are used.

*	At or about the limit of determination
E	Extraneous Residue Limit (ERL).
F (following recommendations for milk)	The residue is fat-soluble and MRLs for milk and milk products are derived as explained in the introduction to Part 2 of the Guide to Codex Maximum Limits for Pesticide Residues and to Volume II of the Codex Alimentarius.
(fat) (following recommendations for neat)	The recommendation applies to the fat of meat.
Po	The recommendation accommodates post-harvest treatment of the commodity.
PoP (following recommendations for processed foods (classes D and E in the Codex Classification)	The recommendation accommodates post-harvest treatment of the primary food commodity.
T (following ADIs)	The ADI is temporary, and due for re-evaluation in the year indicated.
T (following MRLs)	The MRL is temporary, irrespective of the status of the

	ADI, until required information has been provided and evaluated.
V (following recommendations for commodities of animal origin)	The recommendation accommodates veterinary uses.
W (in place of an MRL)	The previous recommendation is withdrawn.

If a recommended MRL is an amendment, the previous value is also recorded. The absence of a figure in the "Previous" column indicates that the recommendation is the first for the commodity or group concerned.

The table includes the Codex Classification Numbers (CCNs) of both the compounds and the commodities listed, to facilitate reference to the Guide to Codex Maximum Limits for Pesticide Residues.

Commodities are listed in alphabetical order. This is a change from previous practice where commodities were listed in the order of the "Types" in the Codex Classification of Foods and Animal Feeds, and in alphabetical order within each Type.

The change has been made to facilitate checking and comparison with the CCPR Tables of MRLs, which are in alphabetical order.

ACCEPTABLE DAILY INTAKES (ADIs) AND MAXIMUM RESIDUE LIMITS (MRLs)

}Pesticide (Codex Ref. No.)	Max. ADI (mg/ kg bw)	Commodity		Recommended MRL or ERL (mg/kg)	
		CCN	Name	New	Previous
Aldicarb (117)	0.003	VB 0402	Brussels sprouts	0.1	0.05 T
			Residue: sum of aldicarb, its sulphoxide and its sulphone, expressed as aldicarb		
Amitrole [1] (079)	0.0005 T (1997)		Note TADI increased from 0.00003 mg/kg bw, conditional		
Azinphos-methyl (002)	0.005	AS 0654	Wheat straw and fodder, dry	W	1 T
			Residue: azinphos-methyl		
Benalaxyl (155)	0.05	VR 0589	Potato	0.02*	0.01*
			Residue: benalaxyl		
Bromopropylate [1] (070)	0.03	FP 0226	Apple	W	5
		FI 0327	Banana	W	5
		VP 0526	Common bean (pods and/or immature seeds)	3	-
		FS 0013	Cherries	W	5
		FC 0001	Citrus fruits	2	5
		SO 0691	Cotton seed	W	1
		VC 0424	Cucumber	0.5	-
		FB 0269	Grapes	2	5
		DH 1100	Hops, dry	W	5
		VC 0046	Melons, except Watermelon	0.5	-
		FS 0245	Nectarine	W	5
		FS 0247	Peach	W	5
		FP 0230	Pear	W	5
		FS 0014	Plums (including prunes)	2	5
		FP 0009	Pome fruits	2	-
		VC 0431	Squash, Summer	0.5	-
		FB 0275	Strawberry	2	5
		DT 1114	Tea, Green, Black	W	5
			Vegetables	W	1
			Residue: bromopropylate		
			Note ADI increased from 0.008 mg/kg bw		
Carbofuran (096)	0.01	FC 0001	Citrus fruits	W	2 T
			Residue: sum of carbofuran and 3-hydroxycarbofuran		

[1] Re-evaluation in periodic review programme

Pesticide (Codex Ref. No.)	Max. ADI (mg/ kg bw)	Commodity		Recommended MRL or ERL (mg/kg)	
		CCN	Name	New	Previous
Carbosulfan (145)	0.01	FC 0001	Citrus fruits Residue: carbosulfan	W	2 T
Chlorothalonil [1] (081)	0.03	FI 0327	Banana	W	0.2
		GC 0640	Barley	0.1	0.2 (cereals)
		AS 0640	Barley straw and fodder, dry	20	-
Chlorothalonil (contd.)		FB 0264	Blackberries	W	10
		VB 0400	Broccoli	W	5
		VB 0402	Brussels sprouts	5	5
		VB 0041	Cabbages, Head	1	5
		VR 0577	Carrot	1	1
		VB 0404	Cauliflower	1	5
		VS 0624	Celery	10	15
		GC 0080	Cereal grains	W	0.2
		FS 0013	Cherries	0.5	10
		FC 0001	Citrus fruits	W	5
		VP 0526	Common bean (pods and/or immature seeds)	5	5
		FB 0265	Cranberry	5	5
		VC 0424	Cucumber	5	5
		FB 0021	Currants, Black, Red, White	W	25
		VL 0476	Endive	W	10
		FB 0269	Grapes	0.5	10
		VL 0480	Kale	W	10
		VL 0482	Lettuce, Head	W	10
		VD 0534	Lima bean (dry)	W	0.5
		VC 0046	Melons, except Watermelon	2	5
		VA 0385	Onion, Bulb	0.5	5
		FS 0247	Peach	1	25
		SO 0697	Peanut	0.05	0.1
		SO 0703	Peanut, whole	W	0.5
		VO 0051	Peppers	W	10
		VR 0589	Potato	0.2	0.1
		VC 0429	Pumpkins	W	5
		FB 0272	Raspberries, Red, Black	W	10
		VC 0431	Squash, Summer	5	5
		VR 0596	Sugar beet	0.2	1
		AV 0596	Sugar beet leaves or tops	20	-
		VO 0447	Sweet corn (corn-on the-cob)	W	1
		VO 0448	Tomato	5	5

[1] Re-evaluation in periodic review programme

		GC 0654	Wheat	0.1	0.2 (cereals)
		AS 0654	Wheat straw and fodder, dry	20	-
		VC 0433	Winter squash	5	5
		VS 0469	Witloof chicory (sprouts)	W	10
			Residue: chlorothalonil		
Chlorpyrifos-methyl (090)	0.01	SO 0495	Rape seed	W	10 Po T
			Residue: chlorpyrifos-methyl		
Cycloxydim (179)	0.07	VD 0071	Beans (dry)	2	-
		VB 0040	Brassica vegetables	2	-
		VR 0577	Carrot	0.5	-
		VP 0526	Common bean (pods and/or immature seeds)	1	-
Cycloxydim (contd.)		FB 0269	Grapes	0.5	-
		VA 0384	Leek	0.2	-
		VL 0482	Lettuce, Head	0.2	-
		VL 0483	Lettuce, Leaf	0.2	-
		VP 0063	Peas	1	-
		VP 0064	Peas, shelled	2	-
		VR 0589	Potato	2	-
		SO 0495	Rape seed	2	-
		VD 0541	Soya bean (dry)	2	-
		FB 0275	Strawberry	0.5	-
		VR 0596	Sugar beet	0.2	-
		AV 0596	Sugar beet tops or leaves	1	-
			Residue: sum of 3-thian-3-ylglutaric acid (TME) and 3-hydroxy-3-thian-3-ylglutaric acid (OH-TME), expressed as cycloxydim		
DDT (021)	0.02	VR 0577	Carrots	0.2 E	-
		PE 0112	Eggs	0.1 E	0.5 E T
		MM 0095	Meat	1 (fat) E	5 (fat) E T
		ML 0106	Milks	0.02 F E	0.05 F E T
			Residue: Sum of p,p_-DDT, o,p_-DDT, p,p_-DDE and p,p_-TDE (DDD) (fat-soluble)		
Diazinon [1] (022)	0.002		Almond, hull	5	-
		TN 0660	Almonds	0.05	0.1
		GC 0640	Barley	W	0.1
		FB 0264	Blackberries	0.1	0.5[1]
		FB 4079	Boysenberry	0.1	0.5[1]
		VB 0400	Broccoli	0.5	0.5[2]
		VB 0041	Cabbages, Head	2	0.5[2]
		VC 4199	Cantaloupe	0.2	0.5[2]
		VR 0577	Carrot	0.5	0.5[2]
		FS 0013	Cherries	1	0.5[1]

[1] Re-evaluation in periodic review programme

		VL 0467	Chinese cabbage	0.05	0.7[3]
		FC 0001	Citrus fruits	W	0.7
		VP 0526	Common bean (pods and/or immature seeds)	0.2	0.5[2]
		SO 0691	Cotton seed	W	0.1
		VC 0424	Cucumber	0.1	0.5[2]
		FB 0021	Currants, Black, Red, White	0.2	0.5[1]
			Fruits (except as otherwise listed)	W	0.5
		VP 0529	Garden pea, shelled	0.2	0.5[2]
		TN 0666	Hazelnuts	W	0.1
		VL 0480	Kale	0.05	0.7[3]
		FI 0341	Kiwifruit	0.2	0.5[1]
		VB 0405	Kohlrabi	0.2	0.5[2]
Diazinon (contd.)		VL 0053	Leafy vegetables	W	0.7
		VL 0482	Lettuce, Head	0.5	0.7[3]
		VL 0483	Lettuce, Leaf	0.5	0.7[3]
		AF 0645	Maize forage	10	-
		GC 0645	Maize	0.02*	-
		MM 0097	Meat of cattle, pigs and sheep	W	0.7 (fat) V
		ML 0106	Milks	W	0.02 F V
		OC 0305	Olive oil, virgin	W	2
		FT 0305	Olives	W	2
		VA 0385	Onion, Bulb	0.05	0.5[2]
		FS 0247	Peach	0.2	0.7
		SO 0697	Peanut	W	0.1
		TN 0672	Pecan	W	0.1
		VO 0445	Peppers, Sweet	0.05	0.5[2]
		FI 0353	Pineapple	0.1	0.5[1]
		FS 0014	Plums (including Prunes)	1	0.5[1]
		FP 0009	Pome fruits	2	0.5[1]
		VR 0589	Potato	0.01*	0.5[2]
		DF 0014	Prunes	2	0.5[1]
		VR 0494	Radish	0.1	0.5[2]
		FB 0272	Raspberries, Red, Black	0.2	0.5[1]
		CM 1205	Rice, polished	W	0.1
		SO 0699	Safflower seed	W	0.1
		VL 0502	Spinach	0.5	0.7[3]
		VA 0389	Spring onion	1	0.5[2]
		VC 0431	Squash, Summer	0.05	0.5[2]
		FB 0275	Strawberry	0.1	0.5[1]
		AV 0596	Sugar beet leaves or tops	5	-
		VR 0596	Sugar beet	0.1	0.5[2]
		SO 0702	Sunflower seed	W	0.1

[1] Re-evaluation in periodic review programme

		VO 0447	Sweet corn (corn-on-the-cob)	0.02	0.7
		VO 0448	Tomato	0.5	0.5[2]
			Vegetables (except as otherwise listed)	W	0.5
		TN 0678	Walnuts	0.01*	0.1
		GC 0654	Wheat	W	0.1
			Residue: diazinon (fat-soluble)		
			Notes ADI confirmed		
			[1] Fruits (except as otherwise listed) [2] Vegetables (except as otherwise listed) [3] Leafy vegetables		
Dichlorvos [1] (025)	0.004	VP 0061	Beans, except broad bean and soya bean	W	0.5 for Vegetables...
		SB 0715	Cacao beans	W	5
		GC 0080	Cereal grains	5 (Po)	2
		SB 0716	Coffee beans	W	2
Dichlorvos (contd.)		PE 0112	Eggs	W	0.05
			Fruits	W	0.1
		MM 0814	Goat meat	W	0.05
		VD 0533	Lentil (dry)	W	2
		VL 0482	Lettuce, Head	W	1
		MM 0097	Meat of cattle, pigs & sheep	W	0.05
		MM 0095	Meat	0.05*	
		ML 0106	Milks	0.02*	0.02
		VD 0541	Soya bean (dry)	W	2 Po
			Vegetables (except as otherwise listed)	W	0.5
		CM 0654	Wheat bran, unprocessed	10	-
		CF 1211	Wheat flour	1	-
		CF 1210	Wheat germ	10	-
		CF 1212	Wheat wholemeal	2	-
			Residue: dichlorvos		
			Note ADI confirmed		
Diquat (031)	0.002		Note ADI is for diquat ion. Lowered from 0.008 mg/kg bw		
Dithiocarbamates [1] (105)	See Note		Almond hulls	20	-
		TN 0660	Almonds	0.1*	-
		VS 0621	Asparagus	0.1	-
		FI 0327	Banana	2	1
		GC 0640	Barley	1	-
		AS 0640	Barley straw and fodder, dry	25	-
		VB 0041	Cabbages, Head	5	-
		VR 0577	Carrot	1	0.5
		VS 0624	Celery	W	5

[1] Re-evaluation in periodic review programme

		FS 0013	Cherries	W	1
		VP 0526	Common bean (pods and/or immature seeds)	W	0.5
		VL 0510	Cos lettuce	10	-
		FB 0265	Cranberry	5	-
		VC 0424	Cucumber	2	0.5
		FB 0021	Currants, Black, Red, White	10	5
		MO 0105	Edible offal (Mammalian)	0.1	-
		PE 0112	Eggs	0.05*	-
		VA 0381	Garlic	0.5	-
		FB 0269	Grapes	5	5
		VL 0480	Kale	15	-
		VA 0384	Leek	0.5	-
		VL 0482	Lettuce, Head	10	5
		AS 0645	Maize fodder	2	-
		FC 0003	Mandarins	10	-
		MM 0095	Meat	0.02*	-
Dithiocarbamates (contd.)		VC 0046	Melons, except Watermelon	0.5	1
		ML 0106	Milks	0.05*	-
		VA 0385	Onion, Bulb	0.5	-
		FC 0004	Oranges, Sweet, Sour	2	-
		FI 0350	Papaya	5	-
		FS 0247	Peach	W	3
		SO 0697	Peanut	0.1*	-
		AL 0697	Peanut fodder	5	-
		VO 0445	Peppers, Sweet	1	-
		FS 0014	Plums (including Prunes)	W	1
		FP 0009	Pome fruits	5	Apple 3 Pear 3
		VR 0589	Potato	0.2	0.1
		PM 0111	Poultry, Edible offal of	0.1	-
		PO 0110	Poultry meat	0.1	-
		VC 0429	Pumpkins	0.2	-
		VA 0389	Spring onion	10	-
		FB 0275	Strawberry	W	3
		VR 0596	Sugar beet	0.5	-
		AV 0596	Sugar beet leaves or tops	20	-
		VC 0431	Squash, Summer	1	-
		VO 0447	Sweet corn (corn-on-the-cob)	0.1*	-
		VO 0448	Tomato	5	3
		VC 0432	Watermelon	1	-
		GC 0654	Wheat	1	0.2
		AS 0654	Wheat straw and fodder, dry	25	-
		VC 0433	Winter squash	0.1	-

[1] Re-evaluation in periodic review programme

			Residue: CS_2		
			Notes 1. Group ADI for ethylenebis(dithiocarbamate)s (EBDCs) - mancozeb, maneb, metiram and zineb - 0-0.03 mg/kg bw, alone or in any combination. ADIs for mancozeb, maneb and zineb previously 0-0.05 mg/kg bw. See also ethylenethiourea, propineb and propylenethi-ourea. 2. Recommendations for MRLs apply to total residues arising from the use of any or each of the groups of dithiocarbamates.		
Endosulfan (032)	0.006	VP 0522	Broad bean (green pods and immature seeds)	0.5	2[1]
		SB 0715	Cacao beans	0.1	-
		SB 0716	Coffee beans	0.1	-
		VC 0424	Cucumber	0.5	2[1]
			Fruits	W	2
		FB 0269	Grapes	1	2[2]
		GC 0645	Maize	0.1	-
		VC 0046	Melons, except Watermelon	0.5	2[1]
		FC 0004	Oranges, Sweet, Sour	0.5	2[2]
		FS 0247	Peach	1	2[2]
		FI 0353	Pineapple	2 Po	2[2]
Endosulfan (contd.)		SO 0495	Rape seed	0.5	-
		VD 0541	Soya bean (dry)	1	2[1]
		VC 0431	Squash, Summer	0.5	2[1]
		SO 0702	Sunflower seed	1	-
		VO 0448	Tomato	0.5	2[1]
			Vegetables, except as otherwise listed	W	2
		GC 0654	Wheat	0.2	-
			Residue: sum of alpha- and beta-endosulfan and endosulfan sulphate (fat-soluble)		
			Notes [1] Vegetables, except as otherwise listed [2] Fruits		
Ethephon (106)	0.05		Note As an ADI has now been allocated, previous GLs would normally be recommended as MRLs. Since ethephon is now scheduled for residue evalu-ation in 1984 however, adoption of the Gls as MRLs is not at present recommended		
Ethylenethiourea [1] (ETU, 108)	0.004	VR 0577	Carrot	W	0.01*
		VS 0624	Celery	W	0.01*
		VL 0482	Lettuce, Head	W	0.01*
		VR 0589	Potato	W	0.01*
			Residue: ethylenethiourea		
			Note ADI increased from TADI of 0.002 mg/kg bw. There are no other TMRLs		
Etofenprox [2]	0.03	FP 0009	Pome fruits	1	-

[1] Re-evaluation in periodic review programme

(184)		VR 0589	Potato	0.01*	-
			Residue: etofenprox (fat-soluble)		
Fenbutatin oxide [1]	0.03	TN 0660	Almonds	0.5	-
(109)		FP 0226	Apple	W[1]	5
		AB 0226	Apple pomace, dry	40	20
		FI 0327	Banana	10	-
		FS 0013	Cherries	10	5
		PO 0840	Chicken, Edible offal of	0.05*	-
		PM 0840	Chicken meat	0.05*	-
		FC 0001	Citrus fruits	W[2]	5
		AB 0001	Citrus pulp, dry	25	7
		VC 0424	Cucumber	0.5	1
		MO 0105	Edible offal (Mammalian)	0.2	-
		VO 0440	Egg plant	W	1
		PE 0112	Eggs	0.05	-
		VC 0425	Gherkin	W	1
		FB 0269	Grapes	5	5
		FC 0203	Grapefruit	5	5 (citrus)
		AB 0269	Grape pomace, dry	100	-
		MO 1292	Horse, kidney	W[3]	0.2
		MO 1293	Horse, liver	W[3]	0.2
Fenbutatin oxide (contd.)		MO 0098	Kidney of cattle, goats, pigs, and sheep	W[3]	0.2
		MO 0099	Liver of cattle, goats, pigs and sheep	W[3]	0.2
		FC 0206	Mandarin	5	5 (citrus)
		MM 0095	Meat	0.05*	-
		MM 0096	Meat of cattle, goats, horses, pigs and sheep	W[4]	0.02*
		VC 0046	Melons, except Watermelon	W	1
		ML 0106	Milks	0.05*	0.02*
		FC 0208	Orange, Sweet	5	5 (citrus)
		FS 0247	Peaches	7	7
		FP 0230	Pear	W[1]	5
		TN 0672	Pecans	0.5	-
		VO 0445	Peppers, Sweet	W	1
		FS 0014	Plums (including Prunes)	3	3
		FP 0009	Pome fruits	5	Apple 5 Pear 5
		DF 0014	Prunes [dried plums]	10	-
		DF 5263	Raisins	20	-
		FB 0275	Strawberry	10	3
		VO 0448	Tomato	1	1
		TN 0678	Walnuts	0.5	-
			Residue: fenbutatin oxide		

[1] Re-evaluation in periodic review programme

[2] New compound

			Notes [1] Replaced by limit for Pome fruit [2] Replaced by separate limits for Grapefruit, Mandarin, and Orange, Sweet [3] Replaced by Edible offal (mammalian) [4] Replaced by revised limit for Meat		
Fenpropathrin [2]	0.03	MO 0812	Cattle, Edible offal of	0.05	-
(185)		MM 0812	Cattle meat	0.5 (fat)	-
		ML 0812	Cattle milk	0.1 F	-
		SO 0691	Cotton seed	1	-
		OC 0691	Cotton seed oil, crude	3	-
		PE 0112	Eggs	0.01*	-
		VO 0440	Egg plant	0.2	-
		VC 0425	Gherkin	0.2	-
		FB 0269	Grapes	5	-
		VO 0445	Peppers, Sweet	1	-
		FP 0009	Pome fruits	5	-
		PO 0111	Poultry, Edible offal of	0.01*	-
		PM 0111	Poultry meat	0.02 (fat)	-
		VO 0448	Tomato	1	-
			Residue: fenpropathrin (fat-soluble)		
Fentin	0.0005	DH 1100	Hops, dry	0.5	1
(040)			Residue: fentin, excluding inorganic tin and di- and mono-phenyltin		
Flucythrinate	0.02	MM 0812	Cattle meat	W	0.5 (fat) T
(152)		ML 0812	Cattle milk	W	0.1 F T
		MM 0814	Goat meat	W	0.5 (fat) T
			Residue: flucythrinate (fat-soluble)		
Flusilazole	0.001	FS 0240	Apricot	0.5	-
(165)		FS 0245	Nectarine	0.5	0.1 T
		FS 0247	Peach	0.5	0.1 T
			Residue: flusilazole		
Folpet	0.01	FP 0226	Apple	W	10 T
(041)	T	FS 0013	Cherries	W	15 T
	(1995)	FC 0001	Citrus fruits	W	10 T
		VC 0424	Cucumber	W	2 T
		FB 0269	Grapes	2	25 T
		VL 0482	Lettuce, Head	W	15 T
		VC 0046	Melons, except Watermelon	W	2 T
		VA 0385	Onion, Bulb	W	2 T
		FB 0275	Strawberry	5	20 T
		VR 0589	Potato	0.02*	-
		VO 0448	Tomato	W	5 T

[2] New compound

[1] Re-evaluation in periodic review programme

			Residue: folpet		
			Note: Existing TADI extended until 1995		
Heptachlor (043)	0.0001	VR 0577	Carrots	W	0.2 E T
		VR 0596	Sugar beets	W	0.05 E
		VO 0448	Tomato	W	0.02 E T
			Vegetables	W	0.05 E T
			Residue: sum of heptachlor and heptachlor epoxide (fat-soluble)		
Hexaconazole (170)	0.005	GC 0654	Wheat	0.1	0.1 T
		AS 0654	Wheat straw and fodder, dry	0.5	0.5 T
			Residue: hexaconazole		
Mancozeb [1] (050)	0.03		Notes: 1. ADI is group ADI for EBDCs: see Dithiocarbamates. Previous ADI 0-0.05 mg/kg bw 2. See Dithiocarbamates for recommended MRLs		
Maneb [1] (Dithiocarbamates, 105)	0.03		Notes: 1. ADI is group ADI for EBDCs: see Dithiocarbamates. Previous ADI 0-0.05 mg/kg bw 2. See Dithiocarbamates for recommended MRLs		
Metiram [2] (186)	0.03		Note: ADI is group ADI for EBDCs: see Dithiocarbamates.		
Monocrotophos (054)	0.0006		Note: ADI increased from 0.00005 mg/kg bw		
Phorate (112)	0.0002	VR 0577	Carrot	0.2	0.5
			Residue: sum of phorate, its oxygen analogue, and their sulphoxides and sulphones, expressed as phorate		
Phosalone (060)	0.001		Note: ADI lowered from 0.006 mg/kg bw		
Procymidone (136)	0.1	FP 0226	Apple	W	5
		FS 0013	Cherries	10	5
		VP 0526	Common bean (pods and/or immature seeds)	1	2
		FB 0021	Currants, Black, Red, White	W	10
		VO 0440	Egg plant	W	2
		FB 0269	Grapes	5	5 T
		FI 0341	Kiwifruit	W	7
		VC 0046	Melons, except Watermelon	W	1
		VA 0385	Onion, Bulb	0.2	0.2
		VR 0589	Potato	W	0.1
		CM 0649	Rice, husked	W	3
		CM 1205	Rice, polished	W	1
		SO 0702	Sunflower seed	0.2	2
		OR 0702	Sunflower seed oil, edible	0.5	-
			Residue: procymidone		
Propineb [1]	0.007		Notes: 1. previous TADI of 0.005 mg/kg bw was withdrawn in		

[1] Re-evaluation in periodic review programme

[2] New compound

		1985 2. See Dithiocarbamates for recommended MRLs			
Propylenethiourea (PTU) (150)	0.0002 T (1999)	Note	As a TADI has now been allocated, Gls for propylenethiourea would normally be recom-mended as TMRLs, but the Meeting did not consider the Gls in this context		
Pyrazophos (153)	0.004	FP 0226	Apple	1	0.5
		AS 0640	Barley straw and fodder, dry	5	-
		VC 0424	Cucumber	0.1	0.1
		VC 0046	Melons, except Watermelon	0.1	0.1
		AS 0654	Wheat straw and fodder, dry	5	-
		Residue:	pyrazophos		
Triazophos (143)	0.001	FI 0327	Banana	W	1
		VR 0577	Carrot	0.5	0.1
		FC 0001	Citrus fruits	2 T	2
		VD 0541	Soya bean (dry)	0.05*	-
		FB 0275	Strawberry	0.05*	-
		Residue:	triazophos		
		Note	ADI increased from TADI of 0.0002 mg/kg bw. All previous TMRLs except that for Citrus fruits become MRLs		
Zineb [1] (Dithiocarbamates, 105)	0.03	Note	ADI is group ADI for EBDCs: see Dithiocarbamates. Previous ADI 0-0.05 mg/kg bw		

[1] Re-evaluation in periodic review programme

[1] Re-evaluation in periodic review programme

ANNEX II

PREVIOUS FAO AND WHO DOCUMENTS

1. FAO/WHO. 1962 — Principles governing consumer safety in relation to pesticide residues. Report of a meeting of a WHO Expert Committee on Pesticide Residues held jointly with the FAO Panel of Experts on the Use of Pesticides in Agriculture. FAO Plant Production and Protection Division Report, No. PL/1961/11; WHO Technical Report Series, No. 240.

2. FAO/WHO. 1964 — Evaluations of the toxicity of pesticide residues in food. Report of a Joint Meeting of the FAO Committee on Pesticides in Agriculture and the WHO Expert Committee on Pesticide Residues. FAO Meeting Report, No. PL/1963/13; WHO/Food Add./23.

3. FAO/WHO. 1965a — Evaluations of the toxicity of pesticide residues in food. Report of the Second Joint Meeting of the FAO Committee on Pesticides in Agriculture and the WHO Expert Committee on Pesticide Residues. FAO Meeting Report, No. PL/1965/10; WHO/Food Add./26.65.

4. FAO/WHO. 1965b — Evaluations of the toxicity of pesticide residues in food. FAO Meeting Report, No. PL/1965/10/1; WHO/Food Add./27.65.

5. FAO/WHO. 1965c — Evaluation of the hazards to consumers resulting from the use of fumigants in the protection of food. FAO Meeting Report, No. PL/1965/10/2; WHO/Food Add./28.65.

6. FAO/WHO. 1967a — Pesticide residues in food. Joint report of the FAO Working Party on Pesticide Residues and the WHO Expert Committee on Pesticide Residues. FAO Agricultural Studies, No. 73; WHO Technical Report Series, No. 370.

7. FAO/WHO. 1967b — Evaluation of some pesticide residues in food. FAO/PL:CP/15; WHO/Food Add./67.32.

8. FAO/WHO. 1968a — Pesticide residues. Report of the 1967 Joint Meeting of the FAO Working Party and the WHO Expert Committee. FAO Meeting Report, No. PL:1967/M/11; WHO Technical Report Series, No. 391.

9. FAO/WHO. 1968b — 1967 Evaluations of some pesticide residues in food. FAO/PL:1967/M/11/1; WHO/Food Add./68.30.

10. FAO/WHO. 1969a — Pesticide residues in food. Report of the 1968 Joint Meeting of the FAO Working Party of experts on Pesticide Residues and the WHO Expert Committee on Pesticide Residues. FAO Agricultural Studies, No. 78; WHO Technical Report Series, No. 417.

11. FAO/WHO. 1969b — 1968 Evaluation of some pesticide residues in food. FAO/PL:1968/M/9/1; WHO/Food Add./69.35.

12. FAO/WHO. 1970a Pesticide residues in food. Report of the 1969 Joint Meeting of the FAO Working Party of experts on Pesticide Residues and the WHO Expert Committee on Pesticide Residues. FAO Agricultural Studies, No. 84; WHO Technical Report Series, No. 458.

13. FAO/WHO. 1970b 1969 Evaluation of some pesticide residues in food. FAO/PL:1969/M/17/1; WHO/Food Add./70.38

14. FAO/WHO. 1971a Pesticide residues in food. Report of the 1970 Joint Meeting of the FAO Working Party of experts on Pesticide Residues and the WHO Expert Committee on Pesticide Residues. FAO Agricultural Studies, No. 87; WHO Technical Report Series, No. 474.

15. FAO/WHO. 1971b 1970 Evaluation of some pesticide residues in food. AGP:1970/M/12/1; WHO/Food Add./71.42.

16. FAO/WHO. 1972a Pesticide residues in food. Report of the 1971 Joint Meeting of the FAO Working Party of experts on Pesticide Residues and the WHO Expert Committee on Pesticide Residues. FAO Agricultural Studies, No. 88; WHO Technical Report Series, No. 502.

17. FAO/WHO. 1972b 1971 Evaluation of some pesticide residues in food. AGP:1971/M/9/1; WHO Pesticide Residues Series, No. 1.

18. FAO/WHO. 1973a Pesticide residues in food. Report of the 1972 Joint Meeting of the FAO Working Party of experts on Pesticide Residues and the WHO Expert Committee on Pesticide Residues. FAO Agricultural Studies, No. 90; WHO Technical Report Series, No. 525.

19. FAO/WHO. 1973b 1972 Evaluation of some pesticide residues in food. AGP:1972/M/9/1; WHO Pesticide Residues Series, No. 2.

20. FAO/WHO. 1974a Pesticide residues in food. Report of the 1973 Joint Meeting of the FAO Working Party of experts on Pesticide Residues and the WHO Expert Committee on Pesticide Residues. FAO Agricultural Studies, No. 92; WHO Technical Report Series, No. 545.

21. FAO/WHO. 1974b 1973 Evaluation of some pesticide residues in food. FAO/AGP/1973/M/9/1; WHO Pesticide Residues Series, No.3.

22. FAO/WHO. 1975a Pesticide residues in food. Report of the 1974 Joint Meeting of the FAO Working Party of experts on Pesticide Residues and the WHO Expert Committee on Pesticide Residues. FAO Agricultural Studies, No. 97; WHO Technical Report Series, No. 574.

23. FAO/WHO. 1975b 1974 Evaluation of some pesticide residues in food. FAO/AGP/1974/M/9/11; WHO Pesticide Residues Series, No.4.

24. FAO/WHO. 1976a Pesticide residues in food. Report of the 1975 Joint Meeting of the FAO Working Party of experts on Pesticide Residues and the WHO Expert Committee on Pesticide Residues. FAO Plant Production and Protection Series, No.1; WHO Technical Report Series, No. 592.

25. FAO/WHO. 1976b 1975 Evaluation of some pesticide residues in food. AGP:1975/M/13; WHO Pesticide Residues Series, No. 5.

26. FAO/WHO. 1977a Pesticide residues in food. Report of the 1976 Joint Meeting of the FAO Panel of Experts on Pesticide Residues and the Environment and the WHO Expert Group on Pesticide Residues. FAO Food and Nutrition Series, No. 9; FAO Plant Production and Protection Series, No. 8; WHO Technical Report Series, No. 612.

27. FAO/WHO. 1977b 1976 Evaluation of some pesticide residues in food. AGP:1976/M/14.

28. FAO/WHO. 1978a Pesticide residues in food - 1977. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 10 Rev.

29. FAO/WHO. 1978b Pesticide residues in food: 1977 evaluations. FAO Plant Production and Protection Paper 10 Sup.

30. FAO/WHO. 1979a Pesticide residues in food - 1978. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 15.

31. FAO/WHO. 1979b Pesticide residues in food: 1978 evaluations. FAO Plant Production and Protection Paper 15 Sup.

32. FAO/WHO. 1980a Pesticide residues in food - 1979. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 20.

33. FAO/WHO. 1980b Pesticide residues in food: 1979 evaluations. FAO Plant Production and Protection Paper 20 Sup.

34. FAO/WHO. 1981a Pesticide residues in food - 1980. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 26.

35. FAO/WHO. 1981b Pesticide residues in food: 1980 evaluations. FAO Plant Production and Protection Paper 26 Sup.

36. FAO/WHO. 1982a Pesticide residues in food - 1981. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 37.

37. FAO/WHO. 1982b Pesticide residues in food: 1981 evaluations. FAO Plant Production and Protection Paper 42.

38. FAO/WHO. 1983a Pesticide residues in food - 1982. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 46.

39. FAO/WHO. Pesticide residues in food: 1982 evaluations.

1983b FAO Plant Production and Protection Paper 49.

40. FAO/WHO. 1984 Pesticide residues in food - 1983. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 56.

41. FAO/WHO. 1985a Pesticide residues in food: 1983 evaluations. FAO Plant Production and Protection Paper 61.

42. FAO/WHO. 1985b Pesticide residues in food - 1984. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 62.

43. FAO/WHO. 1985c Pesticide residues in food: 1984 evaluations. FAO Plant Production and Protection Paper 67.

44. FAO/WHO. 1986a Pesticide residues in food - 1985. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 68.

45. FAO/WHO. 1986b Pesticide residues in food: 1985 evaluations. Part I - Residues. FAO Plant Production and Protection Paper 72/1.

46. FAO/WHO. 1986c Pesticide residues in food: 1985 evaluations. Part II - Toxicology. FAO Plant Production and Protection Paper 72/2.

47. FAO/WHO. 1986d Pesticide residues in food - 1986. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 77.

48. FAO/WHO. 1986e Pesticide residues in food: 1986 evaluations. Part I - Residues. FAO Plant Production and Protection Paper 78.

49. FAO/WHO. 1987a Pesticide residues in food: 1986 evaluations. Part II - Toxicology. FAO Plant Production and Protection Paper 78/2.

50. FAO/WHO. 1987b Pesticide residues in food - 1987. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 84.

51. FAO/WHO. 1988a Pesticide residues in food: 1987 evaluations. Part I - Residues. FAO Plant Production and Protection Paper 86/1.

52. FAO/WHO. 1988b Pesticide residues in food: 1987 evaluations. Part II - Toxicology. FAO Plant Production and Protection Paper 86/2.

53. FAO/WHO. 1988c Pesticide residues in food - 1988. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 92.

54. FAO/WHO. 1988d Pesticide residues in food: 1988 evaluations. Part I - Residues. FAO Plant Production and Protection Paper 93/1.

55. FAO/WHO. 1989a Pesticide residues in food: 1988 evaluations. Part II - Toxicology. FAO Plant Production and Protection Paper 93/2.

56. FAO/WHO. 1989b Pesticide residues in food - 1989. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 99.

57. FAO/WHO. 1990a Pesticide residues in food: 1989 evaluations. Part I - Residues. FAO Plant Production and Protection Paper 100.

58. FAO/WHO. 1990b Pesticide residues in food: 1989 evaluations. Part II - Toxicology. FAO Plant Production and Protection Paper 100/2.

59. FAO/WHO. 1990c Pesticide residues in food - 1990. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 102.

60. FAO/WHO. 1991a Pesticide residues in food: 1990 evaluations. Part I - Residues. FAO Plant Production and Protection Paper 103/1.

61. FAO/WHO. 1991b Pesticide residues in food: 1990 evaluations - Toxicology. WHO/PCS/91.47.

62. FAO/WHO. 1991c Pesticide residues in food - 1991. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 111.

63. FAO/WHO. 1991d Pesticide residues in food: 1991 evaluations. Part I - Residues. FAO Plant Production and Protection Paper 113/1.

64. FAO/WHO. 1992 Pesticide residues in food: 1991 evaluations - Part II - Toxicology. WHO/PCS/92.52.

65. FAO/WHO. 1993a Pesticide residues in food - 1992. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 116.

66. FAO/WHO. 1993b Pesticide residues in food: 1992 evaluations. Part I - Residues. FAO Plant Production and Protection Paper 118.

67. FAO/WHO. 1993c Pesticide residues in food: 1992 evaluations - Part II - Toxicology. WHO/PCS/93.34.

68. FAO/WHO. 1993d Pesticide residues in food - 1993. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 122.

69. FAO/WHO. Pesticide residues in food - 1993. Toxicology evaluations

1994a WHO/PCS/94.4.