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Pesticide residues in food 1997

Joint FAO/WHO Meeting on Pesticide Residues

EVALUATIONS

1997

PART I - RESIDUES

Rome, 1998

CONTENTS

* First evaluation
** Evaluation in CCPR periodic review programme

1997 JOINT MEETING OF THE FAO PANEL OF EXPERTS ON PESTICIDE RESIDUES IN FOOD AND THE ENVIRONMENT AND THE WHO CORE ASSESSMENT GROUP

Lyon (IARC), 22 September - 1 October 1997

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ABBREVIATIONS WHICH MAY BE USED

(Well-known abbreviations in general use are not included)

Ache	acetylcholinesterase
ADI	acceptable daily intake
AFI(D)	alkali flame-ionization (detector)
Ai	active ingredient
ALAT	alanine aminotransferase
AR	applied radioactivity
ASAT	aspartate aminotransferase
	BBABiologische Bundesanstalt für Land- und Forstwirtschaft
Bw	body weight
BOD	biological oxygen demand
CA	Chemical Abstracts
CAS	Chemical Abstracts Services
CCN	Codex Classification Number (this may refer to classification numbers for compounds or for commodities).
CCPR	Codex Committee on Pesticide Residues
CCRVDF	Codex Committee on Residue of Veterinary Drugs in Food
ChE	cholinesterase
CI	chemical ionization
CNS	central nervous system
Cv	coefficient of variation
CXL	Codex Maximum Residue Limit (Codex MRL). See MRL.
DFG	Deutsche Forschungsgemeinschaft
DL	racemic (optical configuration, a mixture of dextro- and laevo-)
DP	dustable powder
DS	powder for dry seed treatment
DT-50	time for 50% decomposition (i.e. half-life)
DT-90	time for 90% decomposition
EBDC	ethylenebis(dithiocarbamate)
EC	(1) emulsifiable concentrate
	(2) electron-capture [chromatographic detector]
ECD	electron-capture detector
EI	electron-impact
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
FP(D)	flame-photometric (detector)

g (not gm)	gram
µg	microgram
GAP	good agricultural practice(s)
GC-MS	gas chromatography - mass spectrometry
GC-MSD	gas chromatography with mass-selective detection
G.I.	gastrointestinal
GL	guideline level
GLC	gas-liquid chromatography
GLP	Good Laboratory Practice
GPC	gel-permeation chromatograph or chromatography
GSH	glutathione
h (not hr)	hour(s)
ha	hectare
Hb	haemoglobin
Hl	hectolitre
HPLC	high-performance liquid chromatography
HPLC-MS	high-performance liquid chromatography - mass spectrometry
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 the WHO Core Assessment Group
LC	liquid chromatography
LC_{50}	lethal concentration, 50%
LC-MS	liquid chromatography - mass spectrometry
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
MFO	mixed function oxidase
µm	micrometre (micron)
min (no stop)	minute(s)
MLD	minimum lethal dose
M	molar
mo (not mth.)	month(s)
MRL	Maximum Residue Limit. 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.
MS	mass spectrometry
MSD	mass-selective detection

MTD	maximum tolerated dose
n (not *n*)	normal (defining isomeric configuration)
NCI	National Cancer Institute (USA)
NMR	nuclear magnetic resonance
NOAEL	no-observed-adverse-effect level
NOEL	no-observed-effect level
NP(D)	nitrogen-phosphorus (detector)
NTE	neuropathy target esterase
OECD	Organization for Economic Co-operation and Development
OP	organophosphorus pesticide
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/l are used).
PT	prothrombin time
PTDI	provisional tolerable daily intake. (See 1994 report, Section 2.3, for explanation)
PTT	partial thromboplastin time
PTU	propylenethiourea
RBC	red blood cell
r.d.	relative density. (Formerly called specific gravity)
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)
SPE	solid-phase extraction
STMR	supervised trials median residue
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
WG	water-dispersible granule

WHO	World Health Organization
WP	wettable powder
<	less than
≤	less than or equal to
>	greater than
≥	greater than or equal to

USE OF JMPR REPORTS AND EVALUATIONS BY REGISTRATION AUTHORITIES

The summaries and evaluations contained in this book are, in most cases, based on unpublished proprietary data submitted for the purpose of the JMPR assessment. A registration authority should not grant a registration on the basis of an evaluation unless it has first received authorization for such use from the owner who submitted the data for JMPR review or has received the data on which the summaries are based, either from the owner of the data or from a second party that has obtained permission from the owner of the data for this purpose.

INTRODUCTION

The report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Core Assessment Group (JMPR), held in Lyon, 22 September-1 October 1997, 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 containes 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 reisude and toxicological considerations will be available. Speciail attention is drawn to Annex I containing updated ADIs, MRLs, and STMR levels which also appears in full as part of the report of the Meeting.

Some of the compounds considered at the 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 CCPR a 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 double asterisks 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.

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

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{PRIVATE }4.10 2,4-D AND ITS SALTS AND ESTERS{TC \l 2 "4.10 2,4-D AND ITS SALTS AND ESTERS"}

EVALUATION OF EFFECTS ON THE ENVIRONMENT

2,4-D, 2,4-dichlorophenoxyacetic acid, is a selective herbicide available as the free acid, salts and esters. 2,4-D has low volatility and is not expected to be lost by evaporation after application. Amine salt formulations of 2,4-D are less volatile than butyl, ethyl, or isopropyl ester formulations.

The 2-ethylhexyl (EH) ester is hydrolysed under alkaline conditions (half-life 48 days at pH 7 and 2.2 days at pH 9). 2,4-D may be degraded slowly by photolysis. The half-life of 2,4-D in aqueous solution was 4.5 days under aerobic conditions and 312 days under anaerobic conditions. The major breakdown product was CO_2, with 2,4-dichlorophenol, 2,4-dichloroanisole, and 4-chlorophenoxyacetic acid also being formed as intermediates. The half-lives of 2,4-D determined in natural waters after aerial application of its dimethylamine (DMA) salt ranged from 1.1 to 20 days. 2,4-D formulations were found to be rapidly hydrolysed or biodegraded in ponds and lakes.

There was no evidence of bioaccumulation of 2,4-D in aquatic organisms.

The behaviour of 2,4-D salts and esters in soils is greatly influenced by the organic matter content and pH. 2,4-D is more strongly adsorbed in soils with higher organic matter content and/or lower pH. The rapid biodegradation of 2,4-D in soil prevents significant downward movement under normal field conditions. Run-off from treated soil has been estimated at between 0.01 and 1% of the applied 2,4-D; the maximum recorded concentrations following run-off were about 0.2 ìg/l. Under non-sterile conditions various esters of 2,4-D are hydrolysed very rapidly in soils (>72% within 72 h). A number of microbial organisms rapidly degrade 2,4-D, which has half-lives of 1.25 h to 40 days, usually between 3 and 10 days. The DMA salt rapidly dissociates, leaving 2,4-D which then undergoes further degradation.

Field trials in the USA using the DMA salt or the EH ester at 2.24 kg acid-equivalent/ha on grass resulted in maximum initial residues at day 0 of 120 or 153 mg acid-equivalent/kg, respectively. These initial residues decreased by a half to a third by day 7.

In general, populations of aerobic bacteria, actinomycetes, and fungi in soils were not affected by 2,4-D at 25 ppm. At an application rate of 0.95 kg/ha, populations of bacteria, fungi, and actinomycetes were reduced by 26.3%, 19.5% and 30%, respectively, by the iso-octyl (IO) ester, but approximately half as much by the DMA salt.

2,4-D at the maximum recommended application rate has a growth-stimulating effect (10%) on *Skeletonema costatum*, whereas it inhibits the growth of *Navicula pelliculosa,* (24%) and *Lemna gibba* (75%). The 5-day EC_{50} for the acute toxicity of 2,4-D and its salts and esters differs widely among algal species and compounds, ranging from 0.23 mg ai/l (EH ester for *Skeletonema costatum*) to 153 mg ai/l (DMA salt for *Anabaena flosaquae*). The acute toxicity of 2,4-D to the aquatic higher plant *Lemna gibba* also depended upon the salt or ester used, with 14-day EC_{50} values of 3.3, 0.58 and 0.5 mg/l for 2,4-D, the DMA salt and the EH ester respectively.

At concentrations ranging from 0.001 to 100 mg/l, 2,4-D had no effect on chlorophyll production in several algal species. *Anabaenopsis raciborskii* was found to tolerate up to 800 ìg/ml of 2,4-D in liquid culture media.

Many studies have been performed on invertebrate fresh water and estuarine or marine species, including *Daphnia*, *Gammarus*, *Macrobranchium*, *Crassostrea*, *Palaemonetes*, *Panaeus,* and *Uca*. Many forms of 2,4-D have been evaluated: 2,4-D itself, the DMA, diethanolamine (DEA), isopropylamine (IPA), tri-isopropanolamine (TIPA), and sodium salts, and the EH, butoxyethyl (BE), and isopropyl (IP) esters. 2,4-D and its salts are generally less toxic to these organisms than the ester forms. Acute toxicity (48-h LC_{50} values) to *Daphnia magna* ranged from 5.2 mg ai/l (IO ester) to 184 mg ai/l (DMA salt) and 21 day NOECs ranged from 0.0015 mg ai/l (EH ester) to 27.5 mg ai/l (DMA salt) and 79 mg ai/l (acid).

At concentrations of 1.0 and 10.0 mg BE ester/l, grass shrimp were observed to avoid water containing these levels. In a life-cycle study on *Daphnia magna*, the NOEC was 23.6 mg ai DEA salt/l. In a chronic toxicity study on *Daphnia magna* using the BE ester, the Maximum Acceptable Toxicant Concentration (MATC) ranged between 0.70 mg/l and 0.29 mg/l.

Frog and toad tadpole 96-hour LC_{50} values ranged from 8 mg/l for the free acid to 477 mg/l for the DMA salt.

Many data were available on the effects of 2,4-D and its salts and esters on various growth stages of fish such as *Oncorhynchus*, *Lepomis*, *Pimephales*, *Gambusia*, *Micropterus*, and *Salm*o. Generally, 2,4-D and its salts are less toxic to fish than are the esters. Typical 96-h LC_{50} values for adult fish ranged from 5 to 10 mg a.i./l for the IO ester, from 200 to 400 mg a.i./l for 2,4-D, and from 250 to 500 mg a.i./l for the DEA salt, although lower figures have been reported. Early life stages appear to be more sensitive, with 32-day NOECs ranging from 0.12 mg a.i./l (EH ester) to 17.1 mg a.i./l (DEA salt) and 63.4 mg a.i./l (acid).

Embryos and larvae of the fathead minnow (*Pimephales promelas*), were exposed to up to 416.1 ìg/l of the BE ester for 32 days. The NOEC was 80.5 ìg/l, with the MATC estimated to be 96.0 ìg/l. The sodium salt of 2,4-D had no inhibitory effect on the hatching of carp eggs at 25 mg/l, but at a concentration of 100 mg/l none hatched. At 50 mg a.i./l the sodium salt was not harmful to carp embryos but induced behavioural changes, some morphopathological changes, and ultimately death in carp larvae.

Honeybee oral and contact LD_{50} values for the DMA salt and EH ester were all >100 ìg/bee. Toxic effects have not been noted for bees in the field.

2,4-D (in combination with MCPA) did not harm *Trichogramma cacoeciae* at 1.5% in water or *Aleochara bilineata* at the label recommended rate. 2,4-D mixed amine salts and mixed isopropyl esters were toxic to coccinellid larvae and to sawflies. No reproductive effects were observed in European cockroaches reared on food containing 1000 mg/kg of 2,4-D (unspecified).

Application of 2,4-D at 1250 g a.i./l in field crops did not affect staphylinids, carabids, or spiders during a 20-month observation period. Mortality of adult millipedes exposed to 2,4-D at a rate of 33.6 kg/ha was noted on the first day and exceeded 50% of control mortality by day 7.

The 14-day LC_{50} for earthworms exposed to the DMA salt was 350 mg/kg soil, with no mortality noted at concentrations less than or equal to 100 mg a.i./kg. A 48-hour LC_{50} of 61.6 ìg/cm^2 has been reported for earthworms exposed on filter paper.

Acute avian LD_{50} values range from 200 to >2000 mg/kg bw for mallards, bobwhite quail, Japanese quail, pheasants, chukar partridges, and rock doves. Dietary LC_{50} values exceed 4640 mg/kg diet for mallards, bobwhite quail, Japanese quail, and pheasants for the acid, salt, and ester. At doses greater than the recommended application rate, the acid, salt, and ester did not adversely affect the reproductive performance of pheasants, quail, partridges, or chickens.

Oral LD_{50} values for rats and rabbits range from 699 to 2322 mg/kg bw for the acid and its salts and esters. Dermal LD_{50} values for the rabbit exceed 2000 mg/kg bw and inhalation LC_{50} values range from 1.8 to 10.7 mg/l.

Risk assessment

The information on use and application rates used for this risk assessment is derived from the agricultural use of 2,4-D within the European Union and the USA. It should be noted that 2,4-D can be formulated as a variety of different salts (e.g. DMA, sodium, DEA, TIPA, and IPA) and esters (EH, IO, and BE). However of all these different forms, the DMA salt and EH ester account for approximately 95% of the global use of 2,4-D. This risk assessment is therefore restricted to the use of these compounds. It should be noted that both of them are rapidly hydrolysed to 2,4-D. The major uses of 2,4-D include application to cereals, corn, sorghum, soya beans, sugar cane, rice, pasture, top fruit, turf, non-cropland, fallow, forestry, and aquatic weeds. Applications can be made by either conventional tractor-mounted or drawn hydraulic sprayers or by aerial application (e.g. in forestry use) at rates varying from 0.25-4.48 kg acid-equivalent/ha.

This risk assessment is based on the principle of calculating Toxicity:Exposure Ratios (TERs) and follows the EPPO/CoE Environmental Risk Assessment models and trigger values.

Aquatic environment

The main risk to aquatic organisms from the use of 2,4-D is from overspray during aerial use, spray drift from ground-based hydraulic applications, or use to control aquatic weeds.

The EPPO/CoE risk assessment scheme for aquatic organisms showed a low acute risk (TERs >10) to fish, aquatic invertebrates, and algae from both spray drift contamination arising from ground-based hydraulic applications and from overspray contamination arising from aerial applications. A potential acute risk (TERs <10) to both aquatic higher plants and amphibians from overspray contamination during aerial applications was identified. The use of 2,4-D to control aquatic weeds also presented a potential acute risk (TERs <10) to algae as well as amphibians and aquatic higher plants. However, the risk to algae and aquatic higher plants can be ignored as these organisms are the targets when 2,4-D is used in this way. A potential acute risk to amphibians still remains from the use for aquatic weed control. It should be noted, however, that such a risk to amphibians needs to be balanced against the risks associated with alternative aquatic weed control practices such as not conducting weed control (e.g. algal bloom leading to water deoxygenation) or the potential damage caused by manual weed control, both of which may pose a higher risk to fish and other aquatic organisms. The EH ester is not recommended for aquatic weed control.

Owing to the very rapid degradation of the salts and esters of 2,4-D in water, the long-term risk to aquatic organisms from these compounds was considered to be low. However the primary breakdown product, 2,4-D acid, is more persistent in water and therefore the long-term risk assessment is based on it. Measured levels of 2,4-D in surface waters associated with approved uses (ranging from 0.00008 mg/l in small watersheds in Saskatchewan to 0.0021 mg/l in ground and surface waters in the UK) indicate that the long-term risk to fish and water-column- and sediment-dwelling invertebrates is low (TERs >10).

Terrestrial environment

Micro-organisms

The most significant routes of exposure of soil micro-organisms to 2,4-D are likely to be from its use by ground or aerial applications. Data from laboratory studies indicate that the risk to soil micro-organisms from the use of 2,4-D should be low at application rates of 7.5 and 18.75 kg 2,4-D/ha, which are higher than the maximum recommended application rates. In another study, application of the DMA salt and the IO ester at rates corresponding to 0.95 kg 2,4-D/ha resulted in 10-30% reductions in populations of soil bacteria, fungi, and actinomyces, with the ester producing greater reductions. As the trigger for concern in the CoE/EPPO micro-organism risk assessment scheme is an effect of >30%, it can be concluded that the risk to soil micro-organisms from the use of 2,4-D should be low.

Plants

2,4-D is a translocated selective herbicide that is used to control a variety of broad-leaved weeds. Consequently, although 2,4-D may pose a risk to broad-leaved non-target plants, this is to be expected from its mode of action and consequent use.

Invertebrates

Bees

Bees may be exposed to 2,4-D by foraging flowering weeds present in treated crops. At the maximum individual application rate of 4.48 kg acid-equivalent/ha, the hazard quotients for both contact and oral toxicity were >45 for both the DMA salt and the EH ester. As the EPPO/CoE trigger for concern is a hazard quotient of >50, the acute risk to honeybees from the use of 2,4-D at this application rate should be low. This is supported by the fact that 2,4-D has never been implicated in any honeybee poisoning incidents in the UK Wildlife Incident Investigation Scheme (WIIS).

Other arthropods

Arthropods may be exposed to 2,4-D from its many agricultural and non-agricultural uses. On the basis of the EPPO/CoE triggers for concern with regard to effects on non-target arthropods in laboratory studies (effects >30%), 2,4-D may pose a risk to arthropods at high application rates, but the laboratory data were either generated with a joint formulation with MCPA, or were old and may be unreliable. Limited field data at the lower and more typical range of application rates (up to 1.25 kg/ha) indicate that this risk may not be realized in the field.

Earthworms

Earthworms may be exposed from either single or multiple applications of 2,4-D to a wide variety of crops but in particular from its use on grass, fallowland, and stubble. The TER from a maximum application rate of 5.37 kg DMA salt/ha is above the EPPO/CoE trigger value of 10, which indicates that the acute risk to earthworms from the use of 2,4-D should be low.

Vertebrates

Vertebrates are likely to be exposed to 2,4-D either from grazing on treated or contaminated vegetation or consuming contaminated insects. For this risk assessment the estimation of residues on food items represents the maximum value determined immediately after application and does not take into account the rapid degradation in the environment. It further assumes that all food consumed contains 2,4-D at the level of the MRL.

Birds

The short-term dietary TERs based on measured initial residues on short grass arising from the application of 2.24 kg acid-equivalent/ha, indicate a potential medium risk (10 <TER <100) to grazing birds from both aerial and ground-based applications. The initial residues declined to a half or a third by 7 days after application. It should be noted that 2,4-D has never been implicated in any bird-poisoning incidents as a result of normal use. This suggests that the risk to grazing birds from 2,4-D is unlikely to be high. The short-term dietary TERs based on initial residues on large insects predicted by the EPPO/CoE vertebrate risk assessment scheme indicate a low acute risk (TER >100) to small insectivorous birds from both aerial and ground applications (4.48 kg acid-equivalent/ha and 2.24 kg acid-equivalent/ha respectively). Large insects are likely to constitute a higher proportion of both bird and mammalian diets than small insects during early growth stage or pre-emergence use.

Mammals

The acute oral TERs based on measured initial residues on short grass arising from application at 2.24 kg acid-equivalent/ha indicate a potential high risk (TER <10) to grazing mammals from both aerial and ground-based applications, but the initial residues declined to a half or a third by 7 days after application. The acute oral TERs based on predicted initial residues on large insects however indicate a medium acute risk (10 <TER <100) from aerial applications, and a low acute risk (TER >100) from ground-based applications, to small insectivorous mammals. It should be noted however that 2,4-D has never been implicated in any mammal-poisoning incidents as a result of normal use. This suggests that the risk to mammals from 2,4-D is unlikely to be high.

ABAMECTIN (177)

EXPLANATION

Abamectin was evaluated in 1992 and 1994 and MRLs were recommended for a number of crops and animal commodities.

The 28th (1996) Session of the CCPR was informed that the LOD of 0.01 mg/kg for abamectin might need to be increased to 0.02 mg/kg (ALINORM 97/24, para 77). The 29th 1997 Session (ALINORM 97/24A, para 105) agreed, on the advice of the *ad hoc* Working Group on Methods of Analysis, to maintain MRLs for abamectin set at or about the LOD at 0.01 mg/kg.

The 29th Session also noted (ALINORM 97/24A, para 89) that the CCRVDF had proposed MRLs for abamectin in animal products and encouraged comment on these proposals.

The Meeting received information on the current registered or approved uses of abamectin on food crops. The Netherlands provided a copy of the official method of analysis for abamectin residues.

The Meeting received information on methods of analysis and residue data from supervised trials on the additional crops apples, potatoes and hops as well as on pears, cucurbits, lettuce and tomatoes. Processing data were available for apples, pears, potatoes and hops.

The predominant residues from the use of abamectin on cropos are avermectin B_{1a}, avermectin B_{1b} and the photoesomers 8,9-*Z*-avermectin B_1 (B_{1a} and B_{1b}).

METHODS OF RESIDUE ANALYSIS

Analytical methods

Abamectin is a mixture of avermectin B_{1a} (80%) and avermectin B_{1b} (20%). In sunlight the photoisomer 8,9-*Z*-avermectin is produced and becomes part of the residue. It is also described as the D-8,9 isomer. Avermectin B_{1a} and 8,9-*Z* avermectin B_{1a} produce the same fluorescent compound in the derivatization step of the analytical methods and hence a single peak on an HPLC chromatogram. Avermectin B_{1b} and its photoisomer 8,9-*Z*-avermectin B_{1b} behave in the same way and appear together in a second peak in the chromatogram.

Analytical methods that measure the components of the residue involve the HPLC separation and fluorescence detection of derivatives formed by converting the cyclohexene ring to an aromatic ring. Analytical methods for abamectin residues in crops, soil, animal tissues, milk and water were reviewed by the 1992 JMPR.

The newer analytical methods rely on a rapid derivatization. The avermectin compounds dissolved in an acetonitrile/triethylamine mixture in the presence of 1-methylimidazole react rapidly with trifluoroacetic anhydride at room temperature to produce the fluorescent derivative. Approximately 3 minutes are required for the reaction as compared with 1 hour in previous methods. Some clean-up steps have also been streamlined.

Cobin (1989), in Method 8920, extracted abamectin residues from cucumbers with methanol. The aqueous methanol extract was washed with iso-octane and passed through a C-8 column that captured the abamectin residues. The C-8 column was connected to 2 small aminopropyl columns in series and the abamectin rinsed through with methanol. A portion of the extract was evaporated and the abamectin was derivatized and determined by HPLC with fluorescence detection. Recoveries were satisfactory, but recoveries of 8,9-*Z*-avermectin were mostly near 70%, which was lower than those of avermectin B_{1a} and B_{1b}.

Trainor (1991) drew attention to the losses of 8,9-*Z*-avermectin B_{1a} which may occur in emulsions during solvent partition steps. Shaking must be done gently to keep the emulsion layer to a minimum.

Hicks (1992a,b) described Method 8000 for abamectin residues in apples and pears. Samples were first treated with pectinase to hydrolyse the pectin. Abamectin residues were extracted from the apple or pear homogenate with acetonitrile/water, and the extract further diluted with water and loaded onto a small C-8 column. Abamectin was eluted from the column with acetonitrile, which was diluted with water and the abamectin partitioned into hexane. The hexane solution was further cleaned up on an aminopropyl column to produce an extract ready for derivatization and HPLC determination. The LOD for each component of the residue in both apples and pears was 0.002 mg/kg. Good recoveries from spiked samples of apples and pears were obtained for avermectin B_{1a} at 0.0019-0.079 mg/kg, avermectin B_{1b} at 0.0038-0.0059 mg/kg and 8,9-Z-avermectin B_{1a} at 0.0046-0.070 mg/kg.

Cobin (1995) described the method (M-007.1) used for analysing apple samples from the supervised residue trials in Europe. Partially thawed whole apples were chopped in a Hobart food processor with dry ice added to achieve better chopping and to keep samples partially frozen. A portion of the chopped homogenate was then blended with acetonitrile, water and hexane. Abamectin residues were extracted into the hexane phase, which after drying with sodium sulfate was introduced into 2 small aminopropyl columns in series. The columns were washed with hexane, toluene and then dichloromethane. The abamectin residues were eluted from the column with acetone/dichloromethane, derivatized and determined by HPLC as described in other methods.

Macdonald *et al.* (1994) validated Method M-007 for apples. Good recoveries of avermectin B_{1a} were obtained from apples fortified at 0.002, 0.010 and 0.030 mg/kg.

The method (91-1) used for the determination of abamectin in tomatoes was very similar to the later method M-007.1 used for apples (Prabhu, 1991c). Maudsley and Clements (1994) validated method 91-1 for residues in lettuce. Good recoveries were obtained from lettuce fortified with avermectin B_{1a} at 0.002, 0.010, 0.030 and 2.00 mg/kg and with avermectin B_{1b} at 0.002, 0.010 and 0.10 mg/kg.

Method 936-92-4 used for the determination of abamectin residues in potatoes was described by Wehner (1992). Residues were extracted with methanol and the extract, after the addition of water, was passed through a small C-8 column which retained the abamectin residue. Further clean-up was achieved by washing the residue from the C-8 column directly through an aminopropyl column with a small volume of methanol. Part of the methanol solution was concentrated to dryness and the fluorescent derivatives were formed by reaction with a mixture of trifluoroacetic anhydride and 1-methylimidazole in acetonitrile. The LOD for each component of the residue was 0.005 mg/kg. Kvaternick (1993g) validated the method for fortified raw potatoes and achieved good recoveries for avermectin B_{1a} at 0.005-0.100 mg/kg, avermectin B_{1b} at 0.0049 mg/kg and 8,9-*Z*-avermectin B_{1a} at 0.005-0.050 mg/kg.

Morneweck (1992) described Method 92-1 for residues of abamectin in apple pomace, apple juice and apple sauce. Abamectin residues were extracted from wet or dry apple pomace with a hexane/water/acetonitrile mixture. Apple juice and apple sauce were extracted with acetonitrile/water and the extracts passed through a C-8 column, which was washed with acetonitrile to recover the abamectin residues. The extracts of all the commodities were then cleaned up on an aminopropyl column. The procedure then followed that of Method 8000 described above.

Recoveries of 8,9-*Z*-avermectin B_{1a} were consistently near 70% from the various commodities, but those of avermectin B_{1a} and B_{1b} were generally higher.

Johnson (1994a) described Method M-036 for abamectin in dried hops. The hops were rehydrated and extracted with a methanol-water mixture. Clean-up was effected by extraction into hexane and passage of the hexane extract through an aminopropyl solid phase extraction column. The abamectin in the cleaned up extract was derivatized with trifluoroacetic anhydride and determined by HPLC in the normal way. The LODs for avermectin B_{1a}, B_{1b} and 8,9-*Z*-avermectin B_{1a} in spiked dried hops were all 0.005 mg/kg. Method M-044 for abamectin residues in fresh hops is essentially the same as M-036 (Johnson, 1994b).

Duchene *et al.* (1997) validated analytical methods M-036.2 and M-044 for abamectin residues in dried hops, fresh hops and immature hops. Good recoveries from spiked samples were obtained for avermectin B_{1a} (0.0025-0.100 mg/kg), avermectin B_{1b} (0.005 mg/kg) and 8,9-*Z*-avermectin B_{1a} (0.005-0.100 mg/kg). The validated LOD for the 3 components in dried, fresh and immature hops was 0.005 mg/kg.

Many recovery experiments were carried out in the course of method validation and during supervised trials and processing studies. The median and mean recoveries were respectively 87% and 88% for avermectin B_{1a} (n=523), 90% and 89% for avermectin B_{1b} (n=100), and 84% and 83% for 8,9-*Z*-avermectin B_{1a} (n=166).

Some of the analytical methods developed to determine abamectin residues in various substrates which were originally in the form of unpublished reports have now been published in the scientific literature. Prabhu *et al.* (1992) described a rapid method which was used for the analysis of tomatoes in field trials. Samples can be prepared for HPLC analysis in batches of 12 in about 4-6 hours.

Cobin and Johnson (1995) have published a modified version of the method described above for abamectin in apples, and have published the residue method for hops (Cobin and Johnson,1996).

In the official method in The Netherlands (Netherlands, 1996) abamectin residues are extracted with ethyl acetate from a portion of the chopped sample. The residue, after evaporation of the ethyl acetate, is taken up in hexane and loaded on to a small aminopropyl column for clean-up. The column is washed with hexane, toluene and dichloromethane and the washings discarded. Abamectin residues are then eluted with dichloromethane/acetone. After the solvent has been evaporated the residue is taken up in methanol for HPLC analysis with UV detection at 245 nm. Good recoveries were obtained for avermectin B_{1a} in fortified cucumber samples at 0.0135 and 0.0675 mg/kg. The limit of determination was 0.003 mg/kg. The recovery of 8,9-*Z*-avermectin B_{1a} was not determined.

Stability of pesticide residues in stored analytical samples

Information on the stability of abamectin residues in dried hops during frozen storage was provided to the Meeting. Studies on the frozen storage stability of abamectin in numerous crops were reviewed by the 1992 JMPR.

Arenas (1997a) tested the stability of avermectin B_{1a} and 8,9-*Z*-avermectin B_{1a} in spiked dried hops stored below -10°C for 6 months. Dried hop cones without detectable abamectin residues were pulverised, weighed in 5 g lots into small bottles, and spiked at 0.020 mg/kg with the avermectin test materials (Table 1). The freezer temperature was always below -10°C and the average was about -20°C. Samples were analysed by method M-036.

The compounds were stable for the 6 months, with perhaps a slow loss estimated at about 3% per month.

Arenas (1997b) used the same method to test the stability of abamectin in fresh hops during frozen storage between -20°C and -5°C for 5 months (Table 1). Abamectin was again quite stable.

Table 1. Freezer storage stability of avermectin B_{1a} and 8,9-*Z*-avermectin B_{1a} added to dried hops and fresh hop cones (Arenas, 1997a,b). Duplicate samples were analysed on each occasion.

Storage interval, days	Avermectin B_{1a}, mg/kg	Storage interval, days	8,9-Z-avermectin B_{1a}, mg/kg
DRIED HOPS			
0	0.023 0.021	0	0.019 0.017
34	0.018 0.021	36	0.016 0.014
60	0.023 0.018	61	0.015 0.017
96	0.019 0.017	97	0.014 0.015
190	0.018 0.018	189	0.014 0.014
FRESH HOP CONES			
0	0.018 0.018	0	0.016 0.015
130	0.016 0.014	131	0.015 0.015
153	0.016 0.016	154	0.015 0.015

Definition of the residue

The abamectin residue is currently defined by the JMPR as the sum of avermectin B_{1a}, avermectin B_{1b} and D-8,9 isomer of avermectin B_{1a}.

The Meeting noted that the definition proposed by JECFA (1997) for residues in the liver, kidney and fat from animals subject to veterinary treatments with abamectin does not include the 8,9-*Z* isomer (D-8,9 isomer), because it is not present in animal tissues when abamectin is used directly on the animal. The JECFA efinition also does not include avermectin B_{1b}, because avermectin B_{1a} was considered the appropriate marker residue.

The Meeting agreed that the wider definition (to include the 8,9-*Z* isomer) was the appropriate one for a laboratory carrying out enforcement or monitoring analyses because the analyst would not know whether the residue in the animal originated from veterinary uses, animal feed, or both. In practice, the wider definition accommodates both sources.

The inclusion or exclusion of avermectin B_{1b} from the residue definition is a matter of judgement. In many crop situations B_{1b} is commonly present at approximately 10% of the total residue, so its inclusion or exclusion has little effect on the measured residue. The analytical methods measure B_{1a} and B_{1b} by the same procedure; they appear as two peaks on the same chromatogram, so the analytical data for both components are always available from an analysis and may as well be used. The avermectin B_{1b} residue can be calculated from the avermectin B_{1a} standard curve because the reaction yields and response factors for derivatized B_{1a} and B_{1b} are the same.

Avermectin B_{1b} forms a photoisomer 8,9-*Z*-avermectin B_{1b} in the same way as avermectin B_{1a}. The studies reviewed by the JMPR in 1992 were with avermectin B_{1a} so the possibility of 8,9-*Z*-avermectin B_{1b} being produced was not taken into account. In practice the contribution of 8,9-*Z*-avermectin B_{1b} to the residue will be small but for the sake of accuracy it should be recognised that the HPLC measurement of avermectin B_{1b} residues includes any 8,9-*Z*-avermectin B_{1b}. The Meeting agreed to adjust the residue definition accordingly.

The recommended definition of the residue for compliance with MRLs and estimation of STMRs is

"sum of avermectin B_{1a}, avermectin B_{1b}, 8,9-Z-avermectin B_{1a} and 8,9-Z-avermectin B_{1b}"

USE PATTERN

Table 2. Registered or approved uses of abamectin on food crops. All EC formulations.

Crop	Country	Application				PHI, days
		Method	Rate, kg ai/ha	Spray conc.,kg ai/hl	No.	
Almond	USA		0.014 0.028	0.00019-0.00038	2	21
Apple	Australia	foliar[1]	0.014	0.0014	1	14[23]
Apple	Brazil	foliar[2]	0.0095-0.032	0.0014-0.0018		14
Apple	Canada	foliar[3]	0.014-0.027	0.00036-0.00072	1 or 2[4]	28
Apple	France*	foliar[1]	0.014-0.027	0.0014	2	21
Apple	Israel	foliar[5]		0.0018	6	7
Apple	South Africa	foliar[6] HV	0.014-0.027	0.00063	2 or 4[7]	14
Apple	USA	foliar[8]	0.013-0.026	0.00035-0.00070	2 or 4[7]	28
Celery	Argentina		0.011-0.022	0.0014	4	7
Celery	Cyprus	foliar	0.011-0.022	0.0011	4	7
Celery	France	foliar	0.009		4	7 or 14
Celery	Israel	foliar	0.0054-0.011	0.0009-0.0018	6	7
Celery	Mexico	foliar	0.0054-0.022	0.0011	10	7
Celery	Spain	foliar	0.0054-0.022	0.0011	3	10
Celery	USA	foliar	0.011-0.021		3 or 6[9]	7
Chicory	France	foliar	0.009		4	7 or 14
Citrus	Argentina	foliar HV[10]	0.011-0.027	0.00027-0.00036	2-5	7
Citrus	Brazil	foliar[2]	0.0054-0.011	0.00036-0.00054		7
Citrus	Cyprus	foliar	0.009-0.018	0.00036	6	7
Citrus	Israel	foliar[5]	0.018-0.025		6	7
Citrus	Mexico	foliar[11]	0.0072-0.027	0.00036	3	7
Citrus	South Africa	foliar[6]		0.00018	3	7
Citrus	Spain	foliar[3]	0.014-0.027	0.00072	3	10
Citrus	USA	foliar[8]	0.0066-0.026		2-8[12]	7
Cotton	Argentina	foliar g & a[13]	0.0054-0.011	0.0027-0.011	5-10	20[14]
Cotton	Australia	foliar g & a	0.0054	0.027 max	2	20[15]
Cotton	Brazil	foliar	0.0054-0.011			21
Cotton	Israel	foliar[5]	0.0054-0.011	0.00054	6	7
Cotton	Mexico	foliar	0.009-0.022		4	20[14]
Cotton	South Africa	foliar g & a	0.0054-0.011	0.0054	3	21[16]
Cotton	Spain	foliar	0.0054-0.018	0.0018-0.0036		20
Cotton	USA	foliar g & a	0.011-0.021	note[17]	2	20[14]
Cotton	USA	foliar g & a	0.0053-0.0079	note[18]		
Cucumber	Brazil	foliar[2]	0.0045-0.009	0.0009-0.0018		3
Cucumber	Cyprus	foliar	0.009-0.018	0.0009	4	3
Cucumber	France	foliar	0.022	0.0009	6	3
Cucumber	Germany	greenhouse[19]	0.023		5	3
Cucumber	Israel	foliar	0.0054-0.011	0.0009-0.0018	6	7
Cucumber	Netherlands	foliar gl[20]	0.023	0.00045-0.0009	5	3
Cucumber	Spain	foliar	0.0054-0.022	0.0011	3	3
Cucumber	Switzerland	foliar	0.009- 0.036	0.00045-0.0009	4	3 gl
Cucumber	USA	foliar	0.011-0.021		3 or 6[9]	7
Cucurbits	Argentina		0.011-0.022	0.0014	4	3

Crop	Country	Application				PHI, days
		Method	Rate, kg ai/ha	Spray conc.,kg ai/hl	No.	
Egg plant	France	foliar	0.022		6	3
Egg plant	Germany		0.023-0.009			
Egg plant	Germany	greenhouse[19]	0.023		5	3
Egg plant	Israel	foliar	0.0054-0.011	0.0009-0.0018	6	7
Egg plant	Netherlands	foliar gl[20]	0.023	0.00045-0.0009	4	3[21]
Egg plant	Switzerland	foliar	0.009-0.036	0.00045-0.0009	4	3 gl
Endive	France	foliar f[22]	0.009		4	28
Endive	Netherlands		0.014	0.0009	4	14[21]
Gherkin	Netherlands	foliar gl[20]	0.023	0.00045-0.0009	5	3
Hops	Czech Republic	foliar	0.022	0.0007	2	28
Hops	Germany	foliar	0.023	0.0011	2	28
Hops	USA		0.022		2	28
Lamb’s lettuce	France	foliar f[22]	0.009		4	28
Lettuce	Cyprus	foliar	0.009-0.018	0.0009	4	7
Lettuce	France	foliar f[22]	0.009		4	28
Lettuce	France	foliar	0.009		4	7 or 14
Lettuce	Spain	foliar	0.011-0.022	0.0009-0.0018	3	14
Lettuce, Head	Netherlands		0.014	0.0009	4	14[21]
Lettuce, Head	USA	foliar	0.011-0.021		3 or 6[9]	7
Lettuce, Iceberg	Netherlands		0.014	0.0009	4	14[21]
Melon	Cyprus	foliar	0.009-0.018	0.0009	4	3
Melon	France	foliar	0.022	0.0009	6	3
Melon	Germany	greenhouse[19]	0.023		5	3
Melon	Israel	foliar	0.0054-0.011	0.0009-0.0018	6	7
Melon	Spain	foliar	0.022	0.0011	3	3
Melon	USA	foliar	0.011-0.021		3 or 6[9]	7
Nectarine	Israel	foliar	0.014	0.009		
Peach	Israel	foliar				
Peanuts	Israel	foliar		0.00054		
Pear	Argentina	foliar HV[10]	0.014-0.027	0.00072-0.0014	2-4	14
Pear	Australia	foliar[1]	0.014	0.0014	1	14[23]
Pear	Canada	foliar[3]	0.014-0.027	0.00036-0.00072	1 or 2[4]	28
Pear	Cyprus	foliar[24]	0.011-0.029	0.00054-0.00072	6	7
Pear	France	foliar[10]	0.014	0.0014	2	15
Pear	France	foliar	0.023	0.0014	4	15
Pear	Greece		0.014-0.036	0.0014-0.0018		10
Pear	Israel	foliar[5]		0.0014	6	7
Pear	Italy	foliar[25]	0.014-0.027	0.0014	2	14
Pear	South Africa	foliar[6] HV	0.014-0.027	0.00063	2 or 4[7]	7
Pear	Spain	foliar[3]	0.014-0.027	0.0014	2	10
Pear	Switzerland	foliar	0.023-0.027	0.0014	4	21
Pear	USA	foliar[8]	0.013-0.026	0.00035-0.00070	2 or 4[7]	28
Peppers	Argentina	foliar HV	0.0.009-0.022	0.0009-0.0013	4	3
Peppers	Brazil	foliar[2]	0.009-0.018	0.0009-0.0018		3
Peppers	Cyprus	foliar[24]	0.009-0.018	0.0009	4	3
Peppers	France	foliar	0.022		6	3
Peppers	Israel	foliar	0.0054-0.011	0.0009-0.0018	6	7
Peppers	Spain	foliar	0.0054-0.022	0.0011	3	3
Peppers (bell)	USA	foliar	0.011-0.021		3 or 6[9]	7
Peppers, sweet	Germany	greenhouse[19]	0.023		5	3
Peppers, Sweet	Netherlands	foliar gl[20]	0.023	0.00045-0.0009	4	3[21]
Peppers, Sweet	Switzerland	foliar	0.009-0.036	0.00045-0.0009	4	3 gl
Pome fruit	New Zealand	foliar[11] HV	0.027	0.00068	1	14
Potato	Brazil	foliar[2]	0.009-0.018	0.0011-0.0023		14
Potato	Israel	foliar	0.011	0.0018	6	7
Potato	USA	foliar	0.011-0.021		3 or 6[9]	14
Radish	Netherlands	foliar gl[20]	0.014	0.0009	1	14
Squash	Cyprus	foliar	0.009-0.018	0.0009	4	3

Crop	Country	Application				PHI, days
		Method	Rate, kg ai/ha	Spray conc.,kg ai/hl	No.	
Squash	USA	foliar	0.011-0.021		3 or 6[9]	7
Strawberry	Argentina	foliar HV	0.011-0.022	0.0014-0.0018	4	3
Strawberry	Australia	foliar	0.011-0.022	0.0018	2	3[26]
Strawberry	Brazil	foliar[2]	0.009-0.017	0.009-0.0014		3
Strawberry	Cyprus	foliar	0.018	0.0009	4	3
Strawberry	France	foliar	0.023		4	3
Strawberry	Israel	foliar[5]	0.009	0.0009	6	7
Strawberry	Mexico	foliar	0.009-0.022	0.0018	4	3
Strawberry	New Zealand	foliar HV	0.011	0.0018	3	3
Strawberry	South Africa	foliar HV	0.022		4	3
Strawberry	Spain	foliar	0.011-0.022	0.0014-0.0018	4	3
Strawberry	Switzerland	foliar	0.009-0.014	0.00045	4	
Strawberry	USA	foliar	0.021		4	3
Tomato	Argentina	foliar	0.009-0.022	0.0009-0.0013	4-9	3
Tomato	Australia	foliar HV	0.0054-0.0081	0.0011-0.0016	2	3[26]
Tomato	Brazil	foliar[2]	0.0068-0.022	0.0014-0.0018		3
Tomato	Cyprus	foliar	0.011-0.022	0.0011	4	3
Tomato	France	foliar	0.022	0.0009	6	3
Tomato	Germany		0.009-0.023			
Tomato	Germany	greenhouse[19]	0.023		5	3
Tomato	Greece			0.011-0.0018		3,7
Tomato	Israel	foliar	0.0054-0.011	0.0009-0.0018	6	3
Tomato	Italy	foliar	0.0054-0.022	0.0011	2	7
Tomato	Mexico	foliar	0.0054-0.022	0.0011-0.0018	3	3
Tomato	Netherlands	foliar gl[20]	0.023	0.00045-0.0009	4	3[21]
Tomato	New Zealand	foliar HV gl[20]	0.0081	0.0011-0.0016	3	3
Tomato	South Africa	foliar HV	0.0054-0.022	0.0011	5	3
Tomato	Spain	foliar	0.0054-0.022	0.0011		3 7 gl
Tomato	Switzerland	foliar	0.009-0.018	0.00045-0.0009	4	3 gl
Tomato	USA	foliar	0.011-0.021		3 or 6[9]	7
Walnut	USA		0.014-0.028	0.00019-0.00038	2	21
Watermelon	Brazil	foliar[2]	0.0045-0.009	0.0009-0.0018		7
Watermelon	Cyprus	foliar	0.009-0.018	0.0009	4	3
Watermelon	Israel	foliar	0.0054-0.011	0.0009-0.0018	6	7
Watermelon	Spain	foliar	0.0054-0.022	0.0011		3
Zucchini	France	foliar		0.0009	6	3
Zucchini	Germany		0.009-0.023			
Zucchini	Germany	greenhouse[19]	0.023		5	3
Zucchini	Netherlands	foliar gl[20]	0.023	0.00045-0.0009	5	3

*Proposed registration
[1] Plus summer oil
[2] Plus mineral or vegetable oil.
[3] Plus paraffinic oil.
[4] At the higher rate, 1 application is permitted; at the lower rate, 2.
[5] Plus ultrafine oil.
[6] Plus light mineral spray oil.
[7] At the higher rate, 2 applications are permitted; at the lower rate, 4.
[8] Plus horticultural spray oil.
[9] At the higher rate, 3 applications are permitted; at the lower rate, 6.
[10] Plus summer mineral oil.
[11] Plus agricultural spray oil.
[12] At the highest rate, 2 applications are permitted; at the lowest rate, 8.
[13] g & a - ground and aerial application
[14] Do not graze or feed cotton foliage.
[15] Do not graze or cut for stockfood for 20 days after application.
[16] Do not allow animals to feed on treated foliage.
[17] Late season
[18] Early season.
[19] Do not use during the months of November till February.

[20] gl: glasshouse.
[21] Only from 1 March to 1 November.
[22] Outdoor use only.
[23] Do not feed treated produce to livestock for 14 days after application.
[24] Plus narrow range oil.
[25] Plus agricultural spray mineral oil.
[26] Do not feed treated produce to livestock for 3 days after application.

RESIDUES RESULTING FROM SUPERVISED TRIALS

Residue data from supervised trials on fruits, vegetables and hops are summarized in Tables 3-10. Detailed comparisons of national GAP with the conditions in the trials which were considered to be valid for the estiamtion of maximum residue levels and STMRs are shown in the "interpretation" Tables 11-17.

Table 3. Apples. *Australia, France, Germany, Italy, New Zealand, Spain, UK, USA*
Table 4. Pears. *USA*
Table 5. Cucurbits. *Brazil, France, Mexico, Spain*
Table 6. Cucurbits *USA*
Table 7. Tomatoes. *Netherlands*
Table 8. Lettuce. France, Netherlands, Spain
Table 9. Potatoes. *Brazil, USA*
Table 10. Hops. *Germany, USA*

Limits of detection and determination are generally reported as 0.002 and 0.005 mg/kg respectively. Where these limits apply, residues in the residue tables reported as NQ are detected but <0.005 mg/kg, while residues reported as ND are <0.002 mg/kg. Residues, application rates and spray concentrations have generally been rounded to 2 significant figures or, for residues near the LOD, to 1 significant figure. The listed B_{1a} residue includes avermectin B_{1a} and its photoisomer 8,9-*Z*-avermectin B_{1a}, and the B_{1b} residue includes avermectin B_{1b} and 8,9-*Z*-avermectin B_{1b}.

Although all trials included control plots, no control data are recorded in the tables except where residues were detected in the controls. Residues are not corrected for recovery.

The trials were fully reported as well as summarized..

Abamectin was applied to apple orchards in supervised trials in 3 States in Australia in 1995 using handgun sprayers or airblast back-pack misters. The trials were with 2 treated replicates of 1 or 8 trees.

Apple trees were treated with abamectin in a large programme of supervised trials in France, Germany, Italy, Spain and the UK from 1991 to 1994 (Table 4). Spray equipment and plot sizes varied but the size of each field sample was consistently 12 apples.

Plot sizes in the French apple trials were 3 or 4 trees or a 3-4 m row. Trees were sprayed with a motorised knapsack sprayer, a plot sprayer or by tractor with an airblast sprayer. Apple trees in the German trials of 1991 were sprayed with a pressurised back-pack sprayer. Sub-plot sizes were 1 or 6 trees. In the Italian trials plots of 3-4 trees were sprayed with a handgun motor-driven pump sprayer.

The analytical report for apple trial 115-94-0004R from New Zealand was unclear as to whether the treatment had included a spraying oil. Also some results were reported as total residues instead of as the separate B_{1a} and B_{1b} components.

In Spain apple orchards were sprayed using a tractor-mounted orchard sprayer in 1993. Plots were 3-5 trees. In 1994 the trees were sprayed by motorised knapsack or handgun motor pump. In the UK trials of 1991 the trees were sprayed with a back-pack airblast sprayer. Plots were 1 or 2 trees. The oil used as a spray additive in trial 074-91-0004R was rape seed oil.

In a programme of supervised trials on apples in the USA abamectin was used on common varieties in seven apple-growing states from 1990 to 1992 (Table 3). Abamectin in association with a horticultural spraying oil (9.4 l/ha) was applied by handgun sprayers or tractor driven airblast sprayers. Treated plots consisted of 16-18 trees with sampling of the 4 inner trees. In the 1990 trials the four trees were sampled as 4 replicate subplots, but in 1991 and 1992 they were sampled as 2 replicate subplots each of 2 trees. The field sample was 12 apples. In trial 001-91-1024R 9 mm of rain fell on the final day of treatment so samples were taken 1 day later instead of on the day of treatment.

Abamectin was applied to pear trees in 4 supervised trials in California, the USA, by commercial airblast orchard sprayer (Table 4). The plot size was 16 trees and samples for analysis were taken from the 4 central trees. There was little rainfall during the trials and irrigation was by furrow in two of the trials and by sprinkler in the other two, but the sprinklers were beneath the trees and irrigation water would not have contacted the fruit. Field samples were of 12 pears and 11-12 kg were taken for processing.

Trials on cucurbits in Brazil, France, Mexico and Spain are summarized in Table 5. In each trial in Mexico a field sample from each of 4 replicate plots was analysed. Abamectin was applied by knapsack in all trials except one on cucumbers (002-90-0016R) where a manual sprinkling pump was used. Spray concentrations were progressively decreased through the number of applications in each trial to maintain the required application rate because larger volumes of spray were required for good coverage as the plants approached maturity. Plot sizes were 8-563 m^2 for cucumbers, 23-563 m^2 for pickling cucumbers, 11-763 m^2 for cantaloupe and 46-648 m^2 for honey-dew melons. Field samples were 5-10 fruit for cucumbers, 0.5 kg for pickling cucumbers, and 4 mature fruit for cantaloupes and honey-dew melons.

In melon trials in Brazil abamectin was applied by knapsack to plot sizes of 30-90 m^2. A field sample of 6 melons (4.2-9 kg) was taken from each plot for analysis. The edible pulp was analysed rather than whole melons. Abamectin was applied by knapsack to melons in 60 m^2 plots in supervised trials in France. Field samples consisted of 4-5 fruit. In Spain abamectin was applied by CO_2 pressurized back-pack sprayer to 6 m^2 plots of melons in glasshouses with each plot comprising 4 subplots. Samples of 4-5 fruit were taken from the subplots for analysis.

Glasshouse tomatoes growing on a rockwool substrate were treated with abamectin using a motor driven high-pressure sprayer equipped with a hand-held wand in 2 trials in The Netherlands in March-April 1993. Plot sizes were 19 m^2. In 4 glasshouse tomato trials in The Netherlands in September -October 1993 abamectin was applied 4 times at weekly intervals to plots of 30 m^2 . The final applications were in the first week (2 trials) or third week (2 trials) of October.

Trials on lettuce are summarized in Table 8. Head lettuce were sprayed by knapsack or compressed air experimental sprayer in field trials in France in 1992. There were 4 replicates of 5-10 m^2 in the trial design. The field sample comprised 6 lettuce. The design and procedures were very similar in Spanish trials on head and leaf lettuce also in 1992. Old leaves were discarded from the harvested lettuce in the field.

Abamectin was applied by hand-held sprayer to head lettuce in a series of glasshouse trials in The Netherlands in 1993 and 1994. Four applications were made in each trial at intervals of approximately 5-7 days. The plots were 22.5 m^2. The number of heads in a field sample varied because of growth during the sampling period, but the sample weight was about 2.5 kg. The trials coincided with the change of season because Netherlands GAP specifies that the use on glasshouse lettuce should be between 1 March and 1 November.

Trials on potatoes were carried out in Brazil and the USA (Table 9). In a series of trials in Brazil in 1994 abamectin was applied to the potato foliage by knapsack sprayer with 2 replicates of 20 m^2 plots in each trial. Field samples were 2 kg of tubers.

In 4 trials in the USA in 1992 abamectin was applied 6 times at an exaggerated rate at 6-9 day intervals by CO_2 pressurised sprayers (some treatments included a paraffin crop oil at 9.4 l/ha). Plot sizes ranged from 50 to 100 m^2. One field sample (2-3 kg tubers) was taken from each of 2 subplots or split plots within each plot. The tubers were commercially acceptable for harvest and during field sampling adhering soil was removed as far as possible with a clean dry brush; the tubers were not washed. Similar practices were followed in the US trials in 1993 except that abamectin was applied at the US recommended label rate.

The US trial 001-94-1022 was designed to produce potatoes for processing. Abamectin was applied 6 times, with intervening intervals of 7 days, at an exaggerated rate (0.11 kg ai/ha). Diquat was applied 2 days after the final abamectin application to kill the potato vines and promote tuber bulking. Maturity for the variety Russet Nugget is determined by frost or vine-kill.

Trials on hops were conducted in Germany and the USA (Table 10). Abamectin was applied by tractor-driven airblast sprayers in 8 supervised trials in Germany in 1994 and 1996. The 2 applications in each trial were approximately 21 days apart. Plot sizes ranged from 288 m^2 to 504 m^2. Field samples were taken from the middle rows of the plots. In 1994 fresh hops were dried for 6 hours at 62°C to produce dry hops. In 1996 the mature cones were dried in a commercial hop kiln for approximately 3 hours to produce the dry hops, the final samples of which were at least 500 g.

In 4 supervised trials in the USA in 1994 abamectin was applied twice to hops by tractor-driven airblast sprayers. The plots consisted of 5-7 rows approximately 9 m long. Field samples of 1-2 kg of cones were taken from the middle rows and dried at 38-60°C for 12 hours to produce the dry hops which were analysed. The dry weight was approximately 30% of the fresh weight and the moisture content of the dry hops was close to 9%.

Table 3. Abamectin residues in apples resulting from foliar applications in supervised trials in Australia, France, Germany, Italy, New Zealand, Spain, the UK and the USA. Double-underlined residues are from treatments according to GAP and are valid for estimating maximum residue levels and STMRs. All EC formulations.

Country, year	Application			PHI, days	Residues, mg/kg[1]		Ref
(variety)	kg ai/ha	kg ai/hl	No.		B_{1a} + 8,9-Z-B_{1a}	B_{1b} + 8,9-Z-B_{1b}	
Australia (NSW), 1995 (Granny Smith)	0.014 + oil	0.0008	1	0 14 21	0.015 0.015 0.003 0.002 NQ NQ	ND ND ND ND ND ND	114-95-0001R
Australia (Tas), 1995 (Red Delicious)	0.014 + oil	0.0007	1	0 14 21	0.013 0.012 0.005 0.002 0.002 0.004	NQ NQ ND ND ND ND	114-95-0003R
Australia (Vic), 1995 (Fuji)	0.014 + oil	0.0007	1	0 14 21	0.009 0.007 ND NQ ND ND	ND ND ND ND ND ND	114-95-0002R
France, 1991 (Golden Delicious)	0.027 + oil	0.0015-0.0019	2	0 28	0.006 0.015 0.003 0.004 0.004 NQ NQ NQ	ND 0.002 ND ND ND (4)	066-91-0017R
France, 1991 (Golden Delicious)	0.054 + oil	0.0031 -0.0037	2	0 28	0.016 0.015 0.010 0.008 0.003 ND 0.003	0.002 0.002 ND ND ND (3)	066-91-0017R

Country, year	Application			PHI, days	Residues, mg/kg[1]		Ref
(variety)	kg ai/ha	kg ai/hl	No.		B_{1a} + 8,9-Z-B_{1a}	B_{1b} + 8,9-Z-B_{1b}	
France, 1991 (Jonagold)	0.027	0.002	2	0 7 14 18 26	0.008 0.011 0.013 0.007 NQ 0.004 0.003 0.008 NQ 0.004 0.003 NQ 0.002 0.003 0.004 NQ NQ NQ ND ND	ND ND NQ ND ND (4) ND (4) ND (4) ND (4)	066-91-0016R
France, 1991 (Jonagold)	0.027 + oil	0.002	2	0 7 14 18 26	0.025 0.018 0.018 0.013 0.007 0.011 0.008 0.009 0.004 0.005 0.009 0.006 NQ 0.012 0.008 0.006 0.002 0.004 0.003	0.003 NQ NQ NQ ND (3) ND (4) ND ND NQ ND ND (4)	066-91-0016R
France, 1991 (Jonagold)	0.054	0.004	2	0 7 14 18 26	0.020 0.027 0.038 0.021 0.005 0.012 0.003 0.008 0.004 0.009 NQ 0.005 0.003 0.008 ND 0.005 0.002 0.002 ND 0.002	NQ 0.003 0.004 0.002 ND NQ ND ND ND (4) ND (4) ND (4)	066-91-0016R
France, 1991 (Jonagold)	0.054 + oil	0.004	2	0 7 14 18 26	0.029 0.043 0.033 0.037 ND 0.023 0.021 0.024 0.008 0.015 0.011 0.014 0.008 0.016 0.025 0.020 0.007 0.005 0.005 0.007	0.003 0.005 0.004 0.004 ND 0.002 0.002 0.002 ND NQ ND NQ ND NQ 0.002 0.002 ND (4)	066-91-0016R
France, 1993 (Golden Delicious)	0.014 + oil	0.0012	2	0 28	0.006 0.010 ND ND	NQ NQ ND ND	066-93-0016R
France, 1993 (Golden Delicious)	0.014	0.0012	2	0 28	NQ NQ ND ND	ND ND ND ND	066-93-0016R
France, 1993 (Golden Delicious)	0.014 + oil	0.0012	2	0 28	0.005 0.007 ND ND	ND ND ND ND	066-93-0016R
France, 1993 (Golden Delicious)	0.027	0.0025	2	0 28	0.030 0.029 0.003 0.005	0.004 0.004 ND ND	066-93-0016R
France, 1993 (Golden Delicious)	0.014 + adj	0.0008 -0.0009	2	0 28	0.002 NQ ND ND	ND ND ND ND	066-93-0017R
France, 1993 (Golden Delicious)	0.014	0.0008 -0.0009	2	0 28	0.003 0.002 ND ND	ND ND ND ND	066-93-0017R
France, 1993 (Golden Delicious)	0.014 + adj	0.0008 -0.0009	2	0 28	0.004 NQ ND ND	ND ND ND ND	066-93-0017R
France, 1993 (Golden Delicious)	0.027 + adj	0.0015 -0.0018	2	0 28	0.010 0.014 ND NQ	NQ NQ ND ND	066-93-0017R
France, 1993 (Idared 106)	0.014 + oil	0.0014	1 2	21 0 7 14 21 28	ND ND 0.017 0.013 NQ ND NQ ND ND NQ ND ND	ND ND NQ NQ ND ND ND ND ND ND ND ND	066-93-0015R
France, 1993 (Idared 106)	0.014	0.0014	1 2	21 0 7 14 21 28	ND ND 0.016 0.016 ND ND ND ND NQ NQ ND ND	ND ND NQ 0.002 ND ND ND ND ND ND ND ND	066-93-0015R
France, 1993 (Idared 106)	0.014 + oil	0.0014	1 2	21 0 7 14 21 28	ND ND 0.012 0.003 NQ ND ND NQ NQ NQ ND ND	ND ND NQ ND ND ND ND ND ND ND ND ND	066-93-0015R

Country, year	Application			PHI, days	Residues, mg/kg[1]		Ref
(variety)	kg ai/ha	kg ai/hl	No.		B_{1a} + 8,9-Z-B_{1a}	B_{1b} + 8,9-Z-B_{1b}	
France, 1993 (Red Chief)	0.014 + adj[2]/	0.0014	1 2	21 0 7 14 21 28	ND ND 0.003 0.009 NQ ND ND ND ND ND ND ND	ND ND ND NQ ND ND ND ND ND ND ND ND	066-93-0014R
France, 1993 (Red Chief)	0.014	0.014	1 2	21 0 7 14 21 28	ND ND 0.009 0.013 ND ND ND ND ND ND ND ND	ND ND NQ NQ ND ND ND ND ND ND ND ND	066-93-0014R
France, 1993 (Red Chief)	0.014 + adj	0.014	1 2	21 0 7 14 21 28	ND ND 0.003 0.014 0.004 NQ ND NQ ND ND ND ND	ND ND ND 0.002 ND ND ND ND ND ND ND ND	066-93-0014R
France, 1994 (Golden Delicious)	0.014	0.0014	1 2	21 0 7 14 21 25	ND ND 0.005 0.008 ND ND ND ND ND ND ND ND	ND ND ND NQ ND ND ND ND ND ND ND ND	066-94-0003R
France, 1994 (Golden Delicious)	0.014 + oil	0.0014	1 2	21 0 7 14 21 25	ND NQ 0.014 0.011 0.002 0.003 NQ NQ NQ NQ ND ND	ND ND 0.002 0.002 ND ND ND ND ND ND ND ND	066-94-0003R
France, 1994 (Golden Delicious)	0.014	0.0014	2	0 26	0.003 0.002 ND ND	ND ND ND ND	066-94-0004R
France, 1994 (Golden Delicious)	0.014 + oil	0.0014	2	0 26	0.007 0.008 ND NQ	NQ NQ ND ND	066-94-0004R
Germany, 1991 (Golden Delicious Smoothee M9)	0.027	0.0027	2	0 7 14 21 28	0.019 0.014 0.013 0.017 ND (3) ND (4) ND (4) ND (4)	NQ (4) ND (3) ND (4) ND (4) ND (4)	072-91-0005R
Germany, 1991 (Golden Delicious Smoothee M9)	0.027 + oil	0.0027	2	0 7 14 21 28	0.026 0.022 0.022 0.020 0.008 0.006 0.005 0.009 0.007 0.007 0.003 0.007 0.007 0.006 0.004 0.006 0.005 0.004 0.004 0.004	0.003 0.002 0.003 0.002 ND (4) ND (4) ND (4) ND (4)	072-91-0005R
Germany, 1991 (Golden Delicious)	0.027 + oil	0.0019 -0.0022	2	0 28	0.030 0.023 0.021 0.014 0.008 0.007 0.007 0.005	0.003 0.002 0.002 NQ ND (4)	072-91-0004R
Germany, 1991 (Golden Delicious)	0.027	0.0020 -0.0025	2	0 7 14 21 28	0.022 0.018 0.009 0.004 0.003 0.002 0.002 ND 0.003 NQ ND NQ NQ ND ND NQ ND ND ND	0.002 NQ ND ND ND (4) ND (4) ND (3) ND (4)	072-91-0006R
Germany, 1991 (Golden Delicious)	0.027 + oil	0.0020 -0.0025	2	0 7 14 21 28	0.026 0.031 0.031 0.027 0.009 0.018 0.013 0.014 0.013 0.010 0.007 0.013 0.013 0.008 0.009 0.010 0.006 0.006	0.002 0.003 0.003 0.002 ND NQ NQ NQ NQ NQ ND NQ NQ ND NQ NQ ND ND	072-91-0006R

Country, year	Application			PHI, days	Residues, mg/kg[1]		Ref
(variety)	kg ai/ha	kg ai/hl	No.		B_{1a} + 8,9-Z-B_{1a}	B_{1b} + 8,9-Z-B_{1b}	
Italy, 1993 (Golden Delicious)	0.014 + adj	0.014	1 2	21 0 7 14 21 28	ND ND 0.006 0.005 NQ ND ND ND ND ND ND ND	ND ND ND ND ND ND ND ND ND ND ND ND	067-93-0005R
Italy, 1993 (Golden Delicious)	0.014	0.014	1 2	21 0 7 14 21 28	ND ND 0.002 NQ ND ND ND ND ND ND ND ND	ND ND ND ND ND ND ND ND ND ND ND ND	067-93-0005R
Italy, 1993 (Golden Delicious)	0.014 + adj	0.014	1 2	21 0 7 14 21 28	ND ND 0.005 0.007 ND ND ND ND ND ND ND ND	ND ND ND NQ ND ND ND ND ND ND ND ND	067-93-0005R
Italy, 1993 (Red Chief)	0.014 + adj	0.0010	2	0 28	0.005 0.002 ND ND	ND ND ND ND	067-93-0007R
Italy, 1993 (Red Chief)	0.014	0.0010	2	0 28	0.003 0.003 ND ND	ND ND ND ND	067-93-0007R
Italy, 1993 (Red Chief)	0.014 + adj	0.0010	2	0 28	0.003 0.003 ND ND	ND ND ND ND	067-93-0007R
Italy, 1993 (Red Chief)	0.027	0.0022	2	0 28	0.006 0.007 ND ND	ND ND ND ND	067-93-0007R
Italy, 1993 (Red Chief)	0.014 + oil	0.0010	2	0 28	0.005 0.004 ND ND	ND ND ND ND	067-93-0006R
Italy, 1993 (Red Chief)	0.014	0.0010	2	0 28	0.003 0.003 ND ND	ND ND ND ND	067-93-0006R
Italy, 1993 (Red Chief)	0.014 + oil	0.0010	2	0 28	0.004 0.003 ND ND	ND ND ND ND	067-93-0006R
Italy, 1993 (Red Chief)	0.027 + oil	0.0020	2	0 28	0.015 0.008 ND ND	ND ND ND ND	067-93-0006R
Italy, 1993 (Staymann Red)	0.014 + oil	0.001	1 2	21 0 7 14 22 28	ND ND 0.003 0.006 ND ND ND ND ND ND ND ND	ND ND ND ND ND ND ND ND ND ND ND ND	067-93-0004R
Italy, 1993 (Staymann Red)	0.014	0.001	1 2	21 0 7 14 22 28	ND ND 0.004 NQ ND ND ND ND ND ND ND ND	ND ND ND ND ND ND ND ND ND ND ND ND	067-93-0004R
Italy, 1993 (Staymann Red)	0.014 + oil	0.001	1 2	21 0 7 14 22 28	ND ND 0.004 0.005 ND ND ND ND ND ND ND ND	ND ND ND ND ND ND ND ND ND ND ND ND	067-93-0004R
Italy, 1994 (Starkcrimson)	0.014	0.0014	1 2	21 0 7 14 21 28	ND ND 0.011 0.014 ND ND ND ND ND ND ND ND	ND ND NQ 0.002 ND ND ND ND ND ND ND ND	067-94-0005R

Country, year	Application			PHI, days	Residues, mg/kg[1]		Ref
(variety)	kg ai/ha	kg ai/hl	No.		B_{1a} + 8,9-Z-B_{1a}	B_{1b} + 8,9-Z-B_{1b}	
Italy, 1994 (Starkcrimson)	0.014 +oil	0.0014	1 2	21 0 7 21 28	NQ ND 0.015 0.019 NQ ND ND ND ND ND	ND ND 0.003 0.003 ND ND ND ND ND ND	067-94- 0005R
New Zealand, 1994 (Braeburn)	0.027 +oil	0.0014	2	0 7 14 21 28 35	0.014 0.018 0.012 0.005 0.003 0.004 0.002 ND 0.003 ND NQ 0.002	ND NQ ND ND ND ND ND ND ND ND ND ND	115-94- 0005R
New Zealand, 1994 (Gala)	0.027 +oil?	0.0014	2	0 7 14 21 28 35	0.018 0.019 0.006 0.006 0.007 0.003 0.003 ND c 0.005 0.003 0.004 NQ 0.003	0.002 0.002 ND ND ND ND ND ND c ND ND ND ND ND	115-94- 0004R
Spain, 1991 (Golden Delicious)	0.027	0.0021	2	0 28	0.011 0.012 0.019 0.013 0.004 NQ NQ NQ	NQ ND NQ NQ ND (4)	065-91- 0008R
Spain, 1991 (Red Delicious, Red Chief)	0.027	0.0022	2	0 28	0.013 0.014 0.014 0.012 ND NQ ND ND	NQ NQ 0.002 NQ ND (4)	065-91- 0007R
Spain, 1991 (Red Delicious, Red Chief)	0.027	0.0022 -0.0026	2	0 7 14 21 28	0.005 0.010 0.004 ND 0.003 0.006 ND ND ND ND ND ND ND ND ND	ND ND ND ND ND ND ND ND ND ND ND ND ND ND ND	065-91- 0009R
Spain, 1991 (Red Delicious, Red Chief)	0.027 + oil	0.0022 -0.0026	2	0 7 14 21 28	0.009 0.016 0.014 0.011 0.002 0.005 NQ 0.003 NQ 0.004 0.003 0.003 ND NQ 0.003 NQ NQ 0.004 0.003 NQ	ND NQ NQ NQ ND (4) ND (4) ND (4) ND (4)	065-91- 0009R
Spain, 1993 (Golden Delicious)	0.014 + oil	0.0011	2	0 28	0.005 0.005 ND ND	ND ND ND ND	065-93- 0006R
Spain, 1993 (Golden Delicious)	0.014	0.0011	2	0 28	0.005 0.007 ND ND	ND ND ND ND	065-93- 0006R
Spain, 1993 (Golden Delicious)	0.014 + oil	0.0011	2	0 28	0.003 0.004 ND ND	ND ND ND ND	065-93- 0006R
Spain, 1993 (Golden Delicious)	0.027 + oil	0.0023	2	0 28	0.018 0.012 ND ND	NQ NQ ND ND	065-93- 0006R
Spain, 1993 (Golden Delicious)	0.014 + adj[2]	0.0012	2	0 28	NQ NQ ND ND	ND ND ND ND	065-93- 0007R
Spain, 1993 (Golden Delicious)	0.014	0.0012	2	0 28	0.005 0.011 ND ND	ND NQ ND ND	065-93- 0007R
Spain, 1993 (Golden Delicious)	0.014 + adj	0.0012	2	0 28	0.009 ND ND ND	NQ ND ND ND	065-93- 0007R
Spain, 1993 (Golden Delicious)	0.027 + adj	0.0025	2	0 28	0.017 0.014 ND NQ	0.002 NQ ND ND	065-93- 0007R
Spain, 1993 (Well Spur)	0.014	0.0014	1 2	21 0 7 14 21 28	ND ND 0.006 0.007 ND ND ND ND ND ND ND ND	ND ND ND ND ND ND ND ND ND ND ND ND	065-93- 0005R
Spain, 1993 (Well Spur)	0.014 + adj	0.0014	1 2	21 0 7 14 21 28	ND 0.003 0.007 0.009 NQ NQ ND NQ NQ NQ NQ ND	ND ND ND NQ ND ND ND ND ND ND ND ND	065-93- 0005R

Country, year	Application			PHI, days	Residues, mg/kg[1]		Ref
(variety)	kg ai/ha	kg ai/hl	No.		B_{1a} + 8,9-Z-B_{1a}	B_{1b} + 8,9-Z-B_{1b}	
Spain, 1993 (Well Spur)	0.014 + adj	0.0014	1 2	21 0 7 14 21 28	ND NQ 0.003 0.004 ND NQ NQ ND ND NQ ND ND	ND ND ND ND ND ND ND ND ND ND ND ND	065-93-0005R
Spain, 1994 (Golden Delicious)	0.014	0.014	2	0 30	0.004 0.004 ND ND	ND ND ND ND	065-94-0009R
Spain, 1994 (Golden Delicious)	0.014 + oil	0.014	2	0 30	0.004 0.005 ND ND	ND ND ND ND	065-94-0009R
UK, 1991 (Cox's Orange Pippin)	0.027 + oil	0.0020	2	0 28	0.026 0.019 0.027 0.020 0.005 0.005 0.010 0.007	0.003 0.002 0.003 0.002 ND (4)	074-91-0003R
UK, 1991 (Cox's Orange Pippin)	0.054 + oil	0.0038 -0.0041	2	0 28	0.044 0.013 0.039 0.028 0.016 0.008 0.010 0.010	0.006 NQ 0.004 0.003 NQ ND ND ND	074-91-0003R
UK, 1991 (Cox's Orange Pippin)	0.027	0.0013 -0.0016	2	0 7 14 21 28	0.026 0.034 0.018 0.021 NQ 0.002 NQ NQ ND ND ND NQ NQ ND ND ND ND (4)	0.002 0.003 NQ NQ ND (4) ND (4) ND (4) ND (4)	074-91-0004R
UK, 1991 (Cox's Orange Pippin)	0.027 + oil	0.0013 -0.0016	2	0 7 14 21 28	0.035 0.033 0.044 0.043 0.009 0.010 0.011 0.009 0.006 0.007 0.008 0.007 0.006 0.004 0.009 0.006 0.005 0.005 0.006 0.005	0.003 0.003 0.004 0.004 ND NQ NQ ND ND (4) ND (4) ND (4)	074-91-0004R
UK, 1991 (Cox's Orange Pippin)	0.054	?	2	0 7 14 21 28	0.056 0.061 0.069 0.051 0.006 0.007 0.012 0.018 0.007 0.004 0.005 0.008 0.003 0.003 0.004 0.005 0.004 0.002 0.003 0.003	0.006 0.007 0.008 0.007 ND ND NQ NQ ND (4) ND (4) ND (4)	074-91-0004R
UK, 1991 (Cox's Orange Pippin)	0.054 + oil	0.0025 -0.0031	2	0 7 14 21 28	0.068 0.069 0.072 0.067 0.030 0.025 0.032 0.029 0.013 0.016 0.021 0.029 0.013 0.020 0.019 0.015 0.012 0.012 0.008 0.015	0.007 0.008 0.008 0.008 0.003 0.002 0.003 0.003 NQ NQ NQ 0.003 NQ (4) ND ND ND NQ	074-91-0004R
USA (CA), 1991 (Golden Delicious)	0.027 +oil	0.0038	2	0 28	0.019 0.020 0.010 0.008	0.003 0.003 NQ ND	001-91-6016R 618-936-AP
USA (CA), 1991 (Granny Smith)	0.027 +oil	0.0010	2	0 28	0.009 0.010 ND ND	ND NQ ND ND	001-91-6024R 618-936-AP
USA (GA), 1992 (Red Delicious)	0.027 +oil	0.0072	2	0 28	0.047 0.045 ND NQ	0.006 0.005 ND ND	001-92-0027R 618-936-AP
USA (MI), 1990 (Golden Delicious)	0.028 +oil	0.0010	2	0 3 7 14 28 45	0.011 0.042 0.026 0.091 0.010 0.010 0.008 0.006 0.009 0.010 0.010 0.006 0.005 0.005 0.010 0.005 0.004 0.006 0.003 0.003 0.004 0.006 0.006 0.005	ND 0.005 0.004 ND NQ (4) NQ NQ NQ ND ND ND NQ NQ ND ND NQ ND ND (4)	001-90-5018R 618-936-AP

Country, year	Application			PHI, days	Residues, mg/kg[1]		Ref
(variety)	kg ai/ha	kg ai/hl	No.		B_{1a} + 8,9-Z-B_{1a}	B_{1b} + 8,9-Z-B_{1b}	
USA (MI), 1990 (Golden Delicious)	0.056 +oil	0.0020	2	0 3 7 14 28 45	0.049 0.031 0.040 0.033 0.020 0.011 0.018 0.012 0.011 0.009 0.021 0.016 0.013 0.006 0.008 0.006 0.005 0.009 0.004 0.004 0.004 0.004 0.004 0.004	0.008 0.006 0.006 0.007 0.003 NQ 0.003 NQ NQ NQ 0.003 NQ NQ ND ND ND ND (4) ND (4)	001-90-5018R 618-936-AP
USA (MI), 1991 (Jonathan)	0.027 +oil	0.0036	2	1 7 14 28	0.008 0.008 0.002 0.003 NQ NQ NQ 0.002	ND ND ND ND ND ND ND ND	001-91-1024R 618-936-AP
USA (NC), 1992 (Red Delicious)	0.026 +oil	0.0071	2	0 28	0.031 0.027 0.003 NQ	0.003 0.003 ND ND	001-92-0026R 618-936-AP
USA (NY), 1990 (Twenty Ounce)	0.028 +oil	0.0007	2	0 3 7 14 28	0.011 0.012 0.030 0.018 NQ 0.004 0.011 0.012 0.002 0.003 0.004 0.005 0.002 NQ 0.003 0.002 ND NQ 0.003 NQ	0.002 0.002 0.004 0.003 ND ND NQ NQ ND (4) ND (4) ND (4)	001-90-5016R 618-936-AP
USA (NY), 1990 (Twenty Ounce)	0.056 +oil	0.0015	2	0 3 7 14 28	0.033 0.028 0.028 0.035 0.062 0.011 0.016 0.009 0.015 0.007 0.003 0.008 0.003 0.002 0.003 0.004 0.002 NQ 0.003 0.003	0.004 0.004 0.004 0.005 0.009 NQ NQ NQ 0.003 NQ ND NQ ND (4) ND (4)	001-90-5016R 618-936-AP
USA (NY), 1991 (Red Delicious)	0.027 +oil	0.0038	2	0 7 14 28	0.040 0.037 0.008 0.008 0.011 0.011 0.007 0.007	0.004 0.004 ND NQ NQ NQ ND ND	001-91-3000R 618-936-AP
USA (NY), 1992 (Rome Beauty)	0.027 +oil	0.0072	2	0 28	0.020 0.020 NQ 0.004	0.002 0.003 ND ND	001-92-3020R 618-936-AP
USA (OR), 1992 (Golden Delicious)	0.027 +oil	0.0008	2	0 28	0.022 0.017 0.003 ND	0.003 NQ ND ND	001-92-6012R 618-936-AP
USA (OR), 1992 (Red Delicious)	0.027 +oil	0.0081	2	0 28	0.009 0.016 ND ND	ND NQ ND ND	001-92-1014R 618-936-AP
USA (WA), 1991 (Red Delicious)	0.027 +oil	0.0011	2	0 28	0.012 0.010 ND NQ	NQ NQ ND ND	001-91-1021R 618-936-AP
USA (WA), 1991 (Red Delicious)	0.026 +oil	0.0037	2	0 7 14 28	0.021 0.027 0.008 0.005 0.007 0.004 0.002 0.003	NQ 0.003 ND ND ND ND ND ND	001-91-1023R 618-936-AP
USA (WA), 1992 (Red Delicious)	0.027 +oil	0.0072	2	0 28	0.018 0.019 NQ ND	0.002 NQ ND ND	001-92-1018R 618-936-AP

1 NQ: not quantified; detected but <0.002 mg/kg.
ND: not detected, <0.001 mg/kg.
2 adj: adjuvant - silicone polyether copolymer surfactant.

Table 4. Abamectin residues in Bartlett pears resulting from foliar applications in supervised trials in California, USA, 1992.

Application				PHI, days	Residues, mg/kg[1]		Ref
Form	kg ai/ha	kg ai/hl	No.		B_{1a} + 8,9-Z-B_{1a}	B_{1b} + 8,9-Z-B_{1b}	
EC + oil	0.027		2	0 21	0.020 0.004	0.002 ND	001-92-6016R
EC + oil	0.027		2	0 21	0.014 (0.015 0.012) 0.006 (0.005 0.006)	<0.002 (NQ NQ) <0.001 (ND ND)	001-92-6017R
EC + oil	0.027		2	0 21	0.023 (0.030 0.016) 0.007 (0.006 0.009)	0.002 (0.003 NQ) <0.002 (ND NQ)	001-92-6018R
EC + oil	0.027		2	0 21	0.021 (0.021 0.020) 0.009 (0.011 0.008)	0.002 (0.002 0.002) <0.002 (NQ NQ)	001-92-6019R

1 NQ: not quantified; detected but <0.002 mg/kg.
ND: not detected, <0.001 mg/kg.

Table 5. Abamectin residues in cucurbits resulting from foliar applications in supervised trials in Brazil, France, Mexico and Spain. Double-underlined residues are from treatments according to GAP and are valid for estimating maximum residue levels and STMRs.

Country, year (variety)	Application				PHI,	Residues, mg/kg[1]		Ref
	Form	kg ai/ha	kg ai/hl	No.	days	B1a + 8,9-Z-B1a	B1b + 8,9-Z-B1b	
CUCUMBER								
Mexico, 1990	EC	0.023	0.009- 0.017	10	0 3 7	0.014 0.007 0.012 0.007 NQ 0.009 0.007 0.011 0.007 0.006 0.006 0.006	ND (4) ND (4) ND (4)	002-90- 0011R
Mexico, 1990 (Poinset 76 & 1810)	EC	0.045	0.018- 0.034	10	0 3 7	0.015 0.015 0.026 0.013 0.009 0.009 0.008 0.018 0.016 0.009 0.006 0.006	ND NQ (3) ND (4) ND (4)	002-90- 0011R
Mexico, 1990 (Jet Set)	EC	0.023	0.009- 0.017	9	0 3 7	0.024 0.013 0.015 0.012 0.014 0.014 0.009 0.011 0.007 0.006 0.005 NQ	NQ NQ ND ND ND (4) ND (4)	002-90- 0012R
Mexico, 1990 (Jet Set)	EC	0.045	0.018- 0.034	9	0 3 7	0.010 0.026 0.010 0.010 0.013 0.024 0.026 0.014 0.006 0.006 0.006 NQ	ND NQ ND ND ND NQ NQ ND ND (4)	002-90- 0012R
Mexico, 1990 (Dasher II)	EC	0.023	0.005- 0.017	9	0 3 7	ND (4) ND (4) ND (4)	ND (4) ND (4) ND (4)	002-90- 0016R
Mexico, 1990 (Dasher II)	EC	0.045	0.010- 0.034	9	0 3 7	NQ NQ ND NQ ND (4) ND (4)	ND (4) ND (4) ND (4)	002-90- 0016R
PICKLING CUCUMBER								
Mexico, 1989 (Carolina)	EC	0.023	0.009- 0.017	7	0 3 7	0.005 0.009 0.006 0.011 ND NQ ND ND ND (4)	ND (4) ND (4) ND (4)	002-90- 0013R
Mexico, 1989 (Carolina)	EC	0.045	0.018- 0.034	7	0 3 7	0.038 0.023 0.013 0.009 NQ 0.007 NQ NQ ND ND ND NQ	NQ ND ND ND ND (4) ND (4)	002-90- 0013R
Mexico, 1990 (Carolina)	EC	0.023	0.009- 0.017	8	0 3 7	0.006 0.008 0.006 NQ NQ ND 0.008 NQ ND (4)	ND (4) ND (4) ND (4)	002-90- 0014R
Mexico, 1990 (Carolina)	EC	0.045	0.019- 0.034	8	0 3 7	0.012 0.025 0.007 0.015 ND 0.020 NQ NQ ND (4)	ND (4) ND (4) ND (4)	002-90- 0014R

Country, year (variety)	Application				PHI,	Residues, mg/kg[1]		Ref
	Form	kg ai/ha	kg ai/hl	No.	days	B1a + 8,9-Z-B1a	B1b + 8,9-Z-B1b	
Mexico, 1990 (Carolina)	Control plot				0 3 7	0.011 NQ 0.005 NQ 0.22 0.036 0.066 ND ND (4)	ND (4) 0.017 NQ NQ ND ND (4)	002-90-0014R
Mexico, 1990 (Flury)	EC	0.023	0.003-0.007	6	0 3 7	NQ (4) ND ND ND NQ ND (4)	ND (4) ND (4) ND (4)	002-90-0015R
Mexico, 1990 (Flury)	EC	0.045	0.006-0.014	6	0 3 7	0.008 0.008 0.007 0.008 NQ 0.008 0.008 NQ ND (4)	ND (4) ND (4) ND (4)	002-90-0015R
MELON, CANTALOUPE								
Brazil, 1993 (Amarelo CAC)	EC	0.014	0.0018	4	0 3 7	pu ND ND pu ND ND pu ND ND	pu ND ND pu ND ND pu ND ND	015-93-0034R
Brazil, 1993 (Amarelo CAC)	EC	0.029	0.0036	4	0 3 7	pu ND ND pu ND ND pu ND ND	pu ND ND pu ND ND pu ND ND	015-93-0034R
Brazil, 1994 (Bonus II)	EC	0.014	0.0018	4	0 3 7	pu ND ND pu ND ND pu ND ND	pu ND ND pu ND ND pu ND ND	015-93-0035R
Brazil, 199 (Bonus II)	EC	0.029	0.0036	4	0 3 7	pu ND ND pu ND ND pu ND ND	pu ND ND pu ND ND pu ND ND	015-93-0035R
Brazil, 1994 (Amarelo CAC)	EC	0.014	0.0018	4	0 3 7	pu ND ND pu ND ND pu ND ND	pu ND ND pu ND ND pu ND ND	015-93-0036R
Brazil, 1994 (Amarelo CAC)	EC	0.029	0.0036	4	0 3 7	pu ND ND pu ND ND pu ND ND	pu ND ND pu ND ND pu ND ND	015-93-0036R
France, 1991 (Pancha)	EC	0.023	0.0028	4	0 3 7	NQ ND NQ NQ ND (4) ND (4)	ND (4) ND (4) ND (4)	066-91-0003R
France, 1991 (Pancha)	EC	0.045	0.0056	4	0 3 7	0.008 0.010 0.008 0.013 NQ NQ ND NQ ND NQ ND ND	ND ND ND NQ ND (4) ND (4)	066-91-0003R
France, 1991 (Pancha)	EC	0.023	0.0032	4	0 3 7	NQ NQ 0.009 0.008 NQ ND ND ND ND (4)	ND (4) ND (4) ND (4)	066-91-0004R
France, 1991 (Pancha)	EC	0.045	0.0064	4	0 3 7	NQ 0.013 NQ 0.013 NQ (4) ND (4)	ND (4) ND (4) ND (4)	066-91-0004R
France, 1991 (Panchito)	EC	0.023	0.0030	4	0 3 7	NQ 0.020 NQ 0.014 NQ NQ ND ND ND (4)	ND NQ ND ND ND (4) ND (4)	066-91-0005R
France, 1991 (Panchito)	EC	0.045	0.0060	4	0 3 7	0.013 0.010 0.012 0.007 NQ (4) ND (4)	ND (4) ND (4) ND (4)	066-91-0005R
Mexico, 1990 (Durango)	EC	0.023	0.010-0.026	12	0 3 7	0.012 0.007 0.006 0.010 NQ (4) ND (4)	ND (4) ND (4) ND (4)	002-90-0035R
Mexico, 1990 (Durango)	EC	0.045	0.021-0.052	12	0 3 7	0.046 0.024 0.027 0.041 NQ NQ 0.007 0.006 NQ (4)	0.005 NQ NQ NQ ND ND NQ ND ND (4)	002-90-0035R
Mexico, 1990 (Durango)	EC	0.023	0.009-0.052	14	0 3 7	0.012 0.012 0.015 0.018 ND NQ NQ NQ NQ ND ND ND	ND ND ND NQ ND (4) ND (4)	002-90-0036R
Mexico, 1990 (Durango)	EC	0.045	0.019-0.11	14	0 3 7	0.034 0.026 0.023 0.040 0.010 NQ 0.013 0.007 NQ NQ 0.005 0.005	NQ (4) ND (4) ND (4)	002-90-0036R
Mexico, 1990 (Easy Rider)	EC	0.023	0.005-0.016	7	0 3 7	ND ND ND NQ ND (4) ND (4)	ND (4) ND (4) ND (4)	002-90-0037R

Country, year (variety)	Application				PHI,	Residues, mg/kg[1]		Ref
	Form	kg ai/ha	kg ai/hl	No.	days	B1a + 8,9-Z-B1a	B1b + 8,9-Z-B1b	
Mexico, 1990 (Easy Rider)	EC	0.045	0.011- 0.032	7	0 3 7	NQ NQ ND NQ ND (4) ND (4)	ND (4) ND (4) ND (4)	002-90- 0037R
Spain, 1991 (Rochet-Solo)	EC	0.022	0.0022	4 gl	0 3 7	0.005 (NQ ND NQ 0.007) ND (ND (4)) ND (ND (4))	ND (ND (4)) ND (ND (4)) ND (ND (4))	065-91- 0003R
Spain, 1991 (Rochet-Solo)	EC	0.043	0.0043	4 gl	0 3 7	0.008 (0.011 0.008 0.007 0.006) NQ (NQ NQ ND NQ) ND (ND (4))	ND (ND (4)) ND (ND (4)) ND (ND (4))	065-91- 0003R
Spain, 1991 (Rochet-Solo)	EC	0.022	0.0022	4 gl	0 3 7	NQ (NQ ND NQ NQ) ND (ND (4)) ND (ND (4))	ND (ND (4)) ND (ND (4)) ND (ND (4))	065-91- 0004R
Spain, 1991 (Rochet-Solo)	EC	0.043	0.0043	4 gl	0 3 7	0.008 (NQ 0.010 0.011 0.006) NQ (NQ (4)) NQ (NQ ND NQ ND)	ND (ND (4)) ND (ND (4)) ND (ND (4))	065-91- 0004R
MELON , HONEY DEW								
Mexico, 1990 (Hy-mark)	EC	0.023	0.009- 0.049	14	0 3 7	0.009 0.008 0.006 0.006 NQ 0.005 NQ NQ NQ (4)	ND (4) ND (4) ND (4)	002-90- 0038R
Mexico, 1990 (Hy-mark)	EC	0.045	0.018- 0.098	14	0 3 7	0.011 0.014 0.012 0.013 NQ NQ 0.009 0.007 0.011 0.013 NQ 0.011	ND (4) ND (4) ND (4)	002-90- 0038R
Mexico, 1990 (Hy-mark)	control plot				0 3 7	ND 0.011 ND ND ND (4) ND (4)	ND (4) ND (4) ND (4)	002-90- 0038R
Mexico, 1990 (Green Flesh)	EC	0.023	0.003- 0.007	9	0 3 7	0.007 NQ NQ NQ NQ (4) NQ NQ ND ND	ND (4) ND (4) ND (4)	002-90- 0039R
Mexico, 1990 (Green Flesh)	EC	0.045	0.006- 0.014	9	0 3 7	0.009 0.010 0.007 0.008 0.007 NQ NQ ND NQ NQ NQ ND	ND (4) ND (4) ND (4)	002-90- 0039R
Mexico, 1990 (Honey Dew)	EC	0.023	0.011	8	0 3 7	ND (4) ND (4) ND (4)	ND (4) ND (4) ND (4)	002-90- 0040R
Mexico, 1990 (Honey Dew)	EC	0.045	0.023	8	0 3 7	ND (4) ND (4) ND (4)	ND (4) ND (4) ND (4)	002-90- 0040R
Mexico, 1990 (Green Flesh)	EC	0.023	0.004- 0.006	7	0 3 7	0.006 NQ NQ NQ ND ND NQ ND ND (4)	ND (4) ND (4) ND (4)	002-90- 0042R
Mexico, 1990 (Green Flesh)	EC	0.045	0.007- 0.012	7	0 3 7	0.008 NQ NQ 0.007 NQ NQ ND ND ND (4)	ND (4) ND (4) ND (4)	002-90- 0042R

[1] NQ: not quantified; detected but <0.005 mg/kg.
ND: not detected, <0.002 mg/kg.
[2] pu: residues in edible pulp
[3] gl: glasshouse trial.

Table 6. Abamectin residues in cucurbits resulting from foliar applications in supervised trials in the USA. Double-underlined residues are from treatments according to GAP and are valid for estimating maximum residue levels and STMRs.

CUCURBITS, Country, year (variety)	Application				PHI, days	Residues, mg/kg[1]		Ref
	Form	kg ai/ha	kg ai/hl	No.		B_{1a} + 8,9-Z-B_{1a}	B_{1b} + 8,9-Z-B_{1b}	
WATERMELON								
TX, 1992 (Royal Sweet)	EC	0.021	0.011	4	0 7	ND ND ND ND	ND ND ND ND	001-91-1025R 618-936-93127
CA,1991 (Calsweet)	EC	0.021	0.011	4	0 7	NQ NQ ND ND	ND ND ND ND	001-91-6010R 618-936-93127
CANTALOUPE								
TX, 1992 (Caravelle)	EC	0.021	0.023	4	0 7	NQ 0.005 ND ND	ND ND ND ND	001-91-1026R 618-936-93127
AZ, 1992 (Top Mark)	EC	0.021	0.011	4	0 7	0.010 0.006 ND ND	ND ND ND ND	001-91-1027R 618-936-93127
CA, 1992 (Top Mark)	EC	0.021	0.023	4	0 7	0.008 NQ ND ND	ND ND ND ND	001-91-6011R 618-936-93127
FL, 1992 (Planters Jumbo)	EC	0.021	0.011	4	0 7	0.008 0.005 ND ND	ND ND ND ND	001-92-0019R 618-936-93127
GA, 1992 (Planters Jumbo)	EC	0.021	0.011	4	0 7	0.012 0.015 ND ND	ND NQ ND ND	001-92-0020R 618-936-93127
SC, 1992 (Edisto)	EC	0.021	0.010	4	0 7	NQ NQ ND ND	ND ND ND ND	001-92-0021R 618-936-93127
MI, 1992 (Super-star)	EC	0.021	0.010	4	0 7	0.011 0.013 ND ND	ND ND ND ND	001-92-1001R 618-936-93127
PA, 1992 (Ball 1776)	EC	0.022	0.011	4	0 7	NQ NQ ND ND	ND ND ND ND	001-92-3014R 618-936-93127
CA, 1992 (Top Mark)	EC	0.022	0.011	5	0 7	0.006 0.006 ND ND	ND ND ND ND	001-92-6013R 618-936-93127
SUMMER SQUASH, ZUCCHINI								
FL, 1992 (Dixie Hybrid VGB 960)	EC	0.021	0.011	4	0 3 7	0.007 0.007 ND ND ND ND	ND ND ND ND ND ND	001-92-0029R 618-936-93127
TX, 1992 (Onyx)	EC	0.021	0.015	4	0 3 7	0.005, NQ ND ND ND ND	ND ND ND ND ND ND	001-92-1020R 618-936-93127
NY, 1992 (Yellow Crookneck)	EC	0.021	0.011	4	0 3 7	NQ NQ ND ND ND ND	ND ND ND ND ND ND	001-92-3019R 618-936-93127
CA, 1992 (Crookneck)	EC	0.025	0.011	4	0 3 7	0.012 0.011 0.009 NQ ND ND	ND ND ND ND ND ND	001-92-6014R 618-936-93127
CUCUMBER								
SC, 1992 (Ashley)	EC	0.021	0.010	4	0 3 7	0.015 0.012 ND ND ND ND	ND ND ND ND ND ND	001-92-0030R 618-936-93127

CUCURBITS, Country, year (variety)	Application				PHI, days	Residues, mg/kg[1]		Ref
	Form	kg ai/ha	kg ai/hl	No.		B_{1a} + 8,9-Z-B_{1a}	B_{1b} + 8,9-Z-B_{1b}	
MI, 1992 (Calypso Hybrid)	EC	0.022	0.012	4	0 3 7	NQ ND ND ND ND ND	ND ND ND ND ND ND	001-92-1019R 618-936-93127
PA, 1992 (Market-more 76)	EC	0.022	0.012	4	0 3 7	NQ NQ ND ND ND ND	ND ND ND ND ND ND	001-92-3018R 618-936-93127
CA, 1992 (Dasher II)	EC	0.021	0.011	4	0 3 7	0.013 0.010 NQ NQ NQ ND	ND ND ND ND ND ND	001-92-6015R 618-936-93127

[1] NQ: not quantified; detected but <0.005 mg/kg.
ND: not detected, <0.002 mg/kg.

Table 7. Abamectin residues in tomatoes resulting from foliar applications in supervised trials in The Netherlands, 1993. Double-underlined residues are from treatments according to GAP and are valid for estimating maximum residue levels and STMRs. All glasshouse trials.

Variety	Application				PHI, days	Residues, mg/kg[1]		Ref
	Form	kg ai/ha	kg ai/hl	No.		B1a + 8,9-Z-B1a	B1b + 8,9-Z-B1b	
Cesar	EC	0.023	0.0011	4	0 3 7	0.019 0.024 0.010 0.017 0.007 0.012	ND NQ ND ND ND ND	070-93-0005R
Pronto	EC	0.023	0.0011	4	0 3 7	0.018 0.017 0.011 0.012 0.008 0.010	ND ND ND ND ND ND	070-93-0006R
Pronto	EC	0.023	0.0011	4	0 3 7	0.015 0.015 0.007 0.011 0.009 0.012	ND ND ND ND ND ND	070-93-0003R
Pronto	EC	0.023	0.0011	4	0 3 7	NQ 0.011 NQ 0.007 NQ 0.006	ND ND ND ND ND ND	070-93-0002R
Pronto	EC	0.045	0.0023	4	0 3 7	0.022 0.011 0.022 0.025 0.013 0.020	ND ND NQ NQ ND NQ	070-93-0002R
Pronto	EC	0.023	0.0011	4	0 3 7	0.005 0.009 NQ 0.009 NQ 0.007	ND ND ND ND ND ND	070-93-0001R
Pronto	EC	0.045	0.0023	4	0 3 7	0.012 0.017 0.010 0.016 0.011 0.048	ND ND ND ND ND NQ	070-93-0001R
Trust	EC	0.023	0.0011	4	0 3 7	NQ 0.006 NQ 0.006 NQ 0.007	ND ND ND ND ND ND	070-93-0004R

[1]NQ: not quantified; detected but <0.005 mg/kg.
ND: not detected, <0.002 mg/kg.

Table 8. Abamectin residues in lettuce resulting from foliar applications in supervised trials in France, The Netherlands and Spain. Double-underlined residues are from treatments according to GAP and are valid for estimating maximum residue levels and STMRs.

Country, year	Application				PHI, days	Residues, mg/kg[1]		Ref
(variety)	Form	kg ai/ha	kg ai/hl	No.		B_{1a} + 8,9-Z-B_{1a}	B_{1b} + 8,9-Z-B_{1b}	
HEAD LETTUCE								
France, 1992 (Balisto)	EC	0.0113	0.0020 -0.0028	4	0 6 13	0.22 0.27 0.15 0.14 ND (4) ND (4)	0.025 0.030 0.017 0.017 ND (4) ND (4)	066-92-0001R
France, 1992 (Balisto)	EC	0.025	0.0039 -0.0056	4	0 6 13	0.23 0.36 0.34 0.36 NQ NQ 0.002 NQ ND (4)	0.026 0.041 0.037 0.042 ND (4) ND (4)	066-92-0001R
France, 1992 (Divina)	EC	0.0113	0.0019 -0.0023	4	0 7 14	0.18 0.20 0.26 0.28 0.003 0.002 0.004 0.003 NQ NQ NQ 0.002	0.020 0.022 0.028 0.029 ND (4) ND (4)	066-92-0003R
France, 1992 (Divina)	EC	0.0225	0.0038 -0.0045	4	0 7 14	0.28 0.30 0.36 0.45 0.004 0.004 0.007 0.005 NQ NQ NQ 0.005	0.029 0.032 0.037 0.046 ND (4) ND (4)	066-92-0003R
France, 1992 (Scarole Maral)	EC	0.0113	0.0019	4	0 7 14	0.086 0.18 0.15 0.16 0.010 0.012 0 010 0.021 0.002 0.003 0.005 0.005	0.007 0.019 0.015 0.016 ND NQ ND NQ ND (4)	066-92-0002R
France, 1992 (Scarole Maral)	EC	0.0225	0.0038	4	0 7 14	0.23 0.30 0.31 0.29 0.029 0.041 0.028 0.025 0.010 0.011 0.009 0.013	0.024 0.032 0.032 0.030 0.003 0.004 0.003 NQ ND (4)	066-92-0002R
Netherlands, 1993 (Kirsten)	EC	0.014	0.0014	4 gl	0 7 14	0.26 0.26 (Oct) [2] 0.016 0.020 0.017 0.020	0.017 0.019 NQ NQ 0.004 0.005	070-93-0007R
Netherlands, 1993 (Kirsten)	EC	0.027	0.0027	4 gl	0 7 14	0.23 0.25 (Oct) 0.13 0.091 0.096 0.084	0.020 0.022 0.006 0.004 0.009 0.006	070-93-0007R
Netherlands, 1993 (Kirsten)	EC	0.014	0.0014	4 gl	0 7 14	0.25 0.28 (Oct) 0.071 0.074 0.026 0.027	0.024 0.028 0.006 0.007 0.002 0.002	070-93-0008R
Netherlands, 1993 (Kirsten)	EC	0.027	0.0027	4 gl	0 7 14	0.53 0.51 (Oct) 0.10 0.15 0.059 0.097	0.051 0.043 0.008 0.011 0.005 0.009	070-93-0008R
Netherlands, 1994 (Rex)	EC	0.014	0.0014	4 gl	0 7 14	0.32 0.37 (Mar) 0.052 0.069 0.020 0.026	0.025 0.0032 0.004 0.006 0.002 0.003	070-94-0002R
Netherlands, 1994 (Vivaldi)	EC	0.014	0.0014	4 gl	0 7 14	0.33 0.25 (Mar) 0.038 0.027 0.012 0.014	0.025 0.021 0.004 0.003 NQ NQ	070-94-0001R
Spain, 1992 (Trocadero)	EC	0.022	0.0022 -0.0044	4	0 7 14	0.96 0.87 0.94 0.79 0.051 0.076 0.067 0.054 0.024 0.020 0.026 0.025	0.10 0.091 0.095 0.083 0.006 0.009 0.009 0.007 0.003 NQ 0.002 0.002	065-92-0001R
Spain, 1992 (Trocadero)	EC	0.043	0.0043 -0.0086	4	0 7 14	1.5 1.7 1.6 1.5 0.15 0.25 0.20 0.14 0.067 0.091 0.072 0.080	0.15 0.18 0.16 0.16 0.016 0.025 0.020 0.015 0.005 0.008 0.006 0.009	065-92-0001R
Spain, 1992 (Trocadero)	EC	0.022	0.0022 -0.0044	4	0 7 14	1.6 1.7 1.9 1.1 0.14 0.10 0.17 0.18 0.037 0.033 0.027 0.027	0.17 0.18 0.20 0.12 0.014 0.011 0.016 0.018 0.003 0.004 0.003 0.002	065-92-0002R
Spain, 1992 (Trocadero)	EC	0.043	0.0043 -0.0086	4	0 7 14	0.86 1.9 1.8 1.1 0.12 0.15 0.093 0.14 0.023 0.022 0.031 0.020	0.089 0.19 0.18 0.11 0.010 0.015 0.009 0.014 0.002 NQ 0.002 NQ	065-92-0002R

Country, year (variety)	Application				PHI, days	Residues, mg/kg[1]		Ref
	Form	kg ai/ha	kg ai/hl	No.		B_{1a} + 8,9-Z-B_{1a}	B_{1b} + 8,9-Z-B_{1b}	
LEAF LETTUCE								
Spain, 1992 (Summer Blond)	EC	0.022	0.0022 -0.0044	4	0 7 14	0.20 0.16 0.17 0.19 0.007 0.008 0.009 0.004 NQ ND ND NQ	0.021 0.018 0.018 0.021 ND (4) ND (4)	065-92-0003R
Spain, 1992 (Summer Blond)	EC	0.043	0.0043 -0.0086	4	0 7 14	0.36 0.44 0.30 0.46 0.025 0.025 0.028 0.024 0.004 0.005 0.002 0.003	0.041 0.045 0.030 0.053 0.002 ND 0.002 NQ ND (4)	065-92-0003R
Spain, 1992 (Romaine, Inverna)	EC	0.022	0.0022 -0.0044	4	0 7 14	0.21 0.17 0.18 0.24 0.005 0.004 0.003 0.004 0.002 NQ ND ND	0.025 0.019 0.021 0.028 ND (4) ND (4)	065-92-0004R
Spain, 1992 (Romaine, Inverna)	EC	0.043	0.0043 -0.0086	4	0 7 14	0.40 0.22 0.54 0.42 0.006 0.005 0.005 0.005 0.003 0.002 NQ 0.002	0.047 0.025 0.061 0.048 ND (4) ND (4)	065-92-0004R

1 NQ: not quantified; detected but <0.002 mg/kg
ND: not detected, <0.001 mg/kg
[2]Because of the seasonal restriction on use specified in Netherlands GAP the month of the final application is reported.
gl: glasshouse

Table 9. Abamectin residues in potatoes resulting from foliar applications in supervised trials in Brazil and the USA.

Country, year (variety)	Application				PHI, days	Residues, mg/kg[1]		Ref
	Form	kg ai/ha	Kg ai/hl	No.		B_{1a} + 8,9-Z-B_{1a}	B_{1b} + 8,9-Z-B_{1b}	
Brazil (SP), 1994 (Achat)	EC	0.018	0.0023	4	0 3 7	<0.005 <0.005 <0.005 <0.005 <0.005 <0.005		015-94-9050R
Brazil (SP), 1994 (Achat)	EC	0.036	0.0045	4	0 3 7	<0.005 <0.005 <0.005 <0.005 <0.005 <0.005		015-94-9050R
Brazil (SP), 1994 (Achat)	EC	0.018	0.0023	4	0 3 7	<0.005 <0.005 <0.005 <0.005 <0.005 <0.005		015-94-9052R
Brazil (SP), 1994 (Achat)	EC	0.036	0.0045	4	0 3 7	<0.005 <0.005 <0.005 <0.005 <0.005 <0.005		015-94-9052R
Brazil (SP), 1994 (Bintje)	EC	0.018	0.0023	4	0 3 7	<0.005 <0.005 <0.005 <0.005 <0.005 <0.005		015-94-9051R
Brazil (SP), 1994 (Bintje)	EC	0.036	0.0045	4	0 3 7	<0.005 <0.005 <0.005 <0.005 <0.005 <0.005		015-94-9051R
USA (FL), 1992 (Atlantic)	EC	0.112	0.025	6	0 3 7	ND ND ND ND ND ND	ND ND ND ND ND ND	001-92-0038R
USA (FL), 1992 (Atlantic)	EC	0.112 + oil	0.025	6	0 3 7	ND ND ND ND ND ND	ND ND ND ND ND ND	001-92-0038R
USA (NY), 1992 (Katahdin)	EC	0.11	0.040	6	0 3 7	ND ND ND ND ND ND	ND ND ND ND ND ND	618-936-3671 001-92-5017R
USA (NY), 1992 (Katahdin)	EC	0.11 + oil	0.040	6	0 3 7	ND ND ND ND ND ND	ND ND ND ND ND ND	618-936-3671 001-92-5017R
USA (OR), 1992 (Russet)	EC	0.11	0.040	6	0 3 7	ND ND ND ND ND ND	ND ND ND ND ND ND	618-936-3671 001-92-5019R

Country, year (variety)	Application				PHI, days	Residues, mg/kg[1]		Ref
	Form	kg ai/ha	Kg ai/hl	No.		B_{1a} + 8,9-Z-B_{1a}	B_{1b} + 8,9-Z-B_{1b}	
USA (OR), 1992 (Russet)	EC	0.11 + oil	0.040	6	0 3 7	ND ND ND ND ND ND	ND ND ND ND ND ND	618-936-3671 001-92-5019R
USA (PA), 1992 (Katahdin)	EC	0.11	0.040	6	0 3 7	ND ND ND ND ND ND	ND ND ND ND ND ND	618-936-3671 001-92-5018R
USA (PA), 1992 (Katahdin)	EC	0.11 + oil	0.040	6	0 3 7	ND ND ND ND ND ND	ND ND ND ND ND ND	618-936-3671 001-92-5018R
USA (CA), 1993 (Norkotah)	EC	0.021 + oil	0.0056	6	0 14	ND ND ND ND	ND ND ND ND	001-93-5006R
USA (CA), 1993 (Red LaSoda)	EC	0.021 + oil	0.0059	6	0 14	ND ND ND ND	ND ND ND ND	001-93-5005R
USA (FL), 1993 (Red La Soda)	EC	0.021 + oil	0.0045	6	0 14	ND ND ND ND	ND ND ND ND	618-936-3671 001-93-0002R
USA (ID), 1993 (Russet Burbank)	EC	0.020 + oil	0.045	6	0 14	ND ND ND ND	ND ND ND ND	001-93-1004R
USA (ID), 1993 (Russet Burbank)	EC	0.021 + oil	0.011	6	0 14	ND ND ND ND	ND ND ND ND	001-93-1005R
USA (MD), 1993 (Superior)	EC	0.021 + oil	0.0076	6	0 14	ND ND ND ND	ND ND ND ND	001-93-7000R
USA (MI), 1993 (Snowden)	EC	0.021 + oil	0.011	6	0 14	ND ND ND ND	ND ND ND ND	001-93-1007R
USA (NY), 1993 (Katahdin)	EC	0.021 + oil	0.014	6	0 14	ND ND ND ND	ND ND ND ND	001-93-7001R
USA (WA), 1993 (Russet Burbank)	EC	0.021 + oil	0.0044	6	0 14	ND ND ND ND	ND ND ND ND	001-93-5004R
USA (CO), 1994 (Russet Nugget)	EC	0.11	0.030	6	0 14	ND ND ND ND	ND ND ND ND	001-94-1022R
USA (ND), 1994 (Norchip)	EC	0.021 + oil	0.014	6	0 14	ND ND ND ND	ND ND ND ND	001-94-1017R
USA (FL), 1993 (FL 1625)	EC	0.021 + oil	0.011	6	0 14	ND ND ND ND	ND ND ND ND	001-93-7002R

[1] ND: not detected, <0.002 mg/kg

Table 10. Abamectin residues in hops resulting from foliar applications in supervised trials in Germany and the USA. Double-underlined residues are from treatments according to GAP and are valid for estimating maximum residue levels and STMRs.

Country, year (variety)	Application				PHI, days	Residues, mg/kg [1,2]		Ref
	Form	kg ai/ha	kg ai/hl	No.		B_{1a} + 8,9-Z-B_{1a}	B_{1b} + 8,9-Z-B_{1b}	
Germany, 1994 (Hallertau Mittelfruw)	EC	0.023	0.0022 +0.0011	2	0 14 21 28 21 28	fh 0.11 0.12 fh 0.003 0.003 fh ND NQ fh NQ NQ dh 0.004 0.005 dh ND ND	fh 0.010 0.012 fh ND ND fh ND ND fh ND ND dh ND ND dh ND ND	072-94-0008R
Germany, 1994 (Hallertau Mittelfruw)	EC	0.023 + adj	0.0022 +0.0011	2	0 14 21 28 21 28	fh 0.24 0.31 fh 0.003 0.004 fh 0.002 0.003 fh 0.002 ND dh 0.004 0.007 dh ND ND	fh 0.025 0.030 fh ND ND fh ND ND fh ND ND dh ND ND dh ND ND	072-94-0008R

Country, year (variety)	Application				PHI, days	Residues, mg/kg [1,2]		Ref
	Form	kg ai/ha	kg ai/hl	No.		B_{1a} + 8,9-Z-B_{1a}	B_{1b} + 8,9-Z-B_{1b}	
Germany, 1994 (Hallertauer Tradition)	EC	0.022	0.0031 +0.0015	2	0 14 21 28 21 28	fh 0.23 0.21 fh 0.011 0.008 fh 0.008 0.006 fh 0.006 0.006 dh 0.029 0.031 dh 0.022 0.020	fh 0.026 0.022 fh ND ND fh ND ND fh ND ND dh ND NQ dh ND ND	072-94-0005R
Germany, 1994 (Hallertauer Tradition)	EC	0.022 + adj	0.0031 +0.0015	2	0 14 21 28 21 28	fh 0.44 0.82 fh 0.022 0.016 fh 0.010 0.012 fh 0.007 0.006 dh 0.031 0.024 dh 0.022 0.012	fh 0.049 0.087 fh 0.002 ND fh ND ND fh ND ND dh ND ND dh ND ND	072-94-0005R
Germany, 1994 (Perle)	EC	0.023	0.0031 +0.0015	2	0 14 21 28 21 28	fh 0.25 0.29 fh 0.015 0.011 fh 0.005 0.006 fh 0.006 0.005 dh 0.034 0.029 dh 0.025 0.020	fh 0.026 0.031 fh ND ND fh ND ND fh ND ND dh NQ ND dh ND ND	072-94-0007R
Germany, 1994 (Perle)	EC	0.023 + oil	0.0031 +0.0015	2	0 14 21 28 21 28	fh 0.20 0.35 fh 0.016 0.009 fh 0.010 0.006 fh 0.005 0.006 dh 0.035 0.036 dh 0.030 0.025	fh 0.021 0.037 fh ND ND fh ND ND fh ND ND dh NQ NQ dh ND ND	072-94-0007R
Germany, 1994 (Perle)	EC	0.023	0.0031 +0.0015	2	0 14 21 28 21 28	fh 0.23 0.31 fh 0.011 0.018 fh 0.008 0.10 fh 0.003 0.003 dh 0.043 0.041 dh 0.017 0.022	fh 0.024 0.031 fh ND ND fh ND ND fh ND ND dh NQ NQ dh ND ND	072-94-0006R
Germany, 1994 (Perle)	EC	0.023 + oil	0.0031 +0.0015	2	0 14 21 28 21 28	fh 0.40 0.28 fh 0.014 0.011 fh 0.010 0.013 fh 0.006 0.005 dh 0.046 0.044 dh 0.017 0.012	fh 0.036 0.027 fh ND ND fh ND ND fh ND ND dh NQ NQ dh ND ND	072-94-0006R
Germany, 1996 (Hallertauer Frühreifer)	EC	0.023	0.0011	2	0 29 29	ih 0.15 0.14 fh ND NQ dh 0.011 0.012	ih 0.010 0.009 fh ND ND dh ND ND	072-96-0011R 96092
Germany, 1996 (Hersbrucker)	EC	0.023	0.0011	2	0 30 30	ih 0.17 0.28 fh ND ND dh ND ND	ih 0.011 0.019 fh ND ND dh ND ND	072-96-0012R 96092
Germany, 1996 (Northern Brewer)	EC	0.023	0.0009	2	0 28 28	ih 0.12 0.10 fh ND NQ dh ND NQ	ih 0.008 0.007 fh ND ND dh ND ND	072-96-0014R 96092

Country, year (variety)	Application				PHI, days	Residues, mg/kg [1,2]		Ref
	Form	kg ai/ha	kg ai/hl	No.		B_{1a} + 8,9-Z-B_{1a}	B_{1b} + 8,9-Z-B_{1b}	
Germany, 1996 (Perle)	EC	0.023		2	0 30 30	ih 0.23 0.22 fh NQ 0.008 dh 0.009 0.011	ih 0.015 0.015 fh ND ND dh ND ND	072-96-0013R 96092
USA (ID), 1994 (Galena)	EC	0.021	0.0045	2	0 28	dh 0.67 0.59 dh 0.055 0.057	dh 0.072 0.064 dh NQ NQ	001-94-1007R 618-936-94035
USA (OR), 1994 (Nugget)	EC	0.022	0.0045	2	0 28	dh 0.97 0.81 dh 0.009 0.015	dh 0.096 0.081 dh ND ND	001-94-1008R 618-936-94035
USA (WA), 1994 (Cluster)	EC	0.021	0.0045	2	0 28	dh 0.16 0.15 dh 0.017 0.023	dh 0.015 0.015 dh ND ND	001-94-1006R 618-936-94035
USA (WA), 1994 (Galena)	EC	0.021	0.0045	2	0 27	dh 0.59 0.73 dh 0.044 0.078	dh 0.059 0.073 dh NQ 0.008	001-94-1005R 618-936-94035

[1] NQ: not quantified; detected but <0.005 mg/kg.
ND: not detected, <0.0025 mg/kg.
[2]ih: immature hops, fh: fresh hops, dh: dry hops.
adj: adjuvant - a non-ionic surfactant containing lecithin derived from soya bean oil.

Table 11. Interpretation Table for abamectin residues in apples from trials in Table 3. GAP and trial conditions are compared for treatments considered valid for estimating maximum residue levels and STMRs.

	Use pattern				Trial	Residues, mg/kg, abamectin
	kg ai/ha	kg ai/hl	No of appl	PHI, days		
Australian GAP	0.014	0.0014	1	14		
Australian trial	0.014	0.0007	1	14	114-95-0003R	0.005
Australian trial	0.014	0.0007	1	14	114-95-0002R	<0.002
Australian trial	0.014	0.0008	1	14	114-95-0001R	0.003
NZ GAP	0.027	0.00068	1	14		
NZ trial	0.027	0.0014	2	14	115-94-0005R	0.004
NZ trial	0.027	0.0014	2	14	115-94-0004R	0.007
US GAP	0.026	0.0007	2	28		
US trial	0.028	0.0007	2	28	001-90-5016R	0.003
US trial	0.027	0.0008	2	28	001-92-6012R	0.003
US trial	0.027	0.0010	2	28	001-91-6024R	<0.001
US trial	0.028	0.0010	2	28	001-90-5018R	0.006
US trial	0.027	0.0011	2	28	001-91-1021R	<0.002
US trial	0.027	0.0036	2	28	001-91-1024R	0.002
US trial	0.027	0.0037	2	28	001-91-1023R	0.003
US trial	0.027	0.0038	2	28	001-91-6016R	0.012
US trial	0.027	0.0038	2	28	001-91-3000R	0.007
US trial	0.026	0.0071	2	28	001-92-0026R	0.003
US trial	0.027	0.0072	2	28	001-92-0027R	<0.002
US trial	0.027	0.0072	2	28	001-92-3020R	0.004
US trial	0.027	0.0072	2	28	001-92-1018R	<0.002
US trial	0.027	0.0081	2	28	001-92-1014R	<0.001

Table 12. Interpretation table for abamectin residues in pears from trials in Table 4 and the 1992 Evaluations. GAP and trial conditions are compared for treatments considered valid for estimating maximum residue levels and STMRs.

	Use pattern				Trial	Residues, mg/kg, abamectin
	kg ai/ha	kg ai/hl	No of appl	PHI, days		
Argentinian GAP	0.027	0.0014	4	14		
Argentinian trial	0.027	0.00063	3	14	1992 ref 58	<0.005
French GAP	0.023	0.0014	4	15		
French trial	0.027	0.0014	3	14	1992 ref 196	<0.002
Italian GAP	0.027	0.0014	2	14		
Italian trial	0.027	0.0014	3	14	1992 ref 139	<0.005
Italian trial	0.027	0.0014	3	14	1992 ref 139	<0.002
Italian trial	0.027	0.0014	3	14	1992 ref 140	<0.005
Italian trial	0.027	0.0014	3	14	1992 ref 198	<0.005
US GAP	0.026		2	28		
US trial	0.027		2	21	001-92-6016R	0.004
US trial	0.027		2	21	001-92-6017R	0.006
US trial	0.027		2	21	001-92-6018R	0.009
US trial	0.027		2	21	001-92-6019R	0.011

Table 13. Interpretation table for abamectin residues in melons from trials in Tables 5 and 6. GAP and trial conditions are compared for treatments considered valid for estimating maximum residue levels and STMRs.

	Use pattern				Trials	Residues, mg/kg abamectin
	kg ai/ha	kg ai/hl	No of appl	PHI, days		
Spanish GAP	0.022	0.0011	3	3		
Spanish trial	0.022	0.0022	4 gl	3	065-91-0003R	<0.002
Spanish trial	0.022	0.0022	4 gl	3	065-91-0004R	<0.002
French trial	0.023	0.0028	4	3	066-91-0003R	<0.002
French trial	0.023	0.0032	4	3	066-91-0004R	<0.005
French trial	0.023	0.0030	4	3	066-91-0005R	<0.005
US GAP	0.021		3	7		
US trial	0.021	0.023	4	7	001-91-1026R	<0.002
US trial	0.021	0.011	4	7	001-91-1027R	<0.002
US trial	0.021	0.023	4	7	001-91-6011R	<0.002
US trial	0.021	0.011	4	7	001-92-0019R	<0.002
US trial	0.021	0.011	4	7	001-92-0020R	<0.002
US trial	0.021	0.010	4	7	001-92-0021R	<0.002
US trial	0.021	0.010	4	7	001-92-1001R	<0.002
US trial	0.022	0.011	4	7	001-92-3014R	<0.002
US trial	0.022	0.011	5	7	001-92-6013R	<0.002

gl: glasshouse

Table 14. Interpretation table for abamectin residues in cucumbers and gherkins from trials in Table 6 and the 1992 Evaluations. GAP and trial conditions are compared for treatments considered valid for estimating maximum residue levels and STMRs.

	Use pattern				Trials	Residues, mg/kg abamectin
	kg ai/ha	kg ai/hl	No of appl	PHI, days		
CUCUMBER						
US GAP	0.021		3	7		
US trial	0.021	0.010	4	7	001-92-0030R	<0.002
US trial	0.022	0.012	4	7	001-92-1019R	<0.002
US trial	0.022	0.012	4	7	001-92-3018R	<0.002
US trial	0.021	0.011	4	7	001-92-6015R	<0.005
German GAP	0.023		5 gl	3		
French trial	0.023	0.0011	4 gl	3	1992 ref 17	<0.002
French trial	0.023	0.0011	4 gl	3	1992 ref 17	<0.005
Spanish GAP	0.022	0.0011	3	3		
Spanish trial	0.023	0.004	5 gl	3	1992 ref 13	0.006
Spanish trial	0.023	0.007	5 gl	3	1992 ref 13	0.008
Italian trial	0.023	0.0023	5	3	1992 ref 169	<0.005
Italian trial	0.023	0.0023	4 gl	3	1992 ref 16	<0.005
Italian trial	0.023	0.0023	4	3	1992 ref 16	<0.002
Netherlands GAP	0.023	0.0009	5 gl	3		
Netherlands trial	0.023	0.0011	4 gl	3	1992 ref 161	0.008
Netherlands trial	0.023	0.0011	5 gl	3	1992 ref 161	0.007
GHERKIN						
Netherlands GAP	0.023	0.0009	5 gl	3		
Netherlands trial	0.023	0.0011	5 gl	3	1992 ref 165	<0.002
Netherlands trial	0.023	0.0011	5 gl	3	1992 ref 165	<0.002

gl: glasshouse

Table 15. Interpretation table for abamectin residues in tomatoes from trials in Table 7 and the 1992 Evaluations. GAP and trial conditions are compared for treatments considered valid for estimating maximum residue levels and STMRs.

	Use pattern				Trials	Residues, mg/kg abamectin
	kg ai/ha	kg ai/hl	No of appl	PHI, days		
Argentinian GAP	0.022	0.0013	9	3		
Argentinian trial	0.020	0.0009	7	3	1992 ref 60	<0.005
Argentinian trial	0.027	0.0009	5	3	1992 ref 61	<0.002
Argentinian trial	0.028	0.0018	9	3	1992 ref 62	<0.002
Brazil GAP	0.022	0.0018		3		
Brazil trial	0.027	0.0018	10	7[1]	1992 ref 125	0.017
Brazil trial	0.023	0.0018	10	3	1992 ref 126	<0.005
Brazil trial	0.028	0.0036	6	3	1992 ref 126	<0.005
German GAP	0.023		5 gl	3		
French trial	0.024	0.0005	5 gl	3	1992 ref 123	<0.005
French trial	0.023	0.0005	5 gl	3	1992 ref 127	<0.002
French trial	0.020	0.0007	10 gl	3	1992 ref 128	<0.002
Italian GAP	0.022		2	7		
Italian trial	0.022	0.0011	10	7	1992 ref 209	<0.002
Italian trial	0.022	0.0011	10	7	1992 ref 210	<0.002
Netherlands GAP	0.023	0.0009	4 gl	3		
Netherlands trial	0.023	0.0011	4 gl	3	070-93-0001R	0.009
Netherlands trial	0.023	0.0011	4 gl	3	070-93-0002R	0.007
Netherlands trial	0.023	0.0011	4 gl	7[1]	070-93-0004R	0.007
Netherlands trial	0.023	0.0011	4 gl	3	070-93-0005R	0.017

	Use pattern				Trials	Residues, mg/kg abamectin
	kg ai/ha	kg ai/hl	No of appl	PHI, days		
Netherlands trial	0.023	0.0011	4 gl	3	070-93-0006R	0.012
Netherlands trial	0.023	0.0011	4 gl	3	070-93-0003R	0.012
Netherlands trial	0.023	0.0011	5 gl	3	1992 ref 217	0.008
Netherlands trial	0.023	0.0011	5 gl	3	1992 ref 218	0.005
Spanish GAP	0.022	0.0011		3		
Spanish trial	0.019	0.0011	10 gl	3 (f)	1992 ref 130	<0.005 (<0.005)
Spanish trial	0.019	0.0005	10 gl	3 (f)	1992 ref 131	0.009 (<0.005)
Spanish trial	0.027	0.0011	10	3	1992 ref 132	<0.005
Spanish trial[2]	0.015	0.0011	10	3	1992 ref 129	<0.005
US GAP	0.021		3	7		
US trial	0.022	0.024	10	7	1992 ref 124	<0.002
US trial	0.022	0.0036	10	3	1992 ref 124	<0.002
US trial	0.022	0.012	10	3	1992 ref 124	<0.002
US trial	0.022	0.0047	10	3	1992 ref 124	<0.002
US trial	0.022	0.017	10	3	1992 ref 124	<0.002
US trial	0.022	0.0078	10	3	1992 ref 124	<0.002
US trial	0.022	0.0044	10	3	1992 ref 124	<0.002
US trial	0.022	0.0043	10	7	1992 ref 124	<0.002
US trial	0.022	0.012	10	3	1992 ref 124	<0.005
US trial	0.022	0.0024	10	7	1992 ref 124	<0.005
US trial	0.022	0.0053	12	7	1992 ref 183	<0.002
US trial	0.022	0.0068	10	5	1992 ref 184	<0.002
US trial	0.022	0.0043	10	7	1992 ref 185	<0.005
US trial	0.022	0.0059	10	7	1992 ref 186	<0.002
US trial	0.022	0.0023	10	7	1992 ref 187	<0.005
US trial	0.022	0.0047	8	3	1992 ref 188	<0.002
US trial	0.022	0.0047	10	7	1992 ref 191	0.005
US trial	0.022	0.024	10	5	1992 ref 193	<0.002

gl: glasshouse
[1]Residue on day 7 higher than on day 3
[2]The companion trial at 0.030 kg ai/ha resulted in residues of 0.007 mg/kg on day 3

Table 16. Interpretation table for abamectin residues in lettuce from trials in Table 8 and the 1992 Evaluations. GAP and trial conditions are compared for treatments considered valid for estimating maximum residue levels and STMRs.

	Use pattern				Trials	Residues, mg/kg abamectin
	kg ai/ha	kg ai/hl	No of appl	PHI, days		
Head Lettuce						
Netherlands GAP	0.014	0.0009	4	14		
Netherlands trial	0.014	0.0014	4 gl	14	070-93-0007R	0.025
Netherlands trial	0.014	0.0014	4 gl	14	070-93-0008R	0.029
Netherlands trial	0.014	0.0014	4 gl	14	070-94-0002R	0.029
Netherlands trial	0.014	0.0014	4 gl	14	070-94-0001R	0.016
French GAP	0.009		4	7 or 14		
French trial	0.0113	0.0028	4	6	066-92-0001R	<0.001
French trial	0.0113	0.0023	4	7	066-92-0003R	0.004
French trial	0.0113	0.0019	4	7	066-92-0002R	0.023
Spanish GAP	0.022	0.0018	3	14		
Spanish trial	0.022	0.0044	4	14	065-92-0001R	0.028
Spanish trial	0.022	0.0044	4	14	065-92-0002R	0.040
French trial	0.025	0.0056	4	13	066-92-0001R	<0.002
French trial	0.0225	0.0045	4	14	066-92-0003R	0.005
French trial	0.0225	0.0038	4	14	066-92-0002R	0.013
US GAP	0.021		3	7		

	Use pattern				Trials	Residues, mg/kg abamectin
	kg ai/ha	kg ai/hl	No of appl	PHI, days		
US trial	0.022	0.0047	7	7	1992 ref 154	0.005
US trial	0.022	0.0047	7	7	1992 ref 154	0.007
US trial	0.022	0.023	8	7	1992 ref 151	<0.002
US trial	0.022	0.023	8	7	1992 ref 151	<0.002
US trial	0.022	0.0047	8	7	1992 ref 155	<0.002
US trial	0.022	0.0047	8	7	1992 ref 155	<0.002
US trial	0.022	0.049	8	7	1992 ref 159	<0.002
US trial	0.022	0.0064	9	7	1992 ref 159	0.030
US trial	0.022	0.0064	9	7	1992 ref 159	0.026
US trial	0.022	0.0059	8	7	1992 ref 159	<0.002
US trial	0.022	0.0059	8	7	1992 ref 159	<0.002
US trial	0.022	0.0078	8	7	1992 ref 159	<0.002
US trial	0.022	0.0078	8	7	1992 ref 159	<0.002
US trial	0.022	0.024	8	7	1992 ref 162	<0.002
US trial	0.022	0.024	8	7	1992 ref 162	<0.002
US trial	0.022	0.036	9	7	1992 ref 162	0.006
US trial	0.022	0.036	9	7	1992 ref 162	0.027
US trial	0.022	0.071	6	7	1992 ref 162	<0.002
US trial	0.022	0.071	6	7	1992 ref 162	<0.002
Leaf lettuce						
Spanish GAP	0.022	0.0018	3	14		
Spanish trial	0.022	0.0044	4	14	065-92-0003R	<0.002
Spanish trial	0.022	0.0044	4	14	065-92-0004R	0.002

gl: glasshouse

Table 17. Interpretation table for abamectin residues in dry hops from trials in Table 10. GAP and trial conditions are compared for treatments considered valid for estimating maximum residue levels and STMRs.

	Use pattern				Trials	Residues, mg/kg abamectin
	kg ai/ha	kg ai/hl	No of appl	PHI, days		
German GAP	0.023	0.0009	2	28		
German trial	0.023	0.0022	2	28	072-94-0008R	<0.003
German trial	0.023	0.0022	2	28	072-94-0008R	<0.003
German trial	0.022	0.0031	2	28	072-94-0005R	<0.003
German trial	0.022	0.0031	2	28	072-94-0005R	0.022
German trial	0.023	0.0031	2	28	072-94-0007R	0.025
German trial	0.023	0.0031	2	28	072-94-0007R	0.030
German trial	0.023	0.0031	2	28	072-94-0006R	0.022
German trial	0.023	0.0031	2	28	072-94-0006R	0.017
German trial	0.023	0.0011	2	29	072-96-0011R	0.012
German trial	0.023	0.0011	2	30	072-96-0012R	<0.003
German trial	0.023	0.0009	2	28	072-96-0014R	<0.005
German trial	0.023		2	30	072-96-0013R	0.011
US GAP	0.022		2	28		
US trial	0.021	0.0045	2	28	001-94-1007R	0.062
US trial	0.022	0.0045	2	28	001-94-1008R	0.015
US trial	0.021	0.0045	2	28	001-94-1006R	0.023
US trial	0.021	0.0045	2	27	001-94-1005R	0.086

FATE OF RESIDUES IN STORAGE AND PROCESSING

The Meeting received information on the fate of abamectin during the processing of apples, pears, potatoes and hops.

A processing study on apples by Morneweck (1992) was reviewed by the 1992 JMPR and the residue data are summarized in the 1992 Residue Evaluations. Processing factors are recorded in Table 18 and were calculated from the B_{1a} rather than the B_{1a} + B_{1b} residues because B_{1b} was undetectable in the initial apples and its inclusion in the calculation would have added an extra error to the processing factor. Avermectin B_{1b} constitutes about 10% of the total residue and probably behaves in the same way as B_{1a} in processing. In cases where no residue was detectable in the processed commodity the processing factor is reported as 0 with a “less than” factor in parentheses calculated from the LOD.

The results suggest that abamectin residues are on the peel only and are reasonably stable during hot drying of the pomace. In products such as juice and apple sauce, which contain no peel, residues are not detectable.

Table 18. Processing factors for apple products (Morneweck, 1992), calculated as the residue levels of B_{1a} in the processed commodities divided by its level in the initial unwashed apples. B_{1a} includes avermectin B_{1a} and its photoisomer 8,9-*Z*-avermectin B_{1a}.

Commodity	Processing factor
Apples, whole unwashed	
Apples, peeled and cored	0 (<0.12)
Apple juice, raw	0 (<0.062)
Apple juice, clarified	0 (<0.062)
Pomace, wet	4.9
Pomace, dry	17.3
Pomace, rehydrated	14.8
Apple sauce	0 (<0.12)

Abamectin was applied twice at 0.027 kg ai/ha with an interval of 14 days to Bartlett pears which were harvested 1-2 hours after the second application for processing (Johnson 1993). The fruit were processed in 11-12 kg lots into canned pear halves and pear purée (Figure 1). Residue levels in the various commodities and calculated processing factors are shown in Table 9.

The processing factors were again calculated from the B_{1a} rather than the B_{1a}+B_{1b} residues because the initial B_{1b} levels were only slightly above the LOD. The processing factors are essentially zero because no residues were detected in any of the final processed commodities or even their immediate precursors. It is likely that vigorous washing and peeling would effectively remove a surface residue such as abamectin.

Figure 1. Processing of pears (Johnson, 1993).

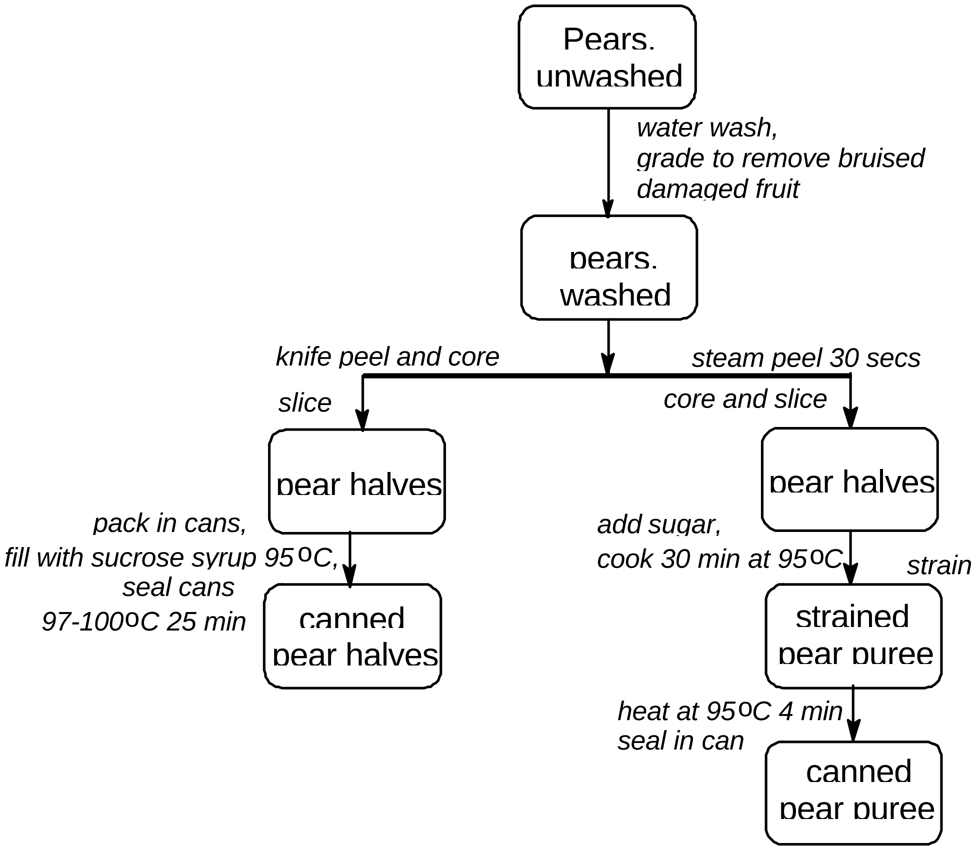


Table 19. Abamectin residues in canned pears and purée produced from Bartlett pears treated twice with abamectin (+ oil) at 0.027 kg ai/ha and harvested 1-2 hours after the second application in the USA (Johnson, 1993). Processing factors were calculated as the residue level of B_{1a} in the processed commodities divided by its level of B_{1a} in the initial unwashed pears.

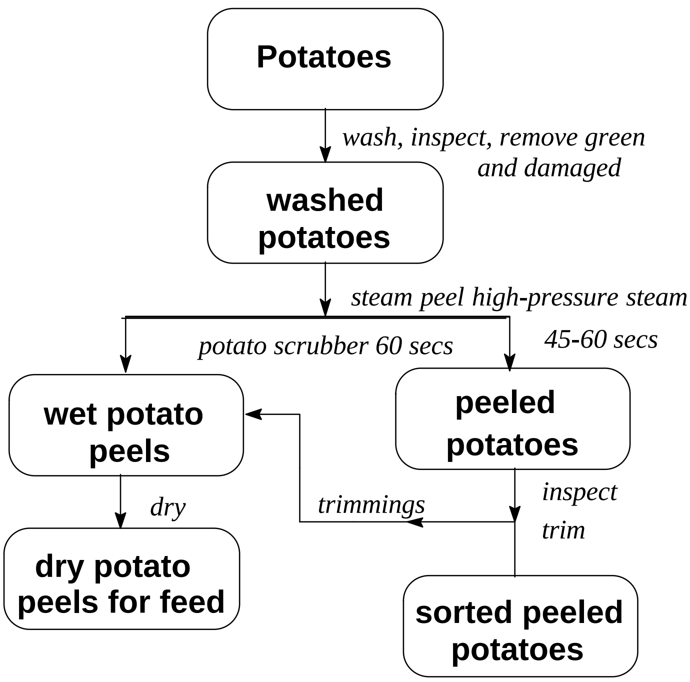
Commodity	Residues, mg/kg		Processing factor
	B_{1a} + 8,9-Z-B_{1a}	B_{1b} + 8,9-Z-B_{1b}	
Unwashed pears	0.0216	0.0025	
Pear halves (knife peeled and cored)	<0.001	<0.001	
Canned pear halves	<0.001	<0.001	0 (<0.046)
Unwashed pears	0.0208	0.0025	
Pear halves (steam peeled and cored)	<0.001	<0.001	
Pear purée, strained	<0.001	<0.001	0 (<0.048)
Pear purée, canned	<0.001	<0.001	0 (<0.048)

Abamectin was applied at 0.11 kg ai/ha (5 times the label rate) on 6 occasions to the foliage of potatoes in a processing trial in the USA (Colorado) (001-94-1022). Potatoes (135 kg) were harvested 14 days after the final application and processed to peeled potatoes and potato peels (Englar, 1994b). The variety, Russet Nugget, is especially grown for processing.

The process (Figure 2) was designed to simulate commercial practice. The first stage was tub washing for 5-10 minutes to simulate flume washing. The next stage was inspection and sorting to remove cull potatoes. The potatoes were then treated with high-pressure steam followed by a scrubber to remove the skin loosened by the steam. The final stage was inspection and trimming of damaged or unsuitable tissue with the trimmings added to the peel. The peel was hydraulically pressed, dried to less than 10% moisture and then hammer milled. In a commercial operation, it would be used to feed cattle.

Kvaternick *et al.* (1995) reported that no abamectin residues were detected (<0.002 mg/kg each of B_{1a} and B_{1b}) in the unwashed and washed potatoes and the wet potato peels. Because no residues were detected in these commodities the dry peels and peeled potatoes were not analysed.

Figure 2. Processing of potatoes (Englar, 1994b).



Englar (1994a) described the commercial processing of <u>hops</u> and its laboratory simulation. In the commercial operation freshly harvested hop cones are dried to approximately 7-10% moisture and then ground to release the bitter principles from the lupulin glands. The ground hops are extracted repeatedly with hexane and the extracted (spent) hops are pressed, dried and pelletized. In the laboratory simulation the dried and ground hops (1.3 kg) were placed in a glass column (135 × 10.2 cm) and extracted with hexane, circulated at 0.5 l/min, for approximately 3.5 hours. The hops were finally extracted with fresh hexane and dried overnight at ambient temperature to produce the spent hops. The extract contains flavour components and is used in the brewing industry while the spent hops become a minor feed commodity.

Hops harvested 27 days after the final treatment in trial 001-94-1005R (Table 10) were processed. The abamectin residues decreased by 19 and 40% (Table 11).

Table 20. Abamectin residues depletion in dry and spent hops (Englar 1994a). See Table 10 for deatails of treatment and harvesting of the hops in trial 001-94-1005R used for processing.

Abamectin residues, mg/kg				Processing factor
Dried hops		Spent hops		
B_{1a}	B_{1b}	B_{1a}	B_{1b}	
0.086	0.0075	0.069	0.0063	0.81
0.082	0.0073	0.049	<0.005	0.60

Johnson (1995b) measured the abamectin residues in fresh and dry hops from supervised residue trials in Germany (Table 10). The hops were dried for 6 hours at 62°C in kilns; the weight of the dried product was approximately 20% of the fresh weight. Processing (drying) factors were calculated for avermectin B_{1a} for 13 cases where the residues were above the LOQ in both the fresh and dried hops. Avermectin B_{1b} was not included in the calculation because it was not detected. The processing factors (in rank order) were 2.22, 2.53, 2.55, 2.64, 3.48, 3.97, 4.22, 4.43, 4.56, 4.60, 5.15, 5.79 and 6.98. The mean was 4.09, so on average about 80% of the abamectin survived the drying process.

Residues in the edible portion of food commodities

Abamectin residues were not detected in the pulp of melons from supervised trials in Brazil where treatment was at 0.014 and 0.029 kg ai/ha. The melons were harvested 0, 3 and 7 days after the final treatment.

Abamectin residues were not detected (<0.001 mg/kg) in peeled and cored apples, raw juice, clarified juice or apple sauce produced from treated apples, or in pear halves or purée from treated pears.

NATIONAL MAXIMUM RESIDUE LIMITS

The Meeting was aware that the following MRLs had been established for abamectin.

Country	MRL, mg/kg	Commodity
Argentina	0.01	Cotton seed, citrus fruit, cucumber, melon, pear, pepper, tomato, watermelon
	0.02	Strawberry
	0.05	Celery
Australia	0.005	Cattle meat, milk
	0.01	Apple, cotton seed, pear, tomato
	0.02	Strawberry
	0.1	Cattle edible offal, cattle fat
Brazil	0.001	Milk
	0.005	Citrus fruit, cotton seed, potato
	0.01	Apple, bell pepper, cucumber, meat, tomato, watermelon
	0.02	Strawberry
Canada	0.02	Apple, pear
France	0.01	Apple, pear
	0.02	Courgette, cucumber, egg plant, endive, lambs' lettuce, lettuce, melon, pepper, strawberry, tomato
Germany	0.02	Cucumber, egg plant, sweet pepper, strawberry, tomato, zucchini
	0.05	Hops

Country	MRL, mg/kg	Commodity
Israel	0.005	Cotton seed, milk
	0.01	Apple, celery, citrus fruit, cucurbits, egg plant, pear, potato, strawberry, tomato
	0.02	Pepper
Italy	0.01	Pear, tomato
Mexico	0.005	Cotton seed
	0.01	Tomato
	0.02	Citrus, strawberry
	0.05	Celery
Netherlands	0.01	Cucumber, gherkin, zucchini
	0.02	Egg plant, pepper, tomato
	0.05	Endive, iceberg lettuce, lettuce
New Zealand	note[1]	Apple, pear, strawberry, tomato
South Africa	0.01	Apple, pear
	0.02	Strawberry
	0.05	Cotton seed, tomato
Spain	0.01	Celery, citrus fruit, cotton seed, cucurbits, lettuce, pear, pepper, strawberry, tomato
Switzerland	0.01	Cucumber, egg plant, pear, strawberry, sweet pepper, tomato
USA	0.005	Almond nutmeat, cotton seed, cucurbits (cucumbers, squashes, melons), milk, potato, walnuts
	0.01	Bell pepper, tomato
	0.015	Cattle, fat
	0.02	Apple, cattle meat, cattle meat byproducts, citrus whole fruit, pear, strawberry
	0.05	Celery, head lettuce
	0.07	Tomato pomace
	0.1	Apple wet pomace, almond hulls, citrus dried pulp, citrus oil,
	0.2	Hops dried

[1]Not required as residue levels are <0.01 mg/kg when product is used according to label instructions

APPRAISAL

Abamectin was first evaluated at the 1992 JMPR and subsequently in 1994. MRLs have been recommended for a number of crops and animal commodities.

The Meeting received information on current registered uses, methods of analysis and data on residues in supervised trials on the additional crops apples, potatoes and hops as well as new trials on pears, cucurbits, lettuce and tomatoes. Processing data were available for apples, pears, potatoes and hops.

The predominant residues from the use of abamectin on crops are avermectin B_{1a}, avermectin B_{1b} and the photoisomers 8,9-*Z*-avermectin B_1 (B_{1a} and B_{1b}) produced during exposure to sunlight. Analytical methods that measure the components of the residue rely on HPLC separation and fluorescence detection of derivatives formed by converting the cyclohexene ring to an aromatic ring. The abamectin residue appears as two peaks on the chromatogram (B_{1a} and its photoisomer in one peak and B_{1b} and its photoisomer in the other). The LOD for each peak is in the range 0.002-0.005 mg/kg.

Abamectin residues were shown to be stable in samples of fresh and dried hops during freezer storage for the periods tested (150-190 days).

The Meeting noted that the definition proposed by JECFA (1997) for residues in the liver, kidney and fat of animals subject to veterinary treatment with abamectin does not include the 8,9-*Z*-isomer ()-8,9- isomer), because it is not present in animal tissues when abamectin is used directly on the animal. However, residues in animal tissues arising from residues in animal feed would include the 8,9-*Z*- isomer. The Meeting agreed that the wider definition (including the 8,9-*Z*- isomer) was appropriate for a laboratory carrying out enforcement or monitoring analyses because the analyst would not know whether the residue in the animal originated only from veterinary treatment or also from the feed. The wider definition accommodates both situations.

Inclusion or exclusion of avermectin B_{1b} from the definition of the residue is a matter of judgement. In many crop situations B_{1b} is present at approximately 10% of the total residue and the analytical methods measure B_{1a} and B_{1b} by the same procedure so B_{1b} results are always available and may as well be used.

Avermectin B_{1b} forms a photoisomer 8,9-*Z*-avermectin B_{1b} in sunlight in the same way as avermectin B_{1a} does. The studies of photolysis were with avermectin B_{1a}, so when the JMPR reviewed the studies in 1992 the possibility of 8,9-*Z*-avermectin B_{1b} being produced was not taken into account. In practice the contribution of 8,9-*Z*-avermectin B_{1b} to the residue will be small but it should be recognized that the HPLC measurement of avermectin B_{1b} residues includes any 8,9-*Z*-avermectin B_{1b}. The Meeting agreed to revise the definition of the residue accordingly, and recommended the following definition for compliance with MRLs and for the estimation of dietary intake.

Sum of avermectin B_{1a}, avermectin B_{1b}, 8,9-Z-avermectin B_{1a} and 8,9-Z-avermectin B_{1b}.

The Meeting received data from supervised residue trials on apples, pears, cucumbers, melons, summer squash, tomatoes, lettuce, potatoes and hops.

The B_{1b} component, when its residues were measurable, was consistently about 10% or less of the total residue. For the purposes of evaluation, when B_{1a} was positively detected and B_{1b} was not detectable the total residue was calculated by taking the undetectable residue to be zero.

When both components in a trial were not detectable (ND) the total residue was taken as below the limit of detection. A residue reported as NQ (not quantifiable, detected but below the limit of determination LOD) is treated as equal to the LOD when it is to be added to a measurable residue.

The method of calculating the total residue for various situations is illustrated by the examples below.

B_{1a}	B_{1b}	Total residue
0.013	NQ (>0.001 but <0.002)	0.015
0.006	ND (<0.001)	0.006
NQ	ND	<0.002
ND	ND	<0.001

Abamectin is registered for single applications on apples in Australia at 0.014 kg ai/ha with harvest after an interval of 14 days. In three trials corresponding to this use pattern the abamectin residues were <0.002, 0.003 and 0.005 mg/kg.

Abamectin is permitted for use on pome fruit in New Zealand with one application at 0.027 kg ai/ha and a PHI of 14 days. Abamectin residues in apples were 0.004 and 0.007 mg/kg in two New Zealand trials where GAP was followed except that two applications were made instead of one.

Abamectin is registered in the USA for two applications on apples at a rate of 0.026 kg ai/ha with harvest 28 days after the final application. In 14 US trials according to these conditions abamectin residues in rank order (median underlined) were <0.001 (2), <0.002 (3), 0.002, 0.003 (4), 0.004, 0.006, 0.007 and 0.012 mg/kg.

The residue data from Australia, New Zealand and the USA appear to be from one population and can therefore be combined. The residues of abamectin in apples in rank order in the 19 trials (median underlined) were <0.001 (2), <0.002 (4), 0.002, 0.003 (5), 0.004 (2), 0.005, 0.006, 0.007 (2) and 0.012 mg/kg.

The Meeting estimated a maximum residue level of 0.02 mg/kg and an STMR level of 0.003 mg/kg for abamectin in apples.

In the USA abamectin is registered for use on pears at 0.013-0.026 kg ai/ha with two applications permitted at the higher rate and a 28-day PHI. Data from four US trials were provided. The results of supervised trials on pears had previously been reported to the 1992 JMPR. A number of residue decline trials on pears in the USA had shown that the typical half-life was approximately 18 days. At such a rate residues at harvest 21 and 37 days after the final treatment would be ±30% of those at 28 days. The range of pre-harvest intervals for acceptance of the residues was therefore taken as 21-37 days. Abamectin residues in pears from the four trials according to US GAP were 0.004, 0.006, 0.009 and 0.011 mg/kg.

The 1992 monograph recorded one pear trial according to Argentinian GAP, (abamectin <0.005 mg/kg), one according to French GAP (<0.002 mg/kg) and four according to Italian GAP (<0.002 and <0.005 (3) mg/kg).

The residues in the trials in different countries appear to be of the same order, giving residues in rank order (median underlined) of <0.002 (2), 0.004, <0.005 (4), 0.006, 0.009 and 0.011 mg/kg.

The Meeting estimated a maximum residue level for abamectin in pears of 0.02 mg/kg, to replace the previous estimate of 0.01* mg/kg, and an STMR level of 0.005 mg/kg.

In the USA melons may be treated with abamectin at 0.011-0.021 kg ai/ha on three occasions at the higher rate and harvested 7 days after the final treatment. Abamectin residues were not detectable (<0.002 mg/kg) in 9 trials in the USA on cantaloupes according to US GAP, except that there were 4 or 5 applications instead of 3, or in two trials on watermelons under the same conditions. Because the use patterns are the same, watermelons and melons can be evaluated together.

Melons may be treated with abamectin three times at rates up to 0.022 kg ai/ha and harvested three days after the final application according to the registered use in Spain. Abamectin residues were not detected (<0.002 mg/kg) in cantaloupes treated according to Spanish GAP, except that there were four applications, in two glasshouse trials in Spain. Three trials on cantaloupe in France with the same treatment yielded residues of <0.002, <0.005 and <0.005 mg/kg.

Trials on cantaloupes in Brazil and Mexico and on honey-dew melons in Mexico could not be evaluated because there was no information on corresponding GAP. In the Brazilian trials the edible pulp was analysed for abamectin and no residues were detected in any samples in any trial, suggesting that abamectin residues are probably absent from the edible parts of melons.

In summary abamectin residues in melons from trials according to GAP were <0.002, <0.005 and <0.005 mg/kg in France, <0.002 mg/kg (2)) in Spain, <0.002 (9) mg/kg in the USA and <0.002 mg/kg (2) in watermelons in the USA. The residues in melons and watermelons in rank order were <0.002 (14) and <0.005 (2) mg/kg.

The Meeting estimated maximum residue levels of 0.01* mg/kg as being a practical limit of determination, and an STMR level of 0.002 mg/kg, for abamectin in melons and watermelons.

Abamectin is registered for use in the USA on cucumbers and squash at 0.011-0.021 kg ai/ha with three applications at the higher or six at the lower rate, and harvest 7 days after the final treatment. In four US trials on cucumbers at 0.021 or 0.022 kg ai/ha, but with four applications instead of three, residues were undetectable in three trials (<0.002 mg/kg) and below the LOD in the other (<0.005 mg/kg). In four US trials on zucchini (summer squash) under the same conditions no abamectin residues were detectable (<0.002 mg/kg).

Mexican trials on cucumbers and pickling cucumbers could not be evaluated because no information on relevant GAP was available.

The registered use of abamectin on glasshouse cucumbers in Germany permits 5 applications of 0.023 kg ai/ha with harvest three days after the final application. Treatment is not permitted between November and February. Two French trials according to this use pattern were recorded in the 1992 monograph. The resultant abamectin residues were <0.002 and <0.005 mg/kg. A third trial with applications during October and November produced a residue of 0.034 mg/kg, but the conditions were no longer according to GAP.

GAP for abamectin on cucumbers in Spain permits three applications at 0.022 kg ai/ha with harvest three days after the last. Two glasshouse trials in Spain and three trials in Italy (one glasshouse) according to this use pattern but with 4 or 5 applications were recorded in the 1992 monograph. The residues were <0.002, <0.005 (2), 0.006 and 0.008 mg/kg.

GAP for glasshouse cucumbers in The Netherlands allows 5 applications of 0.023 kg ai/ha and harvest three days after the final application. In two trials on cucumbers under these conditions the residues were 0.007 and 0.008 mg/kg, as recorded in the 1992 monograph.

In summary, the residues in cucumbers from trials according to GAP were <0.002 (3) and <0.005 mg/kg in the USA, <0.002 and <0.005 mg/kg in France, <0.002, <0.005 (2), 0.006 and 0.008 mg/kg in Spain and Italy, and 0.007 and 0.008 mg/kg in The Netherlands. The residues in rank order (median underlined) were <0.002 (5), <0.005 (4), 0.006, 0.007 and 0.008 (2) mg/kg.

The Meeting estimated a maximum residue level for abamectin in cucumbers of 0.01 mg/kg, to replace the previous estimate of 0.05 mg/kg, and an STMR of 0.005 mg/kg.

The four trials on summer squash in the USA were evaluated with the support of the four on cucumbers. Abamectin residues from the 8 trials were <0.002 (7) and <0.005 mg/kg.

The Meeting estimated a maximum residue level for abamectin on summer squash of 0.01* mg/kg as being a practical limit of determination, and an STMR of 0.002 mg/kg.

Abamectin is registered for four applications to glasshouse tomatoes in The Netherlands at 0.023 kg ai/ha with a PHI of three days. Abamectin residues in tomatoes from trials which complied with GAP were 0.007 (2), 0.009, 0.012 (2) and 0.017 mg/kg. Two of the tomato trials in The Netherlands reported in the 1992 monograph (refs 211 and 212) were not according to current GAP because applications were made during the months of November and December. Current GAP restricts the treatment of glasshouse tomatoes to the months of March to October when photodegradation of abamectin residues is sufficient. Two other trials (refs 217 and 218) were according to current GAP because abamectin was applied in May and June. The residues from these two trials were 0.008 and 0.005 mg/kg.

GAP in Argentina permits 9 applications of abamectin at 0.022 kg ai/ha to tomatoes with a 3-day PHI. In the three trials with conditions close to GAP (0.020-0.028 kg ai/ha and 5-9 applications) recorded in the 1992 monograph the residues were <0.002 (2) and <0.005 mg/kg.

In Brazil abamectin may be applied to tomatoes at 0.022 kg ai/ha with harvest three days after the final application. Three Brazilian trials recorded in the 1992 monograph were close to these conditions, with residues of <0.005 (2) and 0.017 mg/kg.

Three French trials recorded in 1992 were evaluated according to German GAP (5 applications of 0.023 kg ai/ha applied to glasshouse tomatoes with harvest three days after the final application). Tomatoes were treated 10 times in one trial, but it was evaluated because residues apparently disappeared quickly and the number of applications would not influence the final residue. The residues were <0.002 (2), and <0.005 mg/kg.

Two Italian trials recorded in 1992 complied with the Italian application rate (0.022 kg ai/ha) and PHI (7 days), but there were ten applications instead of two. The results were again considered acceptable because the residues were disappearing quickly. The residues in both trials were <0.002 mg/kg.

In Spain abamectin may be used on tomatoes at 0.022 kg ai/ha with a PHI of three days. The residues in tomatoes from four trials recorded in the 1992 monograph with application rates in the range 0.015-0.027 kg ai/ha were <0.005 (3) and 0.009 mg/kg.

GAP in the USA specifies three applications of 0.021 kg ai/ha and harvest 7 days after the final application. Eighteen US trials are recorded in the 1992 monograph at this application rate and a PHI of 7 days or less, but with 8-12 applications. The residues had usually disappeared within a few days so it is unlikely that early applications had any influence on the final residues. The residues were <0.002 (13), <0.005 (4) and 0.005 mg/kg.

In summary, the residues in tomatoes from trials according to GAP were 0.005, 0.007 (2), 0.008, 0.009, 0.012 (2) and 0.017 mg/kg in The Netherlands, <0.002 (2) and <0.005 mg/kg in Argentina, <0.005 (2) and 0.017 mg/kg in Brazil, <0.002 (2) and <0.005 mg/kg in France, <0.002 (2) mg/kg in Italy, <0.005 (3) and 0.009 mg/kg in Spain and <0.002 (13), <0.005 (4) and 0.005 mg/kg in the USA. The residues in rank order (median underlined and Netherlands results in bold) were <0.002 (19), <0.005 (11), 0.005, **0.005**, **0.007 (2)**, **0.008**, 0.009, **0.009, 0.012 (2), 0.017** and 0.017 mg/kg.

The residues in The Netherlands appear to belong to a different population from the others, with a median of 0.0085 mg/kg.

The Meeting estimated a maximum residue level for abamectin in tomatoes of 0.02 mg/kg, the same as the previous estimate, and an STMR of 0.0085 mg/kg.

GAP in The Netherlands permits four applications of abamectin to lettuce at 0.014 kg ai/ha with harvest 14 days after the final application, but only from 1 March to 1 November. In four glasshouse trials in The Netherlands according to GAP the residues in head lettuce were 0.016, 0.025, 0.029 and 0.029 mg/kg.

Abamectin may be used four times on lettuce in France at 0.009 kg ai/ha with harvest 7 days after the final application. In three French trials where the application rate was approximately 25% higher than this, but within the acceptable range for evaluation, the residues were <0.001, 0.004 and 0.023 mg/kg.

In Spain abamectin may be applied three times to lettuce at 0.022 kg ai/ha with harvest 14 days after the final application. In two Spanish and three French trials at this rate and PHI, but with four applications instead of three, the abamectin residues were <0.002, 0.005, 0.013, 0.028 and 0.040 mg/kg.

Trials on lettuce in the USA recorded in the 1992 monograph could not be evaluated because the number of applications, 6-10, was excessive for a sometimes persistent residue compared with the three applications permitted.

In summary, the residues in head lettuce from trials according to GAP were 0.016, 0.025, 0.029 and 0.029 mg/kg in The Netherlands, <0.001, 0.004 and 0.023 mg/kg in France and <0.002, 0.005, 0.013, 0.028 and 0.040 mg/kg in Spain. The residues reported in 1992 in rank order (median underlined) were 0.016, 0.016, 0.033, 0.047, 0.059 and 0.077 mg/kg.

The Meeting estimated a maximum residue level for abamectin in almonds of 0.01* mg/kg as being a practical limit of determination and, because no residues were detected in the trials at normal and double rates, an STMR of 0 mg/kg. The Meeting also estimated maximum residue and STMR levels for abamectin on almond hulls of 0.1 mg/kg and 0.040 mg/kg respectively.

GAP in the USA for walnuts is the same as for almonds. Abamectin residues were not detected (<0.002 mg/kg) in walnuts from six US trials recorded in 1992 according to the maximum US application rate but harvested after 14 days, or in those from four other trials at a double rate.

The Meeting estimated a maximum residue level for abamectin in walnuts of 0.01* mg/kg as being a practical limit of determination, and an STMR of 0 mg/kg.

Abamectin is registered for use on hops in Germany and the USA with two applications of 0.023 and 0.022 kg ai/ha respectively and a PHI of 28 days. The residues in dry hops from 12 German and 4 US trials according to GAP in rank order (median underlined) were <0.003 (4), <0.005, 0.011, 0.012, 0.015, 0.017, 0.022 (2), 0.023, 0.025, 0.030, 0.062 and 0.086 mg/kg.

The Meeting estimated maximum residue and STMR levels of 0.1 mg/kg and 0.016 mg/kg respectively.

A feeding study on dairy cows recorded in the 1992 monograph showed that residues in the milk, liver, muscle, fat and kidney did not exceed 0.004, 0.020, 0.002, 0.014 and 0.005 mg/kg respectively at a feeding level of 0.1 ppm. The residues in animal commodities arising from the consumption of abamectin-treated almond hulls should not exceed current draft MRLs.

Information on the fate of abamectin residues during the processing of apples, pears, potatoes and hops was provided.

Abamectin residues were not detectable in the juice or sauce produced from treated apples, but were concentrated in pomace, a result expected from the nature of abamectin as a surface residue. The calculated processing factors were <0.062 for juice, <0.12 for apple sauce and 17.3 for dry pomace. The "<" signs indicate derivation from the LOD for abamectin in the processed commodities.

The supervised trials median residues for the processed commodities (STMR-Ps) calculated from the processing factors and the STMR level for apples (0.003 mg/kg) are apple juice 0.00019 mg/kg, apple sauce 0.00036 mg/kg and dry apple pomace 0.052 mg/kg.

Abamectin residues were not detectable in pear halves or pear purée produced from treated pears. The calculated processing factors were canned pear halves <0.046 and pear purée <0.048.

The STMR-Ps for the processed commodities calculated from the processing factors and the STMR for pears (0.005 mg/kg) were canned pear halves 0.00023 mg/kg and pear purée 0.00024 mg/kg.

The processing study on potatoes could not be completed because no abamectin residues were detectable in the treated potatoes.

Abamectin-treated hops were processed by exhaustive hexane extraction of dry hops to produce a solvent extract and spent hops. The extract contains flavour components and is used in the brewing industry while the spent hops become a minor feed commodity. Most of the abamectin residues remained in the spent hops. The mean processing factor from dry hops to spent hops was 0.71.

The mean processing factor for abamectin residues during the conversion of fresh hops to dry hops was 4.09, suggesting that approximately 80% of the abamectin survived the drying process.

The 1992 JMPR recommended MRLs for cattle meat and offal of 0.01* and 0.05 mg/kg respectively on the basis of possible abamectin residues in animal feed commodities.

On the basis of veterinary uses the 1996 JECFA recommended MRLs for residues defined as avermectin B_{1a} of 100 µg/kg for cattle fat and liver, and 50 µg/kg for kidney.

The Meeting agreed that MRLs should accommodate both agricultural and veterinary uses where the necessary information is available, and agreed to replace the recommendation for edible offal with recommendations for MRLs in fat, liver and kidney in line with the levels recommended by JECFA.

It is not clear whether the current recommendation for cattle meat (0.01 mg/kg) would accommodate veterinary uses. The Meeting recommended that JECFA be requested to suggest an appropriate maximum residue level in cattle meat, and to consider accepting the broader definition of the residue to accommodate the residues which occur as a result of agricultural as well as veterinary uses.

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 (for compliance with MRL and for estimation of dietary intake): Sum of avermectin B_{1a}, avermectin B_{1b}, 8,9-*Z*-avermectin B_{1a} and 8,9-*Z*-avermectin B_{1b}.

Commodity		Recommended MRL, mg/kg		Based on PHI, days	STMR, mg/kg	STMR-P, mg/kg
CCN	Name	new	current			
TN 0660	Almond	0.01*		21	0	
	Almond hulls	0.1		21	0.040	
FP 0226	Apple	0.02	-	14-28	0.003	
MF 0812	Cattle fat	0.1 V	-			
MO 1289	Cattle kidney	0.05 V	Note[1]			
MO 1281	Cattle liver	0.1 V	Note[1]			
MO 0812	Cattle, Edible offal of	W	0.05			
VC 0424	Cucumber	0.01	0.05	3-7	0.005	
DH 1100	Hops, dry	0.1	-	28	0.016	
VL 0482	Lettuce, Head	0.05	-	7-14	0.020	
VC 0046	Melons, except Watermelon	0.01*	-	3-7	0.002	

Commodity		Recommended MRL, mg/kg		Based on PHI, days	STMR, mg/kg	STMR-P, mg/kg
CCN	Name	new	current			
FP 0230	Pear	0.02	0.01*	14-21	0.005	
VR 0589	Potato	0.01*	-	0-14	0	
VC 0431	Squash, Summer	0.01*	-	7	0.002	
VO 0448	Tomato	0.02	0.02	3	0.0085	
TN 0678	Walnut	0.01*		14	0	
VC 0432	Watermelon	0.01*	-	7	0.002	
	Apple juice					0.00019
	Apple sauce					0.00036
	Canned pears					0.00023
	Pear purée					0.00024

* MRL at or about limit of determination.
W: previous recommendation withdrawn.
V: includes residues which may arise from veterinary uses.
[1]The current recommendation for Cattle edible offal is to be replaced by recommendations for Cattle kidney and Cattle liver to accommodate JECFA recommendations arising from veterinary uses with abamectin.

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EXPLANATION

Bifenthrin was first evaluated at the 1992 JMPR and MRLs of 0.05* mg/kg were recommended for barley, maize and wheat to cover field applications. Information was provided to the 1995 and 1996 Meetings on the use of bifenthrin as a stored grain protectant. The 1996 JMPR recommended MRLs for wheat and milled commodities related to the use on stored grain.

The 28th Session of the CCPR (1996, ALINORM 97/24, para 79) noted that the CXLs for animal products might need to be changed if GAP for barley, wheat and maize is changed. The 29th Session (1997, ALINORM 97/24A, para 74) postponed discussion pending a review by the 1997 JMPR.

The 1996 JMPR listed the following information as desirable in connection with the use of bifenthrin as a grain protectant.

1. Validation of the analytical method for recoveries of bifenthrin residues from bread at the levels occurring in practice and at the LOD.

2. Information on the degree of extraction of bifenthrin residues from bread by the current procedure.

3. Information on national registrations and MRLs for bifenthrin covering its use on stored grain.

4. Information on the fate of bifenthrin during the simulated commercial malting of barley treated post-harvest (from 1995).

The Meeting received a report on the baking process used in the bread trials reviewed in 1996, information on the registration status of bifenthrin as a grain protectant and reports on a malting trial and on laboratory and silo scale grain storage trials.

METHODS OF RESIDUE ANALYSIS

Analytical methods

Slaiding (1995) determined residues of bifenthrin and malathion in barley and malt by a GC-MS method with LODs of 0.01 mg/kg for bifenthrin and 0.02 mg/kg for malathion. The samples were soaked in water and then extracted with an acetone/methanol mixture. The extract was cleaned up by passage through small columns before analysis. Recoveries were 63-86% for bifenthrin and 65-81% for malathion.

The Australian Wheat Board laboratory analysed wheat samples (Table 1) by a method developed by Academy of Grain Technology. Wheat (50 g) was shaken for 15 minutes in a stoppered flask with hexane (50 ml). The mixture was allowed to stand for 42 hours and shaken for a further 15 minutes before filtration to provide a clear solution for GLC on a non-polar column with EC detection. The limit of detection was reported to be 0.01 mg/kg.

Agricultural Chemistry Branch samples (Table 1) were analysed for bifenthrin residues by a method for organochlorine residues method (Simpson, 1993). Wheat (5 g) was ground before extraction by shaking with methanol for 1 hour. Bifenthrin residues were partitioned into hexane after dilution of the methanol with water. The extract was dried before clean-up on a Florisil column. Bifenthrin residues were then determined by GLC with a Hall detector for chlorinated compounds. The limit of detection was 0.05 mg/kg and recoveries from spiked samples were 106% at 0.53 mg/kg and 101% at 0.27 mg/kg.

Maize and wheat samples in the South African trials (Table 3) were extracted with 10% acetone in hexane by shaking for 3 hours (Van der Linde, 1995). The extract was filtered through sodium sulphate and analysed by GLC with an ECD for bifenthrin and an FPD for malathion. Good recoveries were obtained from samples spiked at 0.2 and 1 mg/kg. Only a summary of the method was available.

USE PATTERN

The registered uses of bifenthrin on stored grain were recorded by the 1995 JMPR. Croatia and Romania issued temporary registrations for such uses in 1996 for 2 and 5 years respectively.

RESIDUES RESULTING FROM SUPERVISED TRIALS

In an Australian trial Bengston (1995) treated wheat (500 tonnes) in silo storage with a mixture of bifenthrin at 0.5 g ai/t and chlorpyrifos-methyl at 10 g ai/t and sampled the grain at intervals up to 6 months for residue analysis. The temperature of the stored grain fell from 24.8°C 1.5 months after treatment to 15°C at the end of the storage. The grain moisture level was in the range 9.3 to 9.8%.

Samples of treated grain were transferred to a freezer at -10°C 5-11 days after sampling, where they were held for 7-13 months before delivery to four laboratories for analysis. Samples analysed by Agricultural Chemistry were held at -20°C for approximately 10 weeks before analysis. No information was available on the conditions or duration of the storage of samples analysed by the Australian Wheat Board.

Table 1. Residues in wheat treated with bifenthrin + chlorpyrifos-methyl (0.5 + 10 g ai/t) and stored for 6 months (Bengston, 1995). Four laboratories[1] analysed the wheat.

Storage period, months	Residues, mg/kg				
	Bifenthrin		Chlorpyrifos-methyl		
	Lab AC	Lab AWB	Lab APG	Lab AWB	Lab NSW
0	0.56	0.5	8.9	9.9	6.3
1.5	0.32	0.4	7.1	7.7	-

Storage period, months	Residues, mg/kg				
	Bifenthrin		Chlorpyrifos-methyl		
	Lab AC	Lab AWB	Lab APG	Lab AWB	Lab NSW
3	0.43	0.4	7.0	7.7	6.1
4.5	0.37	-	7.1	7.9	5.6
6	0.43	0.4	6.4	6.6	5.2

[1]AC: Agricultural Chemistry Branch, Queensland Department of Primary Industries.
AWB: Australian Wheat Board, Victoria.
APG: Agricultural Production Group, Queensland Department of Primary Industries.
NSW: New South Wales Department of Agriculture.

De Baptista (1995) treated small lots (2 kg) of rice, wheat and maize with bifenthrin + piperonyl butoxide (0.4 + 2 mg/kg and 0.8 +4 mg/kg), in plastic bags. In each trial the treated grain was divided and stored at room temperature in 4 closed paper sacks, 500 g in each. At each sampling interval one sack of grain was taken for the determination of bifenthrin. Bifenthrin residues (Table 2) were considerably depleted after 60 days storage, whereas bifenthrin is generally persistent on wheat in bulk storage. Details of the GLC analytical method were not provided, but the LOD was 0.02 mg/kg and the analytical recoveries were 81±5%.

Table 2. Residues of bifenthrin in grain treated with bifenthrin and piperonyl butoxide and stored in closed paper sacks at room temperature in Brazil (De Baptista, 1995).

Grain	Treatment, mg/kg. bifenthrin + piperonyl butoxide	Storage interval, days	Bifenthrin, mg/kg
Rice (Araguaia)	b 0.4 + pb 2	0 15	0.3 0.4
Rice (Araguaia)	b 0.8 + pb 4	0 15	0.7 0.6
Rice (Araguaia)	b 0.4 + pb 2	0 15 30 60	0.4 0.4 0.2 0.1
Rice (Araguaia)	b 0.8 + pb 4	0 15 30 60	0.9 0.7 0.4 0.2
Wheat (BR23)	b 0.4 + pb 2	0 15 30 60	0.4 0.3 0.2 0.1
Wheat (BR23)	b 0.8 + pb 4	0 15 30 60	0.6 0.7 0.5 0.3
Wheat (BR23)	b 0.4 + pb 2	0 15	0.4 0.2
Wheat (BR23)	b 0.8 + pb 4	0 15	0.8 0.7
Maize (AG303)	b 0.4 + pb 2	0 15	0.5 0.4
Maize (AG303)	b 0.8 + pb 4	0 15	0.7 0.7

Grain	Treatment, mg/kg. bifenthrin + piperonyl butoxide	Storage interval, days	Bifenthrin, mg/kg
Maize (AG303)	b 0.4 + pb 2	0 15 30 60	0.4 0.3 0.2 0.08
Maize (AG303)	b 0.8 + pb 4	0 15 30 60	0.8 0.8 0.6 0.2

In a South African trail with stored grain Van der Linde (1995) treated maize and wheat grain in 20 kg lots with bifenthrin + malathion at 0.3 + 6.0 and 0.4 + 8.0 g ai/t and stored the grain in 20-l plastic containers with lids in ambient conditions for 20 weeks. The results are shown in Table 3. The residues on the grain were substantially lower than the target application rates. Milled samples were obtained by grinding the whole grain to a fine powder in a laboratory mill. Bifenthrin residues did not decrease during storage.

Table 3. Bifenthrin and malathion residues in stored maize and wheat after treatments of 20 kg lots and storage in 20 litre plastic bins (Van der Linde, 1995).

Grain	Storage interval, weeks	Treatment with bifenthrin 0.3 g ai/t + malathion 6.0 g ai/t				Treatment with bifenthrin 0.4 g ai/t + malathion 8.0 g ai/t			
		bifenthrin, mg/kg		malathion, mg/kg		bifenthrin, mg/kg		malathion, mg/kg	
		grain	milled	grain	milled	grain	milled	grain	milled
Maize	0	0.16	0.11	2.5	1.6	0.19	0.11	3.2	2.7
	4	0.12	0.14	1.7	1.0	0.16	0.12	1.9	1.0
	12	0.10	0.12	1.4	1.1	0.13	0.14	2.6	1.4
	20	0.14	0.14	1.7	1.3	0.19	0.16	2.8	1.6
Wheat	0	0.23	0.13	4.0	3.7	0.27	0.18	5.5	4.4
	4	0.22	0.20	3.9	4.7	0.24	0.26	4.0	4.6
	12	0.18	0.17	3.8	3.4	0.24	0.30	4.3	6.4
	20	0.20	0.38	3.4	4.2	0.27	0.27	4.3	6.0

FATE OF RESIDUES IN STORAGE AND PROCESSING

In processing

Full details of the Chorleywood baking process used in the baking trials reported in 1996 were provided at that time. The report (Collins, 1976) has now been made available. Essential features of the process are intense mechanical working of the dough within a few minutes, addition of ascorbic acid at 75 mg/kg, inclusion of fat at 0.7%, extra water at 3.5% and additional yeast.

Baxter (1995) provided a more detailed report of the barley malting trials reviewed by the 1995 JMPR. The trials were pilot scale (50 kg), whereas the 1995 JMPR had understood them to be with 350 g lots.

When barley was treated at 0.6 + 12 g ai/t with bifenthrin + malathion (twice the recommended rate) and malted, bifenthrin levels were considerably reduced and residues of malathion effectively disappeared. Residue levels in the barley before screening and cleaning were not reported. The study was defective because the barley samples were stored for approximately 100 days at ambient temperature before malting began and no samples were taken at that time. The results are shown below.

Sample	Nominal treatment	
	Bifenthrin	Malathion
	0.60 g/t	12 g/t
	Residues, mg/kg	
Screened and cleaned barley[1]	0.31	4.6
Derooted malt[2]	0.026	<0.02

[1]Barley sampled 23-31 March 1994
[2]Malt prepared from barley stored at room temperature until 6 July 1994

APPRAISAL

Information was provided to the 1995 and 1996 Meetings on the use of bifenthrin as a stored grain protectant. The 1996 Meeting recommended MRLs for wheat and milled commodities related to this use. It was suggested at the CCPR that the CXLs for animal products might be affected by revised GAP for cereals.

The 1996 JMPR listed the following information as desirable in connection with the use of bifenthrin as a grain protectant.

Validation of the analytical method for recoveries of bifenthrin residues from bread at the levels occurring in practice and at the LOD.

Information on the degree of extraction of bifenthrin residues from bread by the current procedure.

Information on national registrations and MRLs for bifenthrin covering its use on stored grain.

Information on the fate of bifenthrin during the commercial malting of barley treated with it post-harvest. The studies should simulate the commercial process. (From 1995 JMPR).

No additional information was available on the analytical method for bifenthrin residues in bread.

Croatia and Romania have issued temporary registrations for the treatment of stored grain with bifenthrin.

The results of a barley malting trial were made available. It suggested that bifenthrin residues decreased substantially during the malting process but the study was defective because no barley samples were taken for analysis at the time the malting commenced.

The Meeting received additional data from trials with bifenthrin on stored grain. Bifenthrin residues are generally persistent during storage.

The recommendations of the 1992 JMPR for MRLs for bifenthrin in cattle fat, kidney, liver, meat and milk were based on the assumption that levels of bifenthrin in the diet of cows were unlikely to exceed 2 ppm, on the basis of a feeding study in which bifenthrin was fed at 5 ppm for 28 days. The 1995 Meeting recommended MRLs of 0.5 mg/kg in wheat and 2 mg/kg in wheat bran because of the post-harvest use on wheat. Because these levels do not exceed the level of 2 ppm in the feed on which the 1992 recommendations were based no change to the draft MRLs for cattle commodities is needed.

In a study of metabolism in laying hens (1992 JMPR) bifenthrin constituted 51.5% of the total residue in abdominal fat of 1.0 mg/kg produced by a feeding level of 40 ppm. In a feeding study on laying hens also reported in 1992 birds were fed for 28 days at 0.25 ppm. Total residues at the limit of detection of 0.01 mg/kg were detected only in fat, suggesting that the residue of bifenthrin in fat was 0.005 mg/kg. Bifenthrin residues in the eggs were 0.002-0.004 mg/kg.

The current CXLs for bifenthrin in chicken fat and the fat of chicken meat are 0.05* mg/kg, which should be adequate for a feed level of 2.5 ppm bifenthrin if the proportionality between levels in the feed and fat is the same as in the trial. The current CXL for bifenthrin in eggs is 0.01* mg/kg, which should be adequate for a feed level of 0.6-1.2 ppm bifenthrin.

The draft MRL for bifenthrin in wheat (to cover post-harvest use) is 0.5 mg/kg, and the STMR is 0.255 mg/kg. The current CXLs for chicken eggs, fat, meat and edible offal should be adequate for chickens consuming bifenthrin-treated wheat, which can be a major part of a poultry diet.

The 1996 Meeting estimated maximum residue and STMR levels for bifenthrin in bran produced from post-harvest-treated wheat of 2 and 0.89 mg/kg respectively. Bran may constitute 50% of the poultry diet; at this level the CXLs for chicken fat, meat and offal should be adequate. The CXL for eggs is probably adequate. It is at the LOD and its adequacy depends on assumptions about the proportion of bran in the poultry diet.

The Meeting made no recommendations to change the existing CXLs or draft MRLs for bifenthrin.

The Meeting noted that the data submitted had not provided the information which the 1995 and 1996 Joint Meetings needed to recommend MRLs for stored grains, except wheat, and their products.

Future submissions of data should meet the requirements of the *FAO Manual on the Submission and Evaluation of Pesticide Residues Data for the Estimation of Maximum Residue Levels in Food and Feed.* The studies should be valid and supply the information listed in the 1995 and 1996 Evaluations.

FURTHER WORK OR INFORMATION

Desirable

1. Validation of the analytical method for recoveries of bifenthrin residues from bread at the levels occurring in practice and at the LOD.

2. Information on the degree of extraction of bifenthrin residues from bread by the current procedure.

3. Information on national registrations and MRLs for bifenthrin covering its use on stored grain.

4. Valid studies on the fate of bifenthrin during the malting of barley treated with it post-harvest. The studies should simulate the commercial process.

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CAPTAN (007)

EXPLANATION

Captan has been evaluated several times since the initial evaluation in 1965, most recently in 1994 (residues) and 1995 (toxicology).

The 1994 JMPR recommended MRLs for apple, blueberries, cherries, grapes, nectarine, peach, pear, plums, strawberry and tomato. The 28th Session of the CCPR (ALINORM 97/24, para 31 and addendum, p 16) returned draft MRLs for apple, cherries, grapes, nectarine, pear, plums, strawberry and tomato to step 3 pending evaluation by the 1997 JMPR.

Information was made available to the Meeting on uses of captan and on supervised trials on apples, cherries, grapes, nectarines, pears, plums and strawberries in the USA, and on strawberries in Canada. Summary data from Germany were provided on trials on grapes, strawberries, radishes and chives.

METHODS OF RESIDUE ANALYSIS

Fujie (1982) described the analytical method (RM-1K-2) used in the analysis of crop samples from the Chevron trials for captan and tetrahydrophthalimide (THPI) the main metabolite of captan. The author noted the instability of captan in macerated crop mixes even when stored at -20°C. Captan-treated crops should be analysed immediately after maceration and subsampling.

Samples were extracted with ethyl acetate in the presence of phosphoric acid and sodium sulfate and the ethyl acetate extract washed with 0.5-1% phosphoric acid. The extract was evaporated and the residue dissolved in dichloromethane/acetone for clean-up by gel-permeation chromatography. Clean-up was continued with a small Nuchar/silica gel column. Part of the eluate from this column was used for the determination of THPI. The remainder was passed through a Florisil column to provide a solution ready for the determination of captan.

Captan was determined by GLC with a flame-photometric detector in the sulfur mode, while THPI required a nitrogen/phosphorus flame-ionization detector. The LOD was typically near 0.01 mg/kg.

USE PATTERN

A US registered label for a captan wettable powder was made available to the Meeting. The UK, Thailand and Germany provided lists of registered uses. These are summarized in Table 1.

Table 1. Registered uses of captan in the USA, Germany, Thailand and the UK.

Crop	Country	Form	Application			Max total	PHI, days
			method	rate per applic. kg ai/ha	spray conc. kg ai/hl	Applic kg ai/ha per crop cycle	
Almond	USA	WP	✈ foliar[1]	2.2-5.9	1.2-13	29	30

Crop	Country	Form	Application			Max total	PHI, days
			method	rate per applic. kg ai/ha	spray conc. kg ai/hl	Applic kg ai/ha per crop cycle	
Almond	USA	WP	Foliar[1]	2.2-5.9	0.08-3.1	29	30
Apple	UK	WG WP	Foliar	2.7-2.9			7
Apple	UK	WG WP	Post-harvest dip		0.1		
Apple	USA	WP	✈ foliar[2]	2.2-4.5	1.2-9.6	36	0
Apple	USA	WP	Foliar[2]	2.2-4.5	0.060-2.4	36	0
Apple	USA	WP	Post-harvest spray or dip		0.15		
Apricot	USA	WP	✈ foliar[3]	1.7-2.8	0.90-3.0	14	0
Apricot	USA	WP	Foliar[3]	1.7-2.8	0.072-1.5	14	0
Blueberries	USA (east)	WP	✈ foliar[4]	2.8	6.0 max	39	0
Blueberries	USA (east)	WP	Foliar[4]	2.8		39	0
Blueberries	USA (west)	WP	✈ foliar[5]	1.1-2.8	0.60-6.0	39	0
Blueberries	USA (west)	WP	Foliar[5]	1.1-2.8	0.060-1.5	39	0
Cherry	USA	WP	post-harvest spray or dip		0.15		
Cherry	USA (east)	WP	✈ foliar[6]	2.2	1.2-2.4	16	0
Cherry	USA (east)	WP	Foliar[6]	2.2	0.12-1.2	16	0
Cherry	USA (west)	WP	✈ foliar[6]	1.7-2.2	0.90-2.4	16	0
Cherry	USA (west)	WP	Foliar[6]	1.7-2.2	0.090-1.2	16	0
Grape	Thailand	WP	high vol.	3.1	0.13		
Grape	USA (CA)	WP	✈ foliar[7]	2.2	1.2-3.4	13	0
Grape	USA (CA)	WP	Foliar[7]	2.2	0.12-1.2	13	0
Grape	USA (except CA)	WP	✈ foliar[8]	1.1-2.2	0.60-3.4	13	0
Grape	USA (except CA)	WP	Foliar[8]	1.1-2.2	0.060-1.2	13	0
Linseed	UK	powder	seed treatment, 0.13 kg ai/ha				
Mango	Thailand	WP	high vol.	0.011-0.025 kg ai/tree	0.11-0.13		
Nectarine	USA	WP	✈ foliar[9]	2.2-4.5	1.2-4.8	27	0
Nectarine	USA	WP	Foliar[9]	2.2-4.5	0.096-2.4	27	0
Oilseed rape	UK	powder	seed treatment, 0.13 kg ai/ha				
Peach	USA	WP	✈ foliar[10]	2.2-4.5	1.2-4.8	36	0
Peach	USA	WP	Foliar[10]	2.2-4.5	0.060-2.4	36	0
Peanut	Thailand	WP	seed treat 1.5 g ai/kg seed				
Pear	UK	WG, WP	Foliar	2.7-2.9			7
Pear	UK	WG, WP	post-harvest dip		0.1		
Pear	USA	WP	post-harvest spray or dip		0.15		
Plum, prune	USA (east)	WP	✈ foliar[11]	3.4	1.8-3.6	30	0
Plum, prune	USA (east)	WP	Foliar[11]	3.4	0.12-1.8	30	0
Plum, prune	USA (west)	WP	✈ foliar[12]	2.2-3.4	1.2-3.6	30	0
Plum, prune	USA (west)	WP	Foliar[12]	2.2-3.4	0.080-1.8	30	0
Pome fruit	Germany	WP	Foliar	1.5-1.9	0.1-0.13	13[n]	14
Rice	Thailand	WP	seed treat 1.5 g ai/kg seed				
Soya bean	Thailand	WP	high vol.		0.075-0.10	4-5[n]	
Strawberry	UK	WP	Foliar	2.8-5.5			
Strawberry	USA	WP	✈ foliar[13]	1.7-3.4	0.90-3.6	27	0

Crop	Country	Form	Application			Max total	PHI, days
			method	rate per applic. kg ai/ha	spray conc. kg ai/hl	Applic kg ai/ha per crop cycle	
Strawberry	USA	WP	Foliar[13]	1.7-3.4		27	0
Sweet corn	UK	WP	seed treat 1.04 g ai/kg seed				
Tomato	UK	WP	foliar, gl[14]		0.25		

✈: aerial application ^{n} number of applications

[1]Apply at popcorn, bloom and petal fall stages and up to 5 weeks after petal fall. Hulls may be fed to livestock.
[2]USA (east). Apply at 5- to 7-day intervals as needed to maintain control in prebloom, bloom, petal fall and first cover sprays. Apply at 10 to 14 day intervals in second and later cover sprays.
USA (west). Make 1 or 2 applications with late cover sprays and 1 final spray before harvest.
[3]Apply in red bud, bloom, 75% petal fall, and cover sprays.
[4]Start spray programme when buds swell or have loose scales. Repeat at 7-day intervals through blossom period. Repeat at 7- to 10-day intervals from late bloom.
[5]Begin at mid-bloom, repeat at 7- to 10-day intervals until maturity.
[6]Apply in pre-bloom, bloom, petal fall, shuck, cover and pre-harvest sprays.
[7]Make 2 applications before bloom and 1 immediately after bloom. Repeat periodically, making 3 cover applications before the bunches close.
[8]Begin application at shoot length of ½ to 1½ inches, continue at 10- to 14-day intervals as necessary.
[9]Apply in full pink, bloom, petal fall, shuck, cover and pre-harvest sprays.
[10]Apply in full pink, bloom, petal fall, shuck stages and in cover and pre-harvest sprays. When conditions are favourable, make applications at 3- to 4-day intervals during bloom to control blossom blight. Then repeat application at 7- to 14-day intervals as needed to maintain control. Continue applications through harvest if conditions favour brown rot.
[11]Apply in full pink, bloom and petal fall sprays. Repeat applications at 7- to 14-day intervals as needed to maintain control. Continue applications through harvest if conditions favour brown rot.
[12]Apply at green bud, popcorn, bloom and petal fall stages. Repeat in cover sprays as conditions warrant.
[13] Begin applications when new growth starts in the spring and before fruit starts to form. Repeat at 7- to 14-day intervals. Under conditions favourable to fruit rot, continue applications through harvest period treating immediately after each picking.
[14]Glasshouse

RESIDUES RESULTING FROM SUPERVISED TRIALS

Details of supervised residue trials are summarized in Tables 2-6.

Table 2 *Apples, pears*. USA, Canada.
Table 3 *Cherries, nectarines, plums*. USA.
Table 4 *Grapes, blueberries, strawberries*. USA, Germany, Canada.
Table 5 *Radishes*. Germany.
Table 6 *Chives*. Germany.

Where residues were not detected the results are recorded in the Tables as less than the limit of determination (LOD), e.g. <0.01 mg/kg. Residues, application rates and spray concentrations have generally been rounded to 2 significant figures or, for residues near the LOD to 1 significant figure. Residues were frequently detected in samples from field control plots, but mostly at or about the LOD. The prefix "c" indicates samples from control plots. Residues are not corrected for analytical recoveries or field controls.

The trials were reported on summary sheets as well as in detailed summaries.

The 1994 JMPR reviewed extensive data on the stability of captan residues during freezer storage. They were stable as field-incurred residues in apples stored at -20°C for 14 months, the longest period tested. Samples in many of the trials had been stored for 6 to 14 months before analysis, but the longest periods of storage were from trials 16038, 12644 and 10068 for 25, 18 and 18 months respectively.

In many of the apple trials apples from replicate plots were composited for analysis.

In trials on applies in 1973-75 in the USA, plot sizes ranged from 1 tree to 0.4 ha. A hand-gun sprayer was used in some trials but the spraying equipment was not identified in others. Captan was applied by hand-gun sprayers to plots of single, 2 or 16 apple trees in 4 trials in 1976 (trials 06081, 12057, 13628 and 16075).

Captan was applied to run-off using hand-gun single nozzle sprayers in two trials on apple trees in 1976 (trials 3513 and 3514). The trial design consisted of 2 or 4 single-tree replicates. Harvested apples were processed (Table 7).

In four of the US trials on apples in 1978 no information was available on the sprayers or the plot size. In one trial captan was applied by hand-gun sprayer to single-tree plots. The trees were planted at 69/acre (170/ha), which would accommodate large trees. Air-blast or high-pressure hand-gun sprayers were used for application in the US trials of 1980. The plot sizes varied from 1 to 5 trees. In one other 1978 trial and in a 1980 trial captan was applied by mist blowers.

Pear trees in plots of 1 tree to 0.4 ha were treated with captan using hand-gun sprayers in the Stauffer trials from 1973 to 1978. Samples in trials 21776 and 21777 were stored for 12 months before analysis. The relatively high levels of THPI in samples from trial 21776 suggested some losses of captan during storage.

No information was available on the sprayers used in the Chevron trials on pears in 1978 in the USA (Table 2). A plot consisted of 12 trees; 10 mature pears were taken as the field sample. In a set of trials in the USA in 1980 captan was applied to pear trees by tractor sprayer on a commercial scale to 2 ha blocks. Analytical recoveries of captan in theses trials were variable: 63%, 70%, 79%, 82%, 86% and 100%.

Hand-gun or air-blast-sprayers were used to apply captan in the cherry trials in the USA from 1975 to 1980 (Table 3). The plot or replicate sizes varied from one tree to a 2 ha block, but were commonly 1-4 trees. In some trials cherries from replicates were composited for analysis. In trial 5062 the sample for analysis consisted of whole fruit + stems, but in most trials the nature of the sample for analysis was not explicitly described. Samples were stored from 4 to 15 months before analysis. Freezer storage studies reviewed by the 1994 JMPR had demonstrated that incurred residues of captan in cherries were stable in storage at -20°C for 12 months, the longest time tested.

Captan was applied with fan-blower sprayers to nectarine trees in a series of trials in the USA in 1975 (Table 3). The plot sizes ranged from 4 to 125 trees. Samples were generally stored for 5-6 months before analysis but in trials 10690 and 10691 the storage periods were 24 and 14 months respectively. The conditions of storage were not explicitly stated, but 24 months storage even under ideal conditions is probably too long. The freezer storage data on cherries can be used as a guide for nectarines. An analytical problem may have occurred with trials 06474, 10693, 10698, 10707 and 10709, samples from which were all analysed on the same day, because captan was detected in the control samples at a level of 0.13 mg/kg.

A high-pressure hand-gun sprayer was used for application to single-tree plots in two nectarine trials in the USA in 1978. The levels of THPI in some samples suggest a loss of captan during sample storage for 15 months. The trial design in a nectarine trial in the USA in 1980 was 4 single-tree replicates. Field samples consisted of 10 mature fruit. No information was available on the sprayer.

Captan was applied by an air-blast sprayer to a plot of 64 trees in the US trial on plums in 1975. The plot sizes were 6 trees in the 1977 trial and one in the trials of 1978. Samples were stored for 13 and 15 months; evidence of some breakdown of captan during sample storage was provided by the relatively high levels of THPI.

A fan duster was used to apply a captan dust formulation in a plum trial (5053) in the USA in 1980 (Table 3). A sample from the control plot in this trial was contaminated with captan (1.9 mg/kg). No information on the sprayers was available in the other 2 plum trials. The trial design was 4 single-tree replicates, and the sample size 20 mature plums or prunes.

Boom sprayers were used to treat grapes with captan in trials in the USA in 1976 (Table 4). The plot sizes were 1 or 2 rows of 90 or 120 m, 22 vines or 0.4 ha. In two trials in 1978 captan was applied by a portable mist blower or a power sprayer with a hand-gun. The plot in trial 12149 was small, consisting of 2 vines. In trial 4627 there were 4 replicate plots, each consisting of 4-5 m of row. Power-driven dusting equipment was used to apply a dust formulation to grapes in three trials in 1979 in the USA. In one of the trials the grapes were also sprayed with a WP formulation. Trials 4965 and 4967 were on a commercial scale, involving treatment of 130 and 13 ha respectively. The design for trial 4978 was 85 vines per replication and 4 replications per treatment. Captan contamination, 1.8 mg/kg, occurred in the control plot.

Grape plots consisting of 4 rows of vines 120 m long were treated with captan and the grapes harvested for processing (Tables 9 and 10) in two US trials in 1980. The grapes were stored at 10°C for 125-152 days before processing. In trial 5049 the grapes in closed plastic bags were fumigated 3 times with methyl bromide during storage. Grapes in trial 5051 were treated with a dust formulation using a tractor-mounted power-take-off duster. Grapes on 15-16 vines per plot were treated with an over-the-row hooded boom spray in trials 5162 and 5163. Grapes in trial 5164 were sprayed with a CO_2-pressurized single cone hand sprayer. The trial was very small, consisting of 4 replicates of single-vine plots.

In a series of grape trials by a captan task-force in the USA in 1986 captan was applied at a rate of 2.2 kg ai/ha and grapes were harvested on the day of the final application for residue analysis. The plot sizes ranged from 8.5 m^2 to 84 m^2 and captan was applied using CO_2-pressurized back-pack sprayers or a mist blower.

Information on grape trials in Germany in 1974 and 1977 (Table 4) was available only as summary tables. The trials were not further evaluated.

Captan was applied by power mist blower to a blueberry plot of 0.8 ha in a trial in the USA in 1976.

Strawberry plots, each of a 5 m row, were treated with captan in a 1975 trial in the USA. Samples were stored for 10 months before analysis. In the Stauffer strawberry trials of 1976-7 captan was applied by plot sprayers or tractor-mounted boom sprayers to plots of 4 m^2, a 5 m row, 0.04 ha or 0.2 ha. Strawberry samples were stored for 4-17 months before analysis, but the storage conditions were not available. The 1994 JMPR reported that field-incurred residues of captan in strawberries had decreased by 26% during 14 months storage at -20°C, but most of the decrease had occurred in the first 3 months.

Captan was applied to strawberries by boom sprayers in two US trials in 1976. In one trial a plot was 2×15 m of bed and in the other a plot was 84 m^2. Under the growing conditions in trial 3517 the interval from blossom to ripe berries was about 25 days. Mature ripe berries with caps removed were analysed.

In a 1978 strawberry trial (4628) captan was applied by tractor-mounted boom sprayer. The control plot suffered contamination to the extent that the captan level in the strawberries was 7.7 mg/kg. In the Stauffer trials of 1978 the plot sizes were 0.1-0.4 ha and captan was applied by commercial boom sprayer or hand-gun sprayer. Samples were stored for 21 days (trial 20856) or 15-

16 months (trials 17637, 17639). The levels of THPI found in samples from trial 17637 suggest some loss of captan during storage.

Information on trials on strawberries (Table 4), radishes (Table 5) and chives (Table 6) in Germany was available only as summary tables. The trials were not further evaluated.

Table 2. Captan and THPI residues in pome fruit resulting from foliar application of captan in supervised trials in the USA and Canada. Double-underlined residues are from treatments according to GAP and were used for estimation of maximum residue levels. Single-underlined residues are from treatments according to GAP, but not close to maximum GAP.

Crop Country, year (variety)	Application				PHI, days	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		Captan	THPI	
APPLES								
USA (WI), 1973 (Cortland)	WP	0.43	0.046	9	8	0.44		Stauffer A-10068
USA (WI), 1973 (Cortland)	WP	0.56	0.060	9	8	0.62		Stauffer A-10068
USA (WI), 1973 (McIntosh)	WP	0.43	0.046	9	8	0.29		Stauffer A-10068
USA (WI), 1973 (McIntosh)	WP	0.56	0.060	9	8	0.30		Stauffer A-10068
USA (CT), 1974 (McIntosh)	WP	2.2	0.12	10	7	3.0 1.8		Stauffer A-10208
USA (IL), 1974 (Golden Delicious)	WP	1.7	0.045	17	0	0.66		Stauffer A-10070
USA (IL), 1974 (Red Delicious)	WP	1.7	0.045	17	0	0.50		Stauffer A-10070
USA (NC), 1974 (Red and Golden Delicious)	WP	3.9	0.12	10	14	0.74		Stauffer A-10069
USA (NH), 1974 (Cortland)	WP	3.4	0.12	12	26	1.8 c 0.37		Stauffer A-10222
USA (NH), 1974 (McIntosh)	WP	0.84	0.90	8	4	3.0 c 0.22		Stauffer A-06034
USA (NH), 1974 (McIntosh)	WP	2.2	0.080	10	8	6.6		Stauffer A-10203
USA (NH), 1974 (McIntosh)	WP	3.4	0.12	12	26	1.8 c 0.31		Stauffer A-10222
Canada (NS), 1975 (McIntosh)	WP	2.5	0.075	8	7 65	0.27 0.06		Stauffer A-10259
USA (WV), 1975 (Golden Delicious)	WP	3.4	1.8	9	2	1.6		Stauffer A-12643
USA (WV), 1975 (Rome)	WP	3.4	1.8	8	24	0.24		Stauffer A-12644
USA (NY), 1976 (7 apple varieties)	WP	1.1	0.040	15	21	0.20 0.09		Stauffer A-16075
USA (CA), 1976 (Red Delicious)	WP	4.5	0.12	1	0	14 12		Stauffer A-12057
USA (MI), 1976 (Rome)	WP	2.2	0.036	12	28	0.73		Stauffer A-13628
USA (MI), 1976 (Rome)	WP	1.1	0.018	12	28	0.33		Stauffer A-13628
USA (NJ), 1976 (Red Delicious)	WP	2.8	0.12	11	0	4.8 5.4 c 0.01		Chevron 3513
USA (NY), 1976 (McIntosh)	WP	1.7	0.30	10	27	0.18		Stauffer A-16038
USA (PA), 1976 (Rome)	WP	2.2	0.12	10	0 20 43	3.2 2.3 2.8 2.8 0.74 0.96 0.96 0.74 0.16 0.15 0.26 0.48 c 0.40		Stauffer A-06081
USA (VA), 1976 (Golden Delicious)	WP	2.8	0.12	13	0	2.7 3.0 c 0.02		Chevron 3514

Crop Country, year (variety)	Application				PHI, days	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		Captan	THPI	
USA (NJ), 1978 (Golden Delicious)	WP	2.8	0.12	13	0 1 7 14 21 28	3.5 5.6 3.3 5.0 3.7 2.9 2.7 2.4 3.8 3.6 2.8 3.3 c 0.03 0.04		Chevron 4430
USA (NJ), 1978 (Red Delicious)	WP	2.8	0.12	13	0 1 7 14 21 28	7.7 5.5 6.1 4.8 4.5 6.0 5.2 6.7 6.2 7.0 3.3 3.2 c 0.01		Chevron 4429
USA (NJ), 1978 (Rome Beauty)	WP	2.4 +3.5	0.12	1 +2	0 1 7 14 21 28	2.4 2.2 3.7 2.7 2.0 1.9 1.9 2.0 1.8 1.9 1.7 1.4 c 0.02		Chevron 4431
USA (NY), 1978 (Idared)	WP	3.4	0.36	9	0 3 14	16 5.0 3.8	<0.05 0.05 0.07	Stauffer A-17648
USA (PA), 1978 (Jonathan)	WP	3.4	0.12	1	0 1 3 7 14	1.6 1.1 0.63 0.40 0.06		Stauffer A-18413
USA (WV), 1978 (Golden Delicious)	WP	2.2	0.080	1	0 1 3 7 14	0.92 0.78 0.54 0.26 0.08 c 0.13		Stauffer A-18425
USA (CA), 1980 (Jonathan)	WP	1.1	0.12	2	1 3 7 10 13	1.5 1.5 1.3 1.0 0.75		Stauffer A-23994
USA (CA), 1980 (Yellow Delicious)	WP	1.1	0.12	2	1 3 7 10 13	2.2 1.5 1.3 1.3 0.67		Stauffer A-23994
USA (MO), 1980 (Red Delicious)	WP	5.0 +5.0 +1.0 +2.0 +3.0 +4.0 +5.0 +5.0 +5.0	0.18 +0.18 +0.36 +0.72 +1.1 +1.4 +0.18 +0.18 +0.18	9	1	6.6 1.2 1.3 2.2 3.3 c 0.10	0.05 0.01 0.02 0.03 0.05	Chevron 5207
USA (NY), 1980 (McIntosh)	WP	4.5	0.12	14	1 16 31	4.8 5.1 5.7 3.2 1.8 2.4 c 0.03 0.07	0.17 0.15 0.12 0.06 0.06 0.08 c 0.01	Chevron 5066B

Crop Country, year (variety)	Application				PHI, days	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		Captan	THPI	
USA (NY), 1980 (McIntosh)	WP	4.5	0.72	14 13 12	1 16 31	5.5 6.1 3.0 3.1 0.43 0.50 c 0.18 0.05	0.09 0.12 0.03 0.03 <0.01 <0.01 c 0.01	Chevron 5066C
USA (NY), 1980 (Wealthy)	WP	4.5	0.72	14 13 12	1 16 31	4.0 3.6 3.5 3.0 3.1 2.5 c 0.03 0.09	0.13 0.10 0.08 0.07 0.07 0.05	Chevron 5066A
PEARS								
USA (NH), 1973 (Bartlett)	WP	0.84 -1.2	0.90 -1.3	8	14	0.28 c 0.26		Stauffer A-06034
USA (NH), 1974 (Clapps Favorite)	WP	2.1 -3.4	0.076 -0.12	10	8	4.1 c 0.10		Stauffer A-10203
USA (NY), 1976 (Bartlett)	WP	2.5	0.090	2	41	<0.05		Stauffer A-16039
USA (WA), 1978 (Anjou)	WP	6.7	0.12	2	0 1 3 7 14	1.6 1.0 0.5 0.6 0.3	2.3 1.4 0.16 1.3 0.05 c 0.11	Stauffer A-21776
USA (WA), 1978 (Bosc)	WP	6.7	0.12	2	0 1 3 7 14	1.5 1.0 0.5 0.6 0.3	<0.05 <0.05 <0.05 <0.05 <0.05	Stauffer A-21777
USA (CA), 1978 (Bartlett)	WP	2.5 +3.4 +3.8 +4.2 +4.7 +4.2 +5.1		2 +2 +2 +1 +1 +1 2	0 1 7 14 21	0.04 0.09 0.07 0.03 0.04 0.10 0.07 0.09 0.07 0.09 c 0.03 0.06 0.02 c 0.09		Chevron 4432
USA (CA), 1978	WP	2.2	0.24	1	2	0.66 0.98 0.94 0.54 0.40 0.31 0.71 0.68 c 0.04 0.05 c 0.06 0.06		Chevron 4443
USA (NY), 1980 (Bartlett)	WP	3.9	0.24	9	1 14 21	0.52 0.19 0.18 0.18 0.20 0.12 c 0.03 0.05 0.04	0.02 0.01 0.01 0.01 0.01 0.01	Chevron 5074A
USA (NY), 1980 (Seckel)	WP	3.9	0.24	9	1 14 21	1.1 1.9 2.6 1.5 0.04 1.5 c 0.06 0.06 0.09	0.05 0.07 0.07 0.05 0.04 0.04 c 0.01 0.01	Chevron 5074B
USA (NY), 1980 (Flemish Beauty)	WP	3.9	0.24	9	1 14 21	0.65 0.50 0.34 0.43 0.24 0.20 c 0.08 0.06 0.08	0.04 0.03 0.01 0.01 0.02 0.02 c 0.01 0.01	Chevron 5074C

Crop Country, year (variety)	Application				PHI, days	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		Captan	THPI	
USA (NY), 1980 (Clapp's)	WP	3.9	0.24	9	1 14 21	0.51 0.58 0.52 0.34 0.44 0.36 c 0.05 0.05 0.04	0.02 0.03 <0.01 0.02 0.02 0.02	Chevron 5074D
USA (NY), 1980 (Bosc)	WP	3.9	0.24	9	1 14 21	0.54 0.34 0.84 0.35 0.32 0.21 c 0.04 0.05 0.03	0.03 0.03 0.03 0.01 0.02 0.01 c 0.01 0.01	Chevron 5074E

c: sample from control plot

Table 3. Captan and THPI residues in stone fruit resulting from foliar application of captan in supervised trials in the USA. Double-underlined residues are from treatments according to GAP and were used for estimation of maximum residue levels. Single-underlined residues are from treatments according to GAP, but not close to maximum GAP.

Crop, State, Year, (Variety)	Application				PHI, days	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		Captan	THPI	
CHERRIES								
MI, 1975 (Sweet)	WP	3.4	0.12	5	0	0.55		Stauffer A-12730
MI, 1975 (Sour)	WP	3.4	0.12	5	0	2.6		Stauffer A-12730
MI, 1976 (Montmorency)	WP	2.2	0.080	6	0 1 3 7 12	13 20 15 5.9 4.3		Stauffer A-13621
MI, 1976 (Montmorency)	WP	2.2	0.080	6	0 1 3 7 14	21 11 19 20 8.2 c 0.1		Stauffer A-13622
MI, 1976 (Montmorency)	WP	1.7	0.060	6	0 1 3 7 12	12 14 8.3 5.5 1.9		Stauffer A-13626
MI, 1976 (Montmorency)	WP	1.7	0.060	6	0 1 3 7 14	20 16 9.7 16 12 c 0.1		Stauffer A-13627
CA, 1976 (Bing)	WP	5.6	0.24	2	1	7.9 c 0.1		Stauffer A-14005
CA, 1977 (Montmorency)	WP	3.4	0.12	7	10	8.2 7.4		Stauffer A-17615
NY, 1977 (Emperor Francis and Napoleon)	WP	2.2	0.060	7	8	0.92		Stauffer A-17616
IL, 1977 (Montmorency)	WP	2.2	0.060	7	1	4.3		Stauffer A-19931

Crop, State, Year, (Variety)	Application				PHI, days	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		Captan	THPI	
NY, 1978 (Napoleon)	WP	1.1	0.12		0 1 3 7	17 5.3 11 8.3 c 0.11	1.6 0.55 1.0 1.5 c 0.19	Stauffer A-17635
NY, 1978 (Montmorency)	WP	1.1	0.12	5	0 1 3 7 10 14	16 15 9.9 7.4 7.1 2.0	1.8 3.0 1.9 2.2 1.6 0.36	Stauffer A-17638
MT, 1980 (Lambert)	WP	2.2	0.12	6	1 2 3 7 14	2.4 1.6 2.4 2.2 1.4		Stauffer A-27008
MT, 1980 (Lambert)	WP	2.2	0.96	6 8	0 1 3 1 8	5.5 3.4 4.7 2.3 2.8		Stauffer A-27013
CA, 1980	WP	14	0.24	4	1 6 7	7.9 4.9 3.3 c 0.03 0.03	0.26 0.49 0.37	Chevron 5062
NY, 1980 (Napoleon)	WP	4.5	0.12	8	1 7	3.7 7.2 7.1 4.6 c 0.03 0.26	0.52 0.49 0.30 0.35 c 0.01	Chevron 5165
NY, 1980 (Montmorency)	WP	3.9	0.71	7	1 7	17 12 6.8 11 c 0.03 0.04	0.83 0.51 0.21 0.32 c 0.02 0.01	Chevron 5166
NECTARINES								
IL, 1975 (Early Blaze)	WP	1.1	0.14	8	1	0.25 c 0.13		Stauffer A-06474
CA, 1975 (Regal)	WP	3.4	0.12	3	18	0.47 c 0.25		Stauffer A-10690
CA, 1975 (Late LeGrand)	WP	3.4	0.12	3	12	0.24		Stauffer A-10691
CA, 1975 (Late LeGrand)	WP	3.4	0.095	3	12	<0.05 c 0.13		Stauffer A-10693
CA, 1975 (Flame Kist)	SC	3.4	0.45	2	20	0.13		Stauffer A-10696
CA, 1975 (September Grand)	WP	2.2	0.24	2	18	0.20 c 0.13		Stauffer A-10698
CA, 1975 (September Grand)	WP	4.5	0.16	2	18	<0.05 c 0.13		Stauffer A-10707
CA, 1975 (Flame Kist)	WP	4.5	0.19	2	17	<0.05 c 0.13		Stauffer A-10709
CA, 1978 (Grand Prize)	WP	6.7	0.12	1	0 1 3 7 14	3.3 3.0 2.0 2.1 0.7	<0.05 <0.05 0.11 0.39 0.40 c 0.43	Stauffer A-17775

Crop, State, Year, (Variety)	Application				PHI, days	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		Captan	THPI	
CA, 1978 (Arm King)	WP	6.7	0.12	1	0 1 3 7 14	2.6 2.3 1.2 1.1 0.8	1.0 0.44 0.44 0.59 <0.05	Stauffer A-17778
CA, 1980 (LeGrande)	WP	6.7	0.12	9	0 1 3 7 10	10 8.5 6.7 9.7 3.2 c 0.03-0.05	0.21 0.24 0.16 0.20 0.16 c 0.01	Chevron 5063
PLUMS, PRUNES								
CA, 1975 (Casselman)	WP	3.4	0.12	3	13	<0.05		Stauffer A-10692
NY, 1977 (Purple Plums)	WP	3.4	0.12	13	2	0.71		Stauffer A-17622
NY, 1978 (Fellenburg)	WP	3.4	0.36	9	0 3 7 10	7.9 4.8 3.4 2.6	1.0 0.16 0.17 0.22	Stauffer A-17649
CA, 1978 (Queen Anne)	WP	6.7	0.12	1	0 1 3 7 14	0.64 0.47 0.31 0.81 0.54 c 0.09	0.67 0.34 0.38 0.60 0.42	Stauffer A-17781
CA, 1980 (Santa Rosa)	WP	6.7	0.12	6	0 1 3 7 10	5.5 5.3 4.0 4.3 4.7 c 0.03 0.03 0.03 c 0.03 0.04	0.07 0.09 0.06 0.09 0.11 c 0.01	Chevron 5052
CA, 1980 (French)	WP	6.7	0.12	6	0 1 3 7 10	4.6 8.6 8.9 6.1 8.8 c 0.02 0.04 0.04 c 0.04 0.05	0.11 0.14 0.16 0.10 0.15 c 0.01	Chevron 5052A
CA, 1980 (French)	dust	5.6		3	0 1 4 8 11	7.3 5.6 4.6 5.5 5.7 6.0 6.0 4.4 5.0 7.1 c 1.9	0.08 0.07 0.05 0.07 0.03 0.03 0.06 0.04 0.12 0.12c 0.03	Chevron 5053

c: sample from control plot

Table 4. Captan and THPI residues in grapes, blueberries and strawberries resulting from foliar application of captan in supervised trials in the USA, Germany and Canada. Double-underlined residues are from treatments according to GAP and were used for estimation of maximum residue levels. Single-underlined residues are from treatments according to GAP, but not close to maximum GAP.

Crop, Country, year (variety)	Application				PHI, days	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		Captan	THPI	
GRAPES								
USA (CA), 1976 (Golden Muscat)	WP	1.1	0.060	2	0	11		Stauffer A-12072
USA (CA), 1976 (Thompson Seedless)	WP	1.1	0.12	5	73	0.32 0.35 c 0.23		Chevron 3511
USA (CA), 1976 (White Malaga)	WP	1.1	0.12	1	0	11		Stauffer A-12155
USA (MD), 1976 (Chancellor)	WP	1.1	0.20	9	0	2.6		Stauffer A-10190
USA (NY), 1976 (Concord)	WP	3.4	0.18	5	43	0.83 c 0.03		Chevron 3512
USA (FL), 1978 (F4-36)	WP	2.2	0.16	6	7 14	0.24 1.6 0.22 053 c 0.01		Chevron 4627
USA (CA), 1978 (Thompson)	WP	11	0.12	1	0 1 3 7	30 29 20 17	<0.05 <0.05 <0.05 <0.05	Stauffer A-12149
USA (CA), 1979 (Ruby Cabernet)	dust	3.9		4	2 4 6 10 17	1.1 0.45 0.72 1.2 2.8 1.8 0.56 0.68 0.48 1.2 c 0.07	0.03 0.01 0.03 0.03 0.05 0.03 0.02 0.03 0.03 0.02 c 0.01	Chevron 4965
USA (CA), 1979 (Ruby Cabernet)	dust	3.9		4	1 3 7 14	1.0 1.8 1.0 0.98 0.28 0.31 0.24 0.23 c 0.08	0.04 0.04 0.03 0.03 0.04 0.02 0.01 0.02 c 0.01	Chevron 4967
USA (CA), 1979 (Zinfandel)	WP + dust	2.2 +4.5	0.60 +1.2	2 + 3	63	4.1 5.5 2.3 2.4 c 1.8	0.18 0.18 0.20 0.17 c 0.14	Chevron 4978
USA (CA), 1980 (Emperor)	WP	3.4	0.36	3	1 3 7 10 13	10 10 6.9 6.4 5.5		Stauffer A-23992
USA (CA), 1980 (Grenache)	dust	4.0		10 10 9 8 3	1 7 14 28 92	1.1 4.7 5.1 6.5 1.2 c 0.05-0.09	0.03 0.38 0.31 0.16 0.03 c 0-0.01	Chevron 5051
USA (CA), 1980 (Thompson Seedless)	dust	4.0		9 9 8 7 3	1 7 14 28 86	13 9.5 13 11 5.3 7.6 11 8.0 1.7 1.6 c 0.35	0.42 0.42 0.45 0.20 0.25 0.37 0.20 0.19 0.07 0.05 c 0.02	Chevron 5049

Crop, Country, year (variety)	Application				PHI, days	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		Captan	THPI	
USA (CA), 1980 (Thompson Seedless)	WP + dust	2.2 +4.0	0.080 +0.14	3 + 6	1 7	7.0 13 10 12	0.57 0.62 0.79 0.97	Chevron 5050
USA (CA), 1980 (Thompson Seedless)	WP + dust	2.2 +4.0	0.080 +0.14	3 + 3	14	7.1 6.4	0.49 0.55	Chevron 5050`
USA (CA), 1980 (Thompson Seedless)	WP + dust	2.2 +4.0	0.080 +0.14	3 + 4	28	3.7 4.8 c 0.43	0.39 0.33 c 0.03	Chevron 5050`
USA (CA), 1980 (Thompson Seedless)	WP	2.2	0.080	3	85	1.2 <u>1.6</u>	0.06 0.08	Chevron 5050`
USA (NY), 1980 (Aurora)	WP	2.2	0.16	3	13	0.77 <u>1.1</u> c 0.04	0.05 0.14 c 0.01	Chevron 5162
USA (NY), 1980 (Chancellor)	WP	2.2	0.12	7	33	<u>9.4</u> 9.1 c 0.02	0.21 0.19 c 0.01	Chevron 5164
USA (NY), 1980 (Elvira)	WP	2.2	0.096	4	9	1.2 <u>2.3</u> c 0.05	0.10 0.14	Chevron 5163
USA (CA), 1986 (Emperor)	WP	2.2	0.12	6	0	<u>3.7</u> 1.3	<0.05 <0.05	86256
USA (CA), 1986 (Emperor)	WP	2.2	0.12	6	0	<u>7.4</u> 5.8 c 8.9	<0.05 <0.05	86994
USA (CA), 1986 (Thompson Seedless)	WP	2.2	0.12	6	0	11 <u>22</u>	0.20 0.28	86814
USA (CA), 1986 (Thompson Seedless)	WP	6.7	0.36	6	0	179 72	1.9 0.69	86814
USA (MI), 1986 (Concord)	WP	2.2	0.12	6	0	<u>11</u> 8.1	0.14 0.12	86218
USA (NY), 1986 (Aurora)	WP	2.2	0.12	6	0	<u>7.2</u> 6.4	0.15 0.14	86719
USA (NY), 1986 (Concord)	WP	2.2	0.12	5	0	<u>6.4</u> 4.5	0.14 0.18	86549
USA (WA), 1986	WP	2.2		6	0	0.93 <u>1.3</u>	<0.05 <0.05	86080
GRAPES, WINE								
Germany, 1974 (Bacchus)	WP	1.6-2.4	0.08	6	0 28 47 77	2.2 0.31 0.11 0.12		BBA TR1074
Germany, 1974 (Müller-Thurgau)	WP	2.0		7	3 45	1.8 0.28		BBA KH1074
Germany, 1974 (Müller-Thurgau)	WP	1.6	0.32	7	0 28 42	7.4 1.8 1.4		BBA GE1074
Germany, 1974 (Müller-Thurgau)	WP		0.08	10	0 21 41	7 2.0 0.48		BBA WU1074
Germany, 1974 (Müller-Thurgau)	WP	1.3	0.33	9	0 28 42 57	8.1 0.74 1.4 0.23		BBA OP1074
Germany, 1974 (Müller-Thurgau)	WP	4.0	0.5	7	3 45	14 2.5		BBA 1174KH
Germany, 1974 (Müller-Thurgau)	WP	5×2.5 +2×3.2	0.64	7	0 28 42	10 2.2 3.2		BBA GE1174
Germany, 1974 (Bacchus)	WP	2.5 -3.7	0.13	6	0 28 47 77	18 0.61 <0.02 0.96		BBA TR1174

Crop, Country, year (variety)	Application				PHI, days	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		Captan	THPI	
Germany, 1977 (Müller-Thurgau)	WP	1.4	0.35	6	0 14 28 35 50	3.8 4.1 4.1 2.8 2.5		BBA 166684
Germany, 1977 (Müller-Thurgau)	WP	0.7- 1.1	0.09	10	0 14 35 46	13 7.2 5.3 3.0 4.7 3.3 4.3 3.0		BBA 13845
Germany, 1977 (Bacchus)	WP	4×1.8 +6+2.2	0.09	10	0 14 28 35 47	5.4 4.9 3.7 6.3 2.0		BBA TR1277
Germany, 1977	WP	1.8	0.09	8	0 14 28 35 55	4.9 1.7 3.3 0.79 0.39		BBA 1377
BLUEBERRIES								
USA (OR), 1976 (Highbush)	WP	2.8	0.94	4	0	6.5 c1.2		Stauffer A-14201
STRAWBERRIES								
Canada (ONT), 1975	WP	3.4	0.30	5	0 1 2	27 19 11		Stauffer A-10269
Canada (ONT), 1975	WP	1.1	0.10	5	0 1 2	6.1 6.1 4.7		Stauffer A-10269
Germany, 1961 (Senga Sengana)	WP	0.75	0.13	1	0 3 7 14	1.8 1 0.7 0.4		BBA
Germany, 1962 (Senga Sengana)	WP	0.75	0.13	1	0 3 7 14	2.2 2 1.1 0.05		BBA
Germany, 1964	WP	1.3	0.13	2	8	<0.1		BBA Cpt 1/1964
USA (NY), 1975	WP	1.1	0.20	4	1 7	0.18 0.45 c 0.09		Stauffer A-12731
USA (CA), 1976 (Shasta)	WP	3.4	0.18	3	12	2.9		Stauffer A-12077
USA (CA), 1976 (Shasta)	WP	3.4	0.18	3	0	13[1]		Stauffer A-12076
USA (CA), 1976 (Heidi, G-3)	WP	3.4	0.18	11	0	7.3 6.9 c 0.03		Chevron 3517
USA (NJ), 1976 (Sparkle)	WP	3.4	0.18	10	0	3.3 3.4 c 0.02		Chevron 3518
USA (OR), 1976 (Hood)	SC	1.1	0.14	3	12	0.73		Stauffer A-14202
USA (OR), 1976 (Northwest)	SC	1.1	0.24	3	13	0.18		Stauffer A-14203
USA (VA), 1976 (Red Chief)	WP	1.7	0.18	4	0 1 3	1.5 0.66 1.0		Stauffer A-10192

Crop, Country, year (variety)	Application				PHI, days	Residues, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		Captan	THPI	
USA (VA), 1976 (Red Chief)	WP	1.7	0.18	4	0 3	1.6 1.2		Stauffer A-10192
USA (VA), 1976 (Red Chief)	WP	1.1	0.12	4	0 1 3	0.97 0.77 1.1		Stauffer A-10192
USA (WI), 1976 (Suregrow, Raritan)	WP	2.2	0.24	6	21	0.22		Stauffer A-13603
USA (VA), 1977 (Red Chief)	WP	2.2	0.24	4	0 1 3	2.8 3.0 2.3		Stauffer A-18620
USA (VA), 1977 (Red Chief)	WP	2.2	0.24	4	0 1 3	3.0 2.4 4.0		Stauffer A-18620
USA (VA), 1977 (Red Chief)	WP	3.4	0.36	4	0 1 3	5.8[2] 3.9 5.6 c 0.11[3]		Stauffer A-18620
USA (FL), 1978 (Tioga)	WP	3.4	0.36	16 20	0 3 7	8.0 8.3[4] 6.0 6.8 7.9 6. c 7.7		Chevron 4628
USA (CA), 1978 (Driscoll G-3)	WP	3.4	0.18	1	0 1 3 7 14	6.4 5.4 4.0 1.3 0.52		Stauffer A-20856
USA (NY), 1978 (Darrow)	WP	3.4	0.29	1	1 3 6 8	3.9 2.1 1.6 1.1 c 0.02	<0.05 0.07 0.54 <0.05 c 0.20	Stauffer A-17639
USA (NY), 1978 (Raritan)	WP	2.2	0.24	6	1 3 7	1.9 1.4 1.0	0.51 0.34 0.65 c 0.15	Stauffer A-17637

c: sample from control plot
[1]Sample stored 16 months before analysis
[2]Samples stored 13 months before analysis
[3]Control plot for the 3 trials in 18620
[4]Discount this trial because of control contamination

Table 5. Captan residues in small radishes (grown indoors) resulting from application of captan in supervised trials in Germany in 1976. Captan was applied pre-emergence by watering at 4 l/m^2 at a rate of 8 g product/m^2.

Year (variety)	Application				PHI, days	Captan, mg/kg	Ref.
	Form	kg ai/ha	kg ai/hl	No.			
1975 (Rota)	WP	66	0.17	1	21	<0.03	BBA 57/75
1975 (Cherry belle)	WP	66	0.17	1	112	<0.03	BBA 2553
1975 (Hilmar Treib)	WP	66	0.17	1	47	<0.03	BBA 2709
1975 (Neckar-perle)	WP	66	0.17	1	47	<0.03	BBA 2710
1975 (Cherry belle)	WP	66	0.17	1	97	<0.03	BBA 3200

Year (variety)	Application				PHI, days	Captan, mg/kg	Ref.
	Form	kg ai/ha	kg ai/hl	No.			
1976 (Cherry belle)	WP	66	0.17	1	38	<0.03	BBA 2559
1976 (Karissima GS kalibriert)	WP	66	0.17	1	48	<0.03	BBA 1482
1976 (Roky)	WP	66	0.17	1	54	<0.03	BBA 72/76 75/76

Table 6. Captan residues in chives (grown indoors) resulting from application of captan in supervised trials in Germany in 1976. Captan was applied at emergence or sprouting by watering at 4 l/m^2 at a rate of 8 g product/m^2.

Variety	Application				PHI, days	Captan, Mg/kg	Ref.
	Form	kg ai/ha	kg ai/hl	No.			
Feinstengeliger	WP	66	0.17	1	12	20 32	BBA 4/75
Hybrid Hild 68	WP	66	0.17	1	22	1.4	BBA 1480
Hybrid Hild 68	WP	66	0.17	1	15	0.62 c 0.64	BBA 1481
Feinröhriger	WP	66	0.17	1	32	0.13	BBA 2708
Feinröhriger	WP	66	0.17	1	26	0.26	BBA 2706
	WP	66	0.17	1	19	3.5	BBA 2569

c: sample from control plot

FATE OF RESIDUES IN STORAGE AND PROCESSING

In processing

The Meeting was provided with information on the fate of captan during the processing of apples and grapes.

Details were not available on the process used for producing juice and pomace from apples in trials 3513 and 3514. Heating and cooking are very influential on the fate of captan, but there is no record of the heating and cooking conditions in these two trials. Apples were quartered, ground and the juice squeezed out. The results are shown in Table 7.

Table 7. Residues of captan in apples and apple products. More details on trials 3513 and 3514 are provided in Table 2.

Sample	Captan residues, mg/kg		Processing factor	
	Trial 3513	Trial 3514	Trial 3513	Trial 3514
Apple	4.8 5.4	2.7 3.0	-	-
Juice	1.6 1.5	0.63 1.1	0.30	0.30
Wet pomace	2.9 3.6	0.77 1.0	0.64	0.31
Dry pomace	0.29 0.34	0.07 0.30	.062	0.065

Details were not available on the process used for producing pomace from grapes in trials 3511 and 3512. In trial 3511 unwashed grapes with stems removed were crushed in a colander and the juice separated, leaving wet pomace. The results are shown in Table 8 and those from other processing trials in the USA in Tables 9-11.

Table 8. Residues of captan in grapes and grape pomace. More details on trials 3511 and 3512 are provided in Table 4.

Sample	Captan residues, mg/kg		Processing factor	
	Trial 3511	Trial 3512	Trial 3511	Trial 3512
Grapes	0.32 0.35	0.83		
Wet pomace	0.30 0.22	0.56 0.40	0.78	0.58
Dry pomace	0.04 0.04	0.08 0.13	0.12	0.13

Table 9. Residues of captan and THPI in fruit and processed fractions from grapes harvested 1, 7 and 86 days after the final captan application in trial 5049 in the USA in 1980. More detail on the trial is provided in Table 4.

Sample	Residues, mg/kg					
	PHI, 1 day		PHI, 7 days		PHI, 86 days	
	Captan	THPI	Captan	THPI	Captan	THPI
Grapes	12.6 9.5	0.42 0.42	13.2 10.7	0.45 0.20	1.74 1.59	0.07 0.05
Juice	11.6 16.9	2.98 3.11	17.3 16.6	2.36 2.25	0.94 1.15	0.31 0.35
Pomace	8.00 7.31	1.24 1.02	2.70 2.85	0.57 0.60	0.27 0.30	0.11 0.09
Raisins	8.80 15.8	2.96 3.00	18.8 12.1	2.19 2.57	3.48 3.58	0.31 0.38
Raisin waste	15.7 19.5	6.01 5.36	15.8 16.3	8.58 8.42	8.77 9.69	1.00 1.17

Table 10. Residues of captan and THPI in fruit and raisins from grapes harvested 1, 7, 14 and 28 days after the final captan application in trial 5050 in the USA in 1980. More detail on the trial is provided in Table 4.

Sample	Residues, mg/kg							
	PHI, 1 day		PHI, 7 days		PHI, 14 days		PHI, 28 days	
	Captan	THPI	Captan	THPI	Captan	THPI	Captan	THPI
Grapes	7.02 13.1	0.57 0.62	10.1 12.2	0.79 0.97	7.12 6.42	0.49 0.55	3.66 4.78	0.39 0.33
Raisins	6.00 8.77	2.60 2.66	21.3 16.1	3.08 3.06	16.1 17.2	2.48 2.16	15.1 12.7	1.91 1.62

Grapes were treated at the label rate (2.2 kg ai/ha) and at an exaggerated rate before harvest and processing into raisins, pomace and juice (Table 11, Smith, 1987). There was no statement about washing the grapes before processing, so it is likely that there was no washing step. Raisins were washed by vigorous shaking in deionised water for 2 minutes. The water wash was discarded and the procedure was repeated 3 times. No information was provided on the conditions of production of the raisins, drying of the pomace, or production of the juice, but it is unlikely that the juice was heated or most of the captan would have been converted to THPI.

Table 11. Residues of captan and THPI in fruit and processed fractions from grapes harvested on the day of the final captan application (Smith 1987, trial 86814). More detail is provided on the trial in Table 4.

Sample	Residues, mg/kg			
	Treatment 2.2 kg ai/ha		Treatment 6.7 kg ai/ha	
	Captan	THPI	Captan	THPI
Grapes	10.9 22.4	0.198 0.276	179 72	1.93 0.69
Raisins	79.5	7.67	188	11.1
Raisin waste	316	5.55	1080	14.3
Washed raisins	13.3	9.43	13.8	10.0
Wet pomace	21.0	0.40	24.2	0.30
Dry pomace	20.4	10.4	55.2	6.41
Juice	82.4	0.654	51.2	0.892

THPI residues in the juice, pomace and raisins arise from the THPI originally in the grapes and by conversion of captan to THPI during the process. Processing yields for THPI can be calculated from the following formula.

$$\text{Processing yield} = \frac{\text{THPI residues in juice, pomace or raisins}}{\text{captan residues in grapes X 0.503 + THPI residues in grapes}}$$

The factor 0.503 is the ratio of the molecular weight of THPI (151.16) to that of captan (300.6).

Processing factors for captan and processing yields for THPI calculated from the data in Tables 9-11 are shown in Table 12.

Table 12. Processing factors for captan and processing yields for THPI calculated from the data in Tables 9-11.

Process	Captan processing factor	THPI processing yield	Trial
Grapes → juice	1.29 1.42 0.63	0.51 0.36 0.37	5049
Grapes → juice	4.9 0.41	0.076 0.014	86814
Grapes → raisins	1.11 1.29 2.12	0.50 0.38 0.39	5049
Grapes → raisins	0.73 1.68 2.46 3.29	0.47 0.47 0.59 0.71	5050
Grapes → raisins	4.8 1.5	0.89 0.17	86814
Grapes → washed raisins	0.80 0.11	1.1 0.16	86814
Grapes → wet pomace	1.3 0.19	0.046 0.0047	86814
Grapes → dry pomace	1.2 0.44	1.2 0.010	86814
Grapes → pomace	0.69 0.23 0.17	0.19 0.09 0.11	5049

Residues in the edible portion of food commodities

The calculated processing factor for apple juice was 0.30, but no information was available on the heating and cooking processes. More detailed studies were available to the 1994 Meeting, which concluded that captan is not present in commodities such as canned juice because it is destroyed by cooking and other processing.

The processing factors for captan in the production of juice and raisins from grapes were highly variable, probably reflecting the sensitivity of captan residues to degradation when food is heated or cooked. The mean processing factors and ranges from the grape processing studies available to the present and the 1994 Meeting are grapes to juice 1.2 (range 0.23-4.9) and grapes to raisins 1.66 (range 0.11-4.8).

NATIONAL MAXIMUM RESIDUE LIMITS

The Meeting was informed of the US tolerances for the following five commodities.

Commodity	MRL, mg/kg
Cherries	100
Grapes	50
Nectarines	50
Plums	100
Strawberries	25

APPRAISAL

Captan was extensively reviewed in 1994 and recommendations were made for new and revised MRLs for a number of fruits, and for tomatoes. Information was made available to the present Meeting on GAP and supervised trials in the USA on apples, cherries, grapes, nectarines, pears, plums and strawberries. The residue data were evaluated together with the relevant data evaluated in 1994 to produce revised recommendations.

MRLs for captan are for residues defined as captan. Captan breaks down under some conditions to form THPI (1,2,3,6-tetrahydrophthalimide) and when a raw agricultural commodity is found to contain captan and THPI it is likely that some captan was converted to THPI during storage of the sample. In most cases the THPI residue is a negligible or minor part of the residue and its inclusion or exclusion makes little difference. The Meeting agreed that the definition of the residue for the estimation of STMR levels should also be captan alone.

Captan is registered for use on apples in the USA at 2.2-4.5 kg ai/ha with up to 36 kg ai/ha applied in a crop cycle, equivalent to 8 applications at the maximum rate. Harvest is permitted on the day of the final application. The decline of captan residues was measured in 7 trials on apples with sampling on at least 5 occasions after the final application. The median half-life of captan from the 7 trials was 11.9 days, which suggested that an increased number of applications would not influence the final residue levels because the contribution from applications more than 40-50 days before harvest would be negligible in comparison with that from the final application. A trial with only one application at the GAP rate was also included (captan residue 14 mg/kg on the day of application).

The residues from the US trials at GAP application rates (3.4-5.0 kg ai/ha) and PHI (0-1 days) but with 1-14 applications were 3.7, 4.0, 5.7, 6.1, 6.6, 14 and 16 mg/kg.

US GAP also permits a post-harvest spray or dip for apples at 0.15 kg ai/hl, which may be used in combination with the pre-harvest treatment. In 13 US trials reported in the 1994 evaluation where captan had been used before, after, or both before and after harvest, the captan residues were 0.86, 1.4, 1.5, 2.3, 3.3, 3.9, 4.0, 4.7, 4.9, 5.2, 5.5, 5.9 and 7.7 mg/kg.

Captan trials on apples in Argentina, Brazil, Canada, Japan and the UK were evaluated against the relevant GAP for these countries in 1994. The residues from 22 trials according to GAP were 0.005, 0.44, 0.68, 0.98, 1.0, 1.4, 2.5, 2.8, 2.9, 2.9, 3.5, 3.8, 4.1, 4.2, 4.2, 4.3, 4.4, 4.5, 4.5, 4.8, 7.2 and 13 mg/kg.

The residues in rank order (median underlined) from the total of 42 trials were 0.005, 0.44, 0.68, 0.86, 0.98, 1.0, 1.4, 1.4, 1.5, 2.3, 2.5, 2.8, 2.9, 2.9, 3.3, 3.5, 3.7, 3.8, 3.9, 4.0, <u>4.0</u>, <u>4.1</u>, 4.2, 4.2, 4.3, 4.4, 4.5, 4.5, 4.7, 4.8, 4.9, 5.2, 5.5, 5.7, 5.9, 6.1, 6.6, 7.2, 7.7, 13, 14 and 16 mg/kg.

The Meeting estimated a maximum residue level of 20 mg/kg for captan on apples to replace the 1994 recommendation of 10 mg/kg, and an STMR level of 4.05 mg/kg.

Information from US supervised trials on pears was made available but could not be evaluated because there was no corresponding GAP.

Captan may be applied in the USA at 2.2 kg ai/ha up to 7 times to cherries, which may be harvested on the day of the final application. It may also be used as a post-harvest spray or dip at a concentration of 0.15 kg ai/hl, and the two treatments may be used in combination. Details of 7 trials according to GAP were available to the Meeting. The captan residues were 2.4, 4.3, 5.5 14, 20, 20 and 21 mg/kg. Two trials where the application rate was 1.1 kg ai/ha (half the label rate) should also be included because residues were 16 and 17 mg/kg. In most of the trials there was no explicit description of the sample for analysis (e.g. whole fruit + stems).

Ten US trials on cherries reported in 1994 included pre-harvest, post-harvest and combined applications according to GAP. The captan residues were 7.3, 10, 11, 14, 14, 15, 19, 23, 25 and 35 mg/kg.

In summary, the captan residues in rank order (median underlined) from the 19 trials on cherries were 2.4, 4.3, 5.5, 7.3, 10, 11, 14, 14, 14, <u>15</u>, 16, 17, 19, 20, 20, 21, 23, 25 and 35 mg/kg.

The Meeting estimated a maximum residue level of 40 mg/kg for captan on cherries to replace the 1994 estimate of 20 mg/kg, and an STMR of 15 mg/kg.

Data from 11 US supervised trials on <u>nectarines</u> could not be evaluated because the trial conditions were not sufficiently close to GAP.

US GAP permits the use of captan on <u>plums</u> at 3.4 kg ai/ha with harvest on the day of the final application. The total application permitted per season is 30 kg ai/ha, which corresponds to 9 applications. Data from 2 US trials on plums were reported to the Meeting and the use pattern in one of them exactly complied with GAP while in the other the application rate was correct but there were 13 applications and the PHI was 2 days. The use pattern in the 3 trials reported in the 1994 monograph complied with US GAP. The captan residues in the 5 valid trials (median underlined) were 0.45, 0.60, <u>0.71</u>, 5.6 and 7.9 mg/kg.

The Meeting concluded that the results suggest that a higher limit than the present draft MRL of 5 mg/kg is required, but the database is limited. The Meeting agreed not to estimate a revised maximum residue level, but to await the periodic review of captan in 1998 when complete information on GAP and residues resulting from supervised trials should be available.

In the USA captan may be applied to <u>grapes</u> at 1.1-2.2 kg ai/ha with no more than 13 kg ai/ha used in a growing season, equivalent to 6 applications at the higher rate. Harvest is permitted on the day of the last application. The conditions in 9 US trials closely matched the maximum conditions of US GAP. Seven of the trials were reported to the present Meeting and 2 had been reported in 1994. The residues in the 9 trials were 1.3, 3.5, 3.7, 6.4, 7.2, 7.4, 8.4, 11 and 22 mg/kg.

Trials on grapes in Argentina, France, Germany and Japan were evaluated in 1994. The residue was 0.74 mg/kg in an Argentinian trial according to Argentinian GAP (1.3 kg ai/ha, 3 applications, 25 days PHI). A French trial and 12 German trials were evaluated against French GAP (10 applications of 3.5 kg ai/ha with a PHI of 33 days). Pre-harvest intervals of 28-38 days in these trials were accepted. The residues in the 13 trials were 1.4, 1.7, 1.7, 1.9, 2.8, 3.0, 3.6, 4.4, 6.5, 7.0, 8.3, 9.8 and 15 mg/kg. In Japan captan may be sprayed 5 times on grapes at a concentration of 0.10 kg ai/hl, with harvest 14 days after the final application. The residues on grapes from 6 Japanese trials complying with GAP were 3.2, 5.8, 6.1, 6.1, 12 and 14 mg/kg.

In summary, the residues in rank order (median underlined) from the 29 trials were 0.74, 1.3, 1.4, 1.7, 1.7, 1.9, 2.8, 3.0, 3.2, 3.5, 3.6, 3.7, 4.4, 5.8, 6.1, 6.1, 6.4, 6.5, 7.0, 7.2, 7.4, 8.3, 8.4, 9.8, 11, 12, 14, 15 and 22 mg/kg.

The Meeting estimated a maximum residue level of 25 mg/kg for captan on grapes to replace the current draft MRL of 20 mg/kg, and an STMR of 6.1 mg/kg.

US GAP permits application rates for captan on strawberries of 1.7-3.4 kg ai/ha and a PHI of 0 days, with a total application for the growing season of 27 kg ai/ha, equivalent to 8 applications at the highest rate. Six US and one Canadian trial according to the US application rate and PHI were reported to the Meeting. The number of applications varied from one to 11 but apparently the number had little effect on the residue levels. The residues in the 7 trials were 3.4, 3.9, 5.8, 6.4, 7.3, 13 and 27 mg/kg.

Trials in the USA, Canada, Chile and Hungary were recorded in the 1994 evaluations. In nine US trials complying with US GAP the residues were 1.0, 2.6, 3.9, 4.4, 5.2, 7.7, 12, 13 and 15 mg/kg. The residue was 3.0 mg/kg in a Canadian trial according to Canadian GAP (3.4 kg ai/ha, PHI 2 days). Chilean GAP allows 2 applications of 3.2 kg ai/ha and a PHI of 2 days. In trials at this rate but with 1 application and a 3-day PHI the residues were 3.8, 4.2 and 4.8 mg/kg. The residue in a Hungarian trial according to GAP (1.3 kg ai/ha, 3 applications, 10-day PHI) was 0.93 mg/kg.

In summary, captan residues in strawberries from the 21 trials (median underlined) were 0.93, 1.0, 2.6, 3.0, 3.4, 3.8, 3.9, 3.9, 4.2, 4.4, 4.8, 5.2, 5.8, 6.4, 7.3, 7.7, 12, 13, 13, 15 and 27 mg/kg.

The Meeting estimated a maximum residue level of 30 mg/kg for captan on strawberries to replace the 1994 estimate of 15 mg/kg, and an STMR of 4.8 mg/kg.

Information was provided to the Meeting on the fate of captan during the processing of apples and grapes.

Details of the processes for producing juice and pomace from apples were very limited. Heating and cooking are very influential on the fate of captan but no information on these operations was provided. Calculated processing factors for the production of juice, wet pomace and dry pomace from apples were 0.30, 0.48 and 0.064 respectively.

More detailed studies were provided to the 1994 JMPR, and that Meeting concluded that captan is not present in processed commodities such as apple sauce, canned apple slices, apple jelly or canned juice because it is destroyed by cooking and heating.

The supervised trials median residues for the processed commodities (STMR-Ps) calculated from the processing factors and the STMR for apples (4.05 mg/kg) were apple juice (unheated) 1.2 mg/kg, apple juice (heated) 0 mg/kg, apple sauce 0 mg/kg and dry apple pomace 0.26 mg/kg.

The Meeting also used the processing factor to estimate a maximum residue level for dry apple pomace of 2 mg/kg after rounding (maximum residue level in apples 20 × processing factor 0.064).

The processing factors for captan in the production of grape products were highly variable from one experiment to another, probably reflecting the sensitivity of captan to degradation under some heating conditions. The processing factors (mean and range) from grape processing studies supplied to the current Meeting and to the 1994 JMPR were grapes to juice 1.2 (range 0.23-4.9), grapes to wet pomace 0.94 (range 0.19-1.4), grapes to dry pomace 0.67 (range 0.12-1.7) and grapes to raisins 1.66 (range 0.11-4.8).

The STMR-Ps calculated from the processing factors and the STMR for grapes (6.1 mg/kg) were grape juice 7.3 mg/kg, dry grape pomace 4.1 mg/kg and raisins 10.4 mg/kg.

The Meeting also used the processing factor for raisins to estimate a maximum residue level for dried grapes of 50 mg/kg after rounding (maximum residue level in grapes 25 mg/kg × processing factor 1.66).

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 (for compliance with MRL and for estimation of dietary intake): captan.

Commodity		Recommended MRL, mg/kg		PHI, days	STMR, mg/kg	STMR-P, mg/kg
CCN	Name	New	Current			
FP 0226	Apple	20	10	0	4.05	
AB 0226	Apple pomace, dry	2				0.26
FS 0013	Cherries	40	20	0	15	
DF 0269	Dried grapes (currants, raisins and sultanas)	50				10.4
FB 0269	Grapes	25	20	0	6.1	
FB 0275	Strawberry	30	15	0	4.8	
	Apple juice (unheated)					1.2
	Apple juice (heated)					0
	Apple sauce					0
	Grape juice					7.3

REFERENCES

Smith, R.D. 1987. Captan: magnitude of residue crop field trials, grape. 056131-K (includes 86080, 86814, 86994, 86256, 86719, 86549, 86218). Chevron Chemical Company, USA. Unpublished.

Fujie, C.H. 1982. Determination of captan and THPI residues in crops (RM-1K-2). File 740.01/CAPTAN. Chevron Chemical Company, USA. Unpublished.

CARBOFURAN (096)

EXPLANATION

Carbofuran, a systemic acaricide, insecticide and nematicide, was first evaluated in 1967 and reviewed in 1979, 1991 and 1993. The Ad Hoc Working Group on Priorities of the CCPR in 1993 proposed carbofuran for re-evaluation, as the ADI was established in 1982 (ALINORM 93/24A para 251). It was scheduled for toxicological review in 1996 by the 1994 CCPR (ALINORM 95/24 Appendix VI) and for residue review in 1997 by the 1995 CCPR (ALINORM 95/24A, Appendix IV).

The toxicology of carbofuran was re-evaluated by the Joint Meeting in 1996. An ADI of 0-0.002 mg/kg bw was allocated on the basis of the NOAEL for erythrocyte acetylcholinesterase inhibition of 0.22 mg/kg bw per day in a four-week study in dogs and a safety factor of 100. The effect observed was reversible and acute. The previous ADI was 0-0.01 mg/kg bw.

Carbosulfan, the subject of a separate residue re-evaluation at the present Meeting, is metabolized to carbofuran and evaluations of carbofuran residues must account for carbofuran and its metabolites resulting from the use of carbosulfan according to GAP.

IDENTITY

ISO common name: carbofuran

Chemical name:

IUPAC: 2,3-dihydro-2,2-dimethylbenzofuran-7-yl methylcarbamate

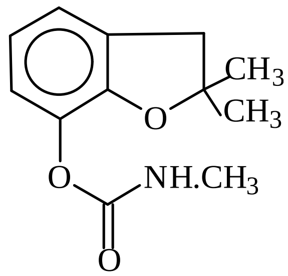
CA: 2,3-dihydro-2,2-dimethylbenzofuran-7-yl methylcarbamate

CAS No.: 1563-55-2

CIPAC No.: 276

Synonyms: Furadan; Curraterr; Yaltox; FMC 10242

Structural formula:



Molecular formula: $C_{12}H_{15}NO_3$

Molecular weight: 221.26

Physical and chemical properties

Pure active ingredient

Vapour pressure: 6 x 10-^{7} mm Hg at 25°C (Alvarez, 1989)

Melting point: 153-154°C (USA Standard, 1968)

Octanol/water partition coefficient:

log P_{ow} 1.3 at 20°C (Brandau, 1975)

Solubility:
g/100 g at 25°C:

acetone	15	(USA Standard, 1968)
acetonitrile	14	
benzene	4	
cyclohexanone	9	
dichloromethane	12	
dimethylformamide	27	
dimethylsulfoxide	25	
ethanol	4	
water	0.035 g/100 ml	(Alvarez, 1987)
xylene	<1	

Specific gravity: 1.18 at 20°C

Hydrolysis:

pH	Temperature, °C	Half-life, h
25	>20,000	(Alvarez, 1987; Dziedzic 1987)
3.1	35	>20,000
3.1	45	>20,000
6.2	25	≥7,000
6.2	35	1400
6.2	45	320
7.0	25	670
7.5	25	220
8.0	25	65
9.1	25	15
9.1	35	3.2
9.1	45	0.76
9.9	25	2.2
9.9	35	0.55
9.9	45	0.16

Photolysis:

Half-life 150 hours in pH 7.0 buffered aqueous solution (5 mg/l) at 25°C when subjected to 300-400 nm radiation with a power of 150 µw/cm^{2}.

Technical material

Purity: 98%

Melting range: 150-152°C

Stability: Stable under neutral or acid conditions. Unstable in alkaline media.

Formulations

Formulated products containing carbofuran are listed in Table 1.

Table 1. Formulations of carbofuran.

Product	Form.	Active ingredient(s)	% ai
Furadan 75 DB	DP	carbofuran	75
Furadan 85 DB	DP	carbofuran	85
Furadan 3G (Carbo 3G)	GR	carbofuran	3
Furadan 5G or 50G (Carbosip 5G)	GR	carbofuran	5
Furadan 10G	GR	carbofuran	10
Furadan 20F	SC	carbofuran	20
Furadan 35 FS	FS	carbofuran	35
Furadan 4F or 40 F	SC	carbofuran	4
Furadan 47F	SC	carbofuran	47
Furadan 300ST	ST	carbofuran	30
Furadan 310ST (Furazin 310 TS)	ST	carbofuran	31
Furadan 35 or 350	FS	carbofuran	35
Furadan 360	FS	carbofuran	36
Furadan 350SC	SC	carbofuran	35
Curraterr 10G	GR	carbofuran	10
Curraterr 5G	GR	carbofuran	5
Furadan Combi	ST	carbofuran + carbendazim + thiram	27 5 5
Yaltox	ST	carbofuran	

METABOLISM AND ENVIRONMENTAL FATE

Animal Metabolism

The metabolism of [^{14}C]carbofuran has been studied in rats, houseflies, laying hens and lactating goats (Table 2). The carbofuran was uniformly labelled in the phenyl ring in all studies except on houseflies, where [*carbonyl*-^{14}C]carbofuran was used. Both labels were used in the rat study.

Table 2: Animal metabolism studies on [^{14}C]carbofuran.

Subject	Treatment	References
Rats Houseflies	4 mg/kg bw, single oral 0.05 µg/fly, topical	Dorough, 1968
Lactating goats	25 ppm for 7 days[1]	Hoffman and Robinson, 1994a
Laying hens	25 ppm for 7 days[1]	Hoffman and Robinson, 1994b

[1]Doses were daily by capsule, equivalent to 25 ppm in feed

Rats. Rats of 200 g each were treated orally with either 0.4 mg per kg bw [*carbonyl*-^{14}C]carbofuran or 4.0 mg per kg bw *phenyl*-^{14}C]carbofuran in a single dose. The urine and faeces were collected and assayed for the total radioactivity. ^{14}C was collected from the rats given the carbonyl label. The cumulative percentages of the administered doses found in air, urine and faeces are shown in Table 3. About 86-90% of the radiolabelled carbofuran was eliminated by 32 hours after treatment. Additional elimination was minimal. About 45% of the radiolabelled carbofuran was eliminated by cleavage of the carbamoyl moiety.

Table 3. Cumulative percentage of administered ^{14}C in air, urine and faeces from orally-dosed rats.

Time after dosing (h)	Cumulative % of administered dose				
	[carbonyl ^{14}C]carbofuran			[phenyl ^{14}C]carbofuran	
	CO_2	Urine	Faeces	Urine	Faeces
2	5.6	2.7	0.0	5.9	0.3
6	31	25	0.8	21	0.5
24	43	37	1.9	72	2.3
32	45	38	2.6	88	2.4
48	45	38	3.8	89	2.5
72	45	38	4.4	91	3.3
96	45	38	4.4	92	3.3
120	45	38	4.4	92	3.3

Urine from the [*phenyl*-^{14}C]carbofuran-treated rats from the 2-24 hour periods was extracted with an organic solvent. Less than 5% of the radioactivity was organosoluble. The major component identified by TLC in the 24-hour sample was 2,3-dihydro-2,2-dimethyl-3-hydroxybenzofuran-7-yl-hydroxymethylcarbamate, 1.1% of the total radioactivity in the urine. Pooled urine collected for 72 hours from the [*phenyl*-^{14}C]carbofuran-treated rats was acidified to 0.5 N, boiled for 10 minutes and extracted with chloroform. About 95% of the water-soluble residue was converted to chloroform-soluble material. The compounds tentatively identified by TLC, with their percentages of the total radioactivity in the pooled sample, were 2,3-dihydro-2,2-dimethyl-3-hydroxybenzofuran-7-yl-hydroxymethylcarbamate (3.8%), 3-hydroxy-carbofuran (14%), 2,3-dihydro-2,2-dimethylbenzofuran-3,7-diol (1.4%), 2,3-dihydro-2,2-dimethyl-3-oxobenzofuran-7-ol (48%) and 2,3-dihyrdro-2,2-dimethylbenzofuran-7-ol (20%). Control experiments showed that carbofuran, 2,3-

dihyrdro-2,2-dimethylbenzofuran-7-ol, 2,3-dihydro-2,2-dimethylbenzofuran-3,7-diol and 2,3-dihydro-2,2-dimethyl-3-oxobenzofuran-7-ol were not altered by the treatment.

Houseflies (6 days old) were treated topically with [*carbonyl*-^{14}C]carbofuran at 0.05 µg per fly and analysed in groups of 100 one hour after application. Surface radioactivity was removed with an acetone rinse. Internal radioactivity was extracted by homogenizing with acetone/water (1:1) and partitioning with chloroform. The vials were rinsed with acetone/water to collect excreted ^{14}C and the wash was partitioned with chloroform. Water-soluble fractions were hydrolysed with acid and extracted with chloroform. The organic extracts were analysed by TLC, with the results given in Table 4. Identities were not confirmed.

Table 4. Tentative identification of the radiolabelled residue from the topical application of [*carbonyl*-^{14}C]carbofuran to houseflies.

Compound	% of applied dose		
	Surface residue	Internal residue	Excretion
Carbofuran	23	12	7.1
3-hydroxy-carbofuran	0.3	5.7	0.4
3-hydroxy-carbofuran conjugated[1]	0	11	4.3
2,3-dihydro-2,2-dimethyl-3-hydroxybenzofuran-7-yl hydroxymethylcarbamate	0.2	0.7	0.3
2,3-dihydro-2,2-dimethyl-3-hydroxybenzofuran-7-yl hydroxymethylcarbamate conjugated[1]	0	2.1	0.1
3-oxo-carbofuran	0.2	1.2	2.0
2,3-dihydro-2,2-dimethylbenzofuran-7-yl-N-hydroxymethylcarbamate conjugated[1]	0	1.8	0.2
Total[2]	24	34	14

[1]Released by mild acid hydrolysis
[2]An unknown (free and conjugated) accounted for 9% of the applied dose. Also, part of the dose may have been lost as $^{14}CO_2$ from hydrolysis of the carbamate group

Fifteen laying hens (1.34-1.68 kg, randomly divided into groups of 5) each received a capsule containing 3 mg of [^{14}C]carbofuran on each of 7 consecutive days. Eggs were collected on each day, separated into yolk and whites and pooled by group. Excreta were collected daily and pooled by group. Within 22 hours of the final dose, the hens were killed and samples of breast, thigh, fat with skin, liver and kidneys were collected from each hen and pooled by group.

Most of the administered dose was eliminated in the excreta, with the cumulative percentage of it ranging from an average of 71% on day 1 to 83% on day 7. The distribution of the radiocarbon in the eggs, excreta and tissues is shown in Table 5.

Table 5. Total radioactive residues as cumulative percentage of administered dose and as carbofuran equivalents.[1]

Sample	Day	% of applied dose	Total ^{14}C as carbofuran, mg/kg
Excreta	1	70.6	
	3	75.2	
	7	82.8	
Egg white	1	0.18	0.032
	3	0.21	0.069
	7	0.27	0.059
Egg yolk	1	0.07	0.027
	3	0.09	0.078

Sample	Day	% of applied dose	Total ^{14}C as carbofuran, mg/kg
	7	0.21	0.141
Liver	7	0.11	0.137
Kidneys	7	0.01	0.034
Breast muscle	7	0.02	<0.010
Thigh muscle	7	<0.01	<0.010
Skin and fat	7	<0.01	<0.010
Total recovery	7	83.4	

[1] Average of three groups.

The tissue samples containing >0.01 mg/kg total radioactive residue (TRR) were extracted sequentially with acetonitrile and methanol/water. Egg white was extracted with acetonitrile and egg yolk with a mixture of acetonitrile and hexane. The extractions removed the following percentages of the TRR: egg yolk 91%; egg white 91%; liver 16%; kidneys 41%. The post-extraction solids from the liver and kidneys were treated sequentially with protease, acid and base. Protease released 25% of the TRR from the liver and 19% from the kidneys. Acid and base treatments released an additional 48% from the liver and 28% from the kidneys.

The radiolabelled residues released by solvent extraction and enzyme, acid and base hydrolyses were investigated by normal-phase TLC and reverse-phase HPLC. The characterizations and identifications are shown in Table 6. The structures of the metabolites are given in Figure 1.

Table 6. Characterization and identification of the total radiolabelled residue from the administration of [^{14}C]carbofuran to hens.

Metabolite or characterization	Liver		Kidneys		Egg white		Egg yolk	
	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
3-hydroxy-carbofuran	--	-	-	-	-	-	12	0.019
2,3-dihydro-2,2-dimethylbenzofuran-7-ol	5.7[1]	0.008	4.9[1]	0.001	-	-	16	0.026
2,3-dihydro-2,2-dimethylbenzofuran-3,7-diol	-	-	-	-	-	-	39	0.062
2,3-dihydro-2,2-dimethyl-3-oxobenzofuran-7-ol	-	-	-	-	-	-	8.5	0.014
Phenolic conjugates	-	-	-	-	90	0.060	-	-
Enzyme digestion aqueous fraction	7.3	0.010	4.6	0.002	-	-	4.6	0.007
Mild acid hydrolysis aqueous fraction	3.1	0.004	5.8	0.002	-	-	-	-
Strong acid hydrolysis aqueous fraction	12	0.016	8.2	0.003	-	-	-	-
Mild base hydrolysis aqueous fraction	4.0	0.005	3.6	0.001	-	-	-	-
Polar residues from initial extractions	12	0.016	8.2	0.003	-	-	-	-

[1] conjugated, released by enzyme treatment.

The total radiolabelled residues in the muscle and fat with skin were negligible and the residues in the kidneys, liver and eggs ranged from 0.03 to 0.2 mg/kg. The parent compound was not detected. The metabolic pathway includes oxidation to 3-hydroxy- and 3-keto-carbofuran and hydroxylation to phenolic metabolites. See Figure 2.

[14]C]carbofuran, uniformly labelled in the phenyl ring, was administered orally to 2 goats for 7 consecutive days. The dose was equivalent to 25 mg/kg carbofuran in the feed. Urine, faeces and milk were collected twice daily and pooled. The goats were slaughtered within 24 hours of the final dose and samples of muscle (leg and loin), liver, kidney, omental fat and blood were taken. The distribution of the ^{14}C is shown in Table 7.

Table 7. Total radioactive residue as cumulative percentage of administered dose and as carbofuran equivalents.[1]

Sample	Day	% of applied dose	TRR as carbofuran, mg/kg
Milk	1	0.32	0.010
	3	0.29	0.14
	7	0.30	0.098
Urine	1	95	
	3	90	
	7	88	
Faeces	1	4.1	
	3	5.1	
	7	5.0	
Liver	7	0.025	0.11
Kidneys	7	<0.01	0.18
Leg muscle	7	<0.01	<0.01
Loin muscle	7	<0.01	0.01[2]
Omental fat	7	<0.01	<0.01
Total recovery	7	95	

[1]Average of 2 goats
[2]Goat B only. Goat A was <0.01 mg/kg

Milk (day 5 pm, containing 0.32 mg/kg carbofuran equivalents) was extracted with acetone. Muscle tissue (from goat B), liver and kidneys were sequentially extracted with chloroform and methanol/water. The percentages of the total radioactive residue extracted were milk 99%; muscle 30%; liver 27%; kidney 20%. The post-extraction liver and kidney samples were sequentially treated with protease, mild acid extraction and strong acid hydrolysis. Protease released 41% of the total radioactive residue from the liver and 49% from the kidneys. The mild acid extraction released 12% from the liver and kidneys.

The released radioactive residues were characterized and the components identified by normal-phase TLC and reverse-phase HPLC, with the results shown in Table 8. The structures of the metabolites are given in Figure 1.

Table 8. Characterization and identification of total radioactive residue from the administration of [^{14}C]carbofuran to lactating goats.

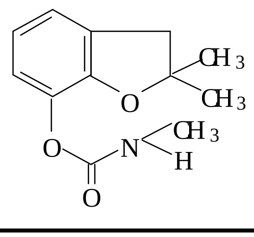
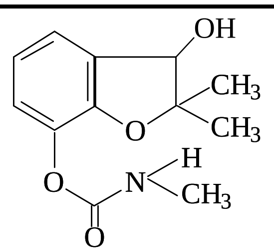
Metabolite or characterization	^{14}C, % of the TRR and mg/kg as carbofuran							
	Milk		Muscle		Liver		Kidney	
	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
carbofuran	0.41	0.001	-	-	-	-	-	-
3-hydroxy-carbofuran	10	0.032	-	-	4.0[3]	0.005	11[6]	0.029
2,3-dihydro-2,2-dimethylbenzofuran-7-ol	15	0.048	-	-	2.4[4]	0.003	-	-
2,3-dihydro-2,2-dimethylbenzofuran-3,7-diol	6.8[1]	0.021	-	-	12[5]	0.017	16[7]	0.042
2,3-dihydro-2,2-dimethyl-3-	32[2]	0.10	-	-	-	-	-	-

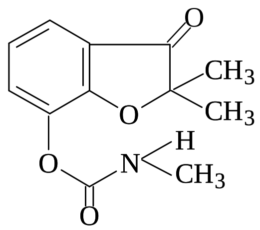
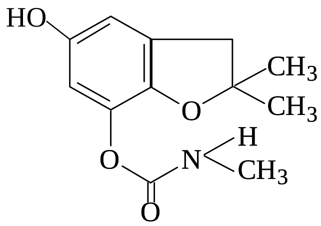
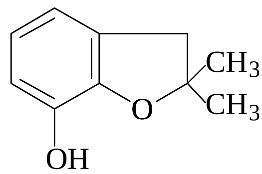
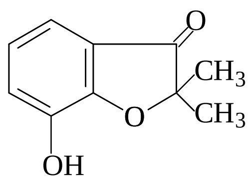
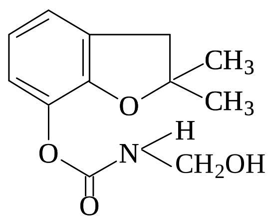
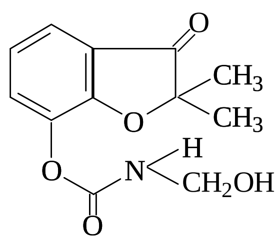
Metabolite or characterization	^{14}C, % of the TRR and mg/kg as carbofuran							
	Milk		Muscle		Liver		Kidney	
	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
oxobenzofuran-7-ol								
Aqueous fraction from initial extractions	6.3	0.020	28	0.003	5.0	0.007	3.5	0.009
Aqueous fraction from enzyme digestion	-	-	-	-	16	0.022	13	0.035
Aqueous fraction from mild acid hydrolysis	-	-	-	-	4.5	0.007	5.1	0.014
Aqueous fraction from strong acid hydrolysis	-	-	-	-	6.3	0.009	6.7	0.018
Polar residues (in initial extracts)	22	0.070	-	-	6.9	0.010	17	0.044

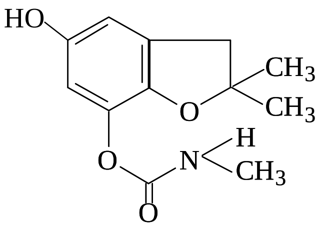
[1]Including 2% conjugated, released by sulfatase treatment
[2]Including 29% conjugated, released by sulfatase treatment
[3]Including 2.2% conjugated, released by protease treatment
[4]Conjugated, released by protease treatment
[5]Including 11% conjugated, released by protease treatment
[6]Including 8.2% conjugated, released by protease treatment
[7]Conjugated, released by protease treatment

The total radioactive residues in the tissues and fat were negligible (≤001 mg/kg) after the dietary equivalent of 25 mg/kg for 7 days. Residues in the kidneys, liver and milk ranged from 0.09 to 0.39 mg/kg. The identified metabolites are the same as those found in poultry, but the parent compound was also detected in milk. Figure 2 shows the probable metabolic pathways in poultry and ruminants. Two paths are indicated, in which oxidation at C-3 is followed or preceded by hydrolysis of the carbamate linkage. Oxidation of the carbamate methyl was not observed in goats or hens.

Figure 1. Names and chemical structures of carbofuran and its potential metabolites.

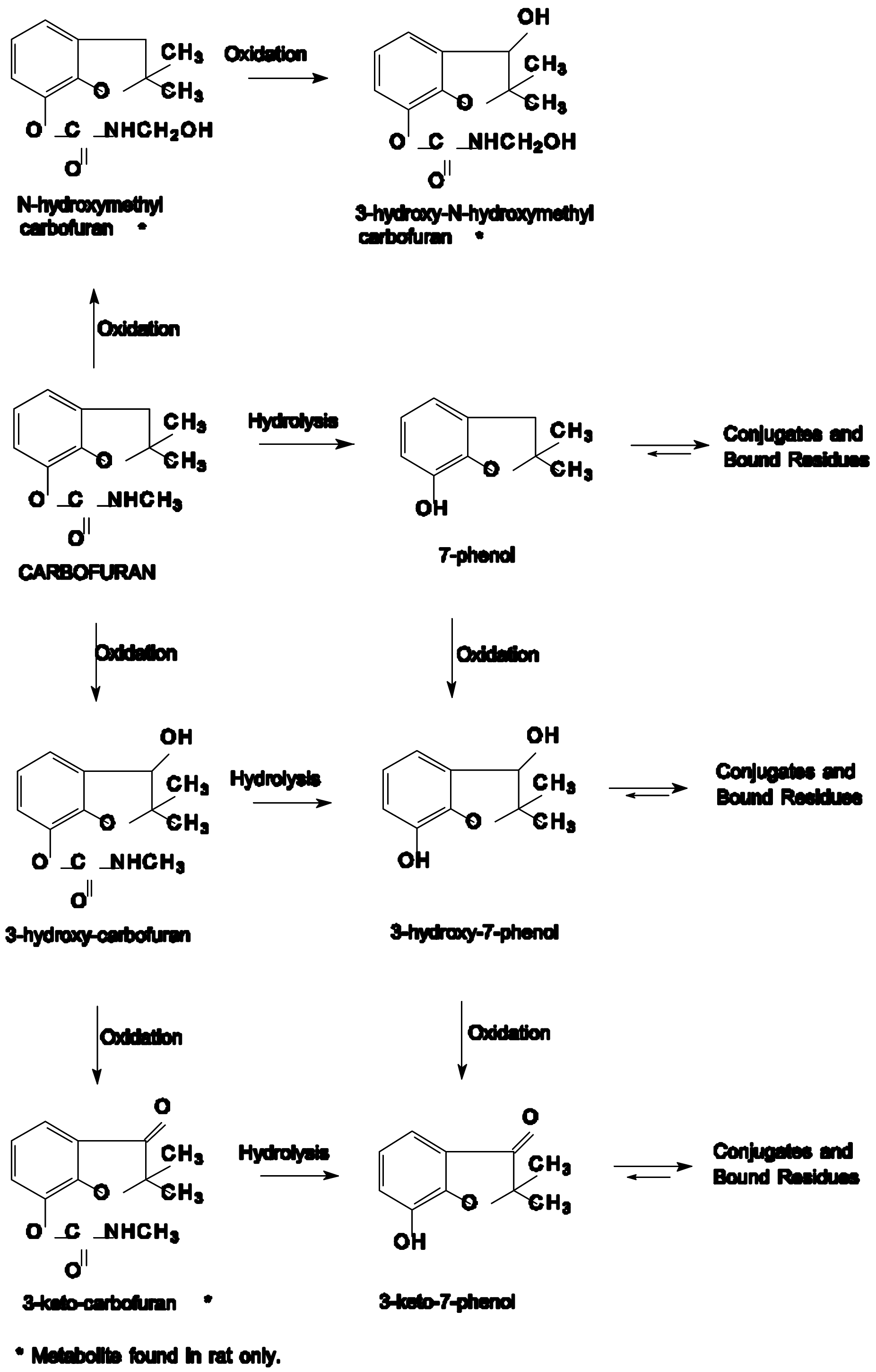
{PRIVATE }Common or derived[1] name Abbreviation used in Tables FMC number	Chemical name	Structure
Carbofuran CF FMC 10242	2,3-dihydro-2,2-dimethylbenzofuran-7-yl methylcarbamate	
3-hydroxy-carbofuran 3-OH-CF	2,3-dihydro-3-hydroxy-2,2-dimethylbenzofuran-7-yl methylcarbamate	

{PRIVATE }Common or derived[1] name Abbreviation used in Tables FMC number	Chemical name	Structure
3-keto-carbofuran 3-K-CF	2,3-dihydro-2,2-dimethyl-3-oxobenzofuran-7-yl methylcarbamate	
5-hydroxy-carbofuran 5-OH-CF FMC 27552	2,3-dihydro-5-hydroxy-2,2-dimethylbenzofuran-7-yl methylcarbamate	
7-phenol 7-P FMC 10272	2,3-dihydro-2,2-dimethylbenzofuran-7-ol	
3-keto-7-phenol 3-K-7-P FMC 16490	2,3-dihydro-2,2-dimethyl-3-oxobenzofuran-7-ol	
N-hydroxymethyl carbofuran N-CH_2OH CF FMC 53858	2,3-dihydro-2,2-dimethylbenzofuran-7-yl hydroxymethylcarbamate	
3-keto-N-hydroxymethyl carbofuran 3-K-N-CH_2OH CF FMC 53895	2,3-dihydro-2,2-dimethyl-3-oxobenzofuran-7-yl hydroxymethylcarbamate	

{PRIVATE }Common or derived[1] name Abbreviation used in Tables FMC number	Chemical name	Structure
{PRIVATE } 5-hydroxy-carbofuran 5-OH-CF FMC 27552	2,3-dihydro-2,2-dimethyl-5-hydroxybenzofuran-7-yl N-methylcarbamate	

[1]Names such as 3-hydroxy-carbofuran, 7-phenol etc., derived from the common name carbofuran

Figure 2. Proposed biotransformation pathways of carbofuran in poultry and ruminants.



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Plant metabolism

Metabolism studies were reported for potatoes, soya beans and maize (field corn). Supplementary information was submitted on the metabolism of radiolabelled carbofuran in several rotational crops.

Potatoes. Greenhouse-grown potato plants, height about 20 cm, were treated with [*phenyl*-^{14}C]carbofuran (2.65 mCi/mmol, 26,548 dpm/µg) in a single directed application to the soil surface at 7.4 kg ai/ha (Chang, 1994). The [^{14}C]carbofuran was formulated as a 0.5 kg ai/l flowable formulation (4F) and was diluted with water before application. Immature vines were sampled after 56 days and mature tubers harvested after 104 days. The total radioactive residues were 30.5 mg/kg as carbofuran in the vines and 0.80 mg/kg in the potatoes. Extraction of immature vine and mature potatoes with methanol/water followed by methylene chloride partition of the acidified and concentrated extract yielded 6.0% of the foliage and 22% of the tuber TRR. The aqueous from the methylene chloride partition, containing 87% of the foliage and 61% of the tuber TRR, was sequentially incubated with β-glucosidase (7.9% of the tuber and 51% of the foliage ^{14}C was organosoluble) and hydrolysed with 0.25 N HCl (32% of the tuber and 14% of the foliage TRR was organosoluble) and 2 N HCl (9.4% of the tuber and 13% of the foliage TRR was organosoluble). The parent compound and metabolites were identified or characterized by reverse-phase HPLC and normal-phase TLC. Tentative identifications were confirmed by GC-MS, both EI and CI. The major metabolite identified in the mature tubers was the 7-phenol (45% of the TRR) and in the foliage 5-hydroxy-carbofuran (34%). The results are shown in Table 9.

Table 9. Identification or characterization of radiolabelled residues in or on potatoes from the application of [*phenyl*-^{14}C]carbofuran to soil at 7.4 kg ai/ha after plant emergence.

Compound	Mature tuber (104-day PHI)		Immature foliage, 56-day PHI	
	%of TRR	mg/kg	%of TRR	mg/kg
Carbofuran	-	-	3.5%	1.071
3-OH-carbofuran	2.9%	0.023	22.6%	6.906
3-keto-carbofuran	-	-	1.1%	0.324
7-phenol	45.3%	0.361	6.7%	2.044
3-OH-7-phenol	13.4%	0.107	5.4%	1.658
3-keto-7-phenol	6.6%	0.052	9.4%	2.858
5-OH-carbofuran	-	-	34.4%	10.522
Total Identified[1]	68.2% (22% unconjugated)	0.543	83.1% (4.6% unconjugated)	25.383
Other	3.7%	0.029	2.6%	0.807
Polar residues	23.3%	0.185	11.0%	3.354
Unextractable	4.9%	0.039	3.3%	1.002
Total Residues[1]	100.0%	0.80	100.1%	30

[1]Results are normalized for recovery (91-101%).

Soya beans. Sandy loam soil in two 61 x 120 x 61 cm boxes was treated with carbofuran uniformly labelled with ^{14}C in the phenyl ring at 5.5 kg ai/ha in Watsonville, CA, USA. The treatment solution also contained carbofuran labelled with ^{13}C on one of the two gem-dimethyl groups and was prepared as a 0.5 kg ai/l flowable formulation (4F) in acetone/water. As applied the solution had a specific activity of 8.03 mCi/mmol. The test material was applied in a 15 cm band to a 1.3 cm deep furrow. Immediately after the application, soya bean seeds were sown in a single row down the middle of the furrow and covered with untreated soil. The soya beans were grown outdoors and samples of forage at 45 days PHI, beans at 139 days and hay at 139 days were collected.

Samples were assayed for the total radioactive carbon by oxidation and liquid scintillation counting. The forage contained 63 mg/kg carbofuran, the beans 0.32 mg/kg and the hay 36 mg/kg. Samples were then extracted with methanol/water (4:1 v/v) and subsamples of the extracts were concentrated and refluxed for one hour with 0.25 N HCl. The product mixtures were extracted with methylene chloride and the residual solids sequentially hydrolysed with 0.25 N HCl (60°C), cellulase, β-glucosidase, amyloglucosidase, pectinase, protease, 6N HCl (60°C) and 2N NaOH (65°C). The solid residues from the hay samples after solvent extraction were solubilized with dioxane/water (3/1 v/v) to release lignin (85°C, 48 hrs). After each hydrolysis the aqueous product solutions were adjusted to pH 2 and extracted with acetonitrile to recover organosoluble residues. The distribution and characterization of the radiolabelled residues are shown in Table 10.

The methanol/water and acid-refluxed methanol/water extracts were analysed by HPLC (reverse-phase) and fractions were collected for radioanalysis. Confirmation was by normal-phase (silica gel) TLC. The main metabolites were identified by GC-MS in both CI and EI modes. Unknown compounds separated by TLC or HPLC were investigated by HPLC-MS. The compounds identified are shown in Table 11.

Table 10. Distribution of the ^{14}C in hydrolysates and extracts of soya bean forage, beans and hay.

Sample	Fraction	[^{14}C]carbofuran equivalents, mg/kg	% of TRR
Forage (63 mg/kg)	Methanol/water extract	50	80
	0.25 N HCl treatment of PES[1], organosoluble	1.4	2.3
	Cellulase of PES, organosoluble	0.11	0.18
	B-glucosidase of PES, organosoluble	0.10	0.16
	Amyloglucosidase of PES, organosoluble	0.24	0.38
	Pectinase of PES, organosoluble	0.14	0.22
	Protease of PES, organosoluble	0.84	1.3
	6.0 N HCl treatment of PES, organosoluble	0.82	1.3
	2.0 N NaOH of PES, organosoluble	4.1	6.5
	Final Residual Solid	4.3	6.9
	TOTAL	62	99
Beans (0.32 mg/kg)	Methanol/water extract	0.19	59
	0.25 N HCl treatment of PES[1], organosoluble	0.030	9.3
	Cellulase of PES, organosoluble	0.011	3.6
	B-glucosidase of PES, organosoluble	0.003	0.92
	Amyloglucosidase of PES, organosoluble	0.006	1.8
	Pectinase of PES, organosoluble	0.014	4.3
	Protease of PES, organosoluble	0.022	6.9
	6.0 N HCl treatment of PES, organosoluble	0.013	4.0
	2.0 N NaOH of PES, organosoluble	0.019	5.9
	Final Residual Solid	0.019	5.9
	TOTAL	0.33	102
Hay [2](36 mg/kg)	Methanol/water extract	13	35
	0.25 N HCl treatment of PES organosoluble	5.4	15
	Cellulase of PES organosoluble	0.41	1.1
	B-glucosidase of PES organosoluble	0.26	0.72
	Amyloglucosidase of PES organosoluble	0.23	0.65

Sample	Fraction	[^{14}C]carbofuran equivalents, mg/kg	% of TRR
	Pectinase of PES organosoluble	0.13	0.36
	Protease of PES Organosoluble	0.17	0.48
	6.0 N HCl treatment of PES organosoluble	0.35	0.97
	2.0 N NaOH of PES organosoluble	0.40	1.1
	Dioxane (lignin release) of PES	2.1	5.9
	Final Residual Solid (before lignin release)	15	43
	TOTAL	35	98

[1]Post-extraction
[2] Moisture content of the hay was not determined: figures refer to undried hay

Table 11. Identification of carbofuran and metabolites in the radiolabelled residue isolated from methanol/water extracts of soya bean seed, forage and hay.

Compound	^{14}C, % of TRR and mg/kg as carbofuran											
	Extract		Acid-refluxed extract		Extract		Acid-refluxed extract		Extract		Acid-refluxed extract	
	% TRY	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
carbofuran	11.6	7.3	11.4	7.2	-	-	0.42	0.001	0.30	0.11	0.62	0.22
3-keto carbofuran	1.7	1.1	1.6	1.0	-	-	5.3	0.02	0.41	0.15	-	-
3-hydroxy-carbofuran	10.6	6.6	28	18	0.56	0.002	1.5	0.005	3.2	0.50	7.8	2.8
7-phenol	-	-	1.4	0.90	0.38	0.001	4.0	0.013	0.67	0.24	0.72	0.26
3-keto-7-phenol	1.6	1.0	13	8.1	0.71	0.002	9.2	0.030	4.3	1.6	9.8	3.5
O-glucoside conjugate of 3-hydroxy or 3-keto-7-phenol[1]	16	9.9	3.4	2.1	11	0.036	-	-	3.6	1.3	-	-
2-hydroxymethyl-3-keto carbofuran[2]	-	-	3.6	2.2	-	-	0.93	0.03	-	-	0.84	0.30
TOTAL identified	42		62		13		21		12		20	

[1]Identification by LC-MS. No comparison with reference standard
[2]Identification by GC-MS (EI and CI). No comparison with reference standard

Maize. A 1.5 x 1.5 m plot of tilled Crosby Loam soil in Ohio was treated with carbofuran uniformly labelled with ^{14}C in the phenyl ring at a rate of 8.3 kg ai/ha treated area, equivalent to 3.0 kg ai/ha broadcast (Curry, 1994). The radiolabelled material was isotopically diluted with [^{13}C]carbofuran labelled in one of the gem-dimethyl groups and with unlabelled carbofuran to a specific activity of 2.65 mCi/mmol or 26548 dpm/µg. The carbofuran was prepared as a 0.5 kg ai/l flowable formulation (4F) and was mixed with water before application. The test material was sprayed in a 15 cm band on the soil and incorporated to a depth of about 5 cm before planting maize seed (Pioneer Hybrid 3394).

Maize samples were taken at three growth stages: forage (immature stage, 47 days PHI), silage (reproductive stage, 99 days PHI) and stover and grain (mature stage, kernels without cob and husk, 158 days PHI). The samples were assayed for total ^{14}C by combustion and liquid scintillation counting. Each sample was extracted with methanol/water (1:1 v/v), and the extracts acidified to pH 1 and partitioned with methylene chloride/ether (3:1 v/v). The aqueous fractions from the methylene chloride/ether partitions of the forage and silage samples were divided into two equal portions: one was treated with ß-glucosidase and the other was acidified to 0.25 N, refluxed for one hour, and extracted with methylene chloride/ether. The aqueous layer from this extract of the silage samples was acidified to 1 N, refluxed for one hour, and extracted with methylene chloride/ether.

The post-extraction solids (PES) from the initial methanol/water extractions were refluxed with 0.25 N HC1 for one hour. The hydrolysate from the grain was tested to determine the presence of reducing sugars with Benedict's solution and by osazone formation. Both tests indicated reducing sugars. The residue after acid hydrolysis was treated with a surfactant, sodium dodecyl sulfate. The distribution of radioactivity in the various fractions, as determined by liquid scintillation counting, is shown in Table 12.

Table 12. Distribution of the radiolabelled residue in the extracts and hydrolysates of maize grain, forage, silage and stover (fodder) from the pre-plant application of [^{14}C]carbofuran

Fraction	Grain (0.023 mg/kg)		Forage (0.81 mg/kg)		Stover (0.075 mg/kg)		Silage (0.14 mg/kg)	
	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
Methylene chloride/ether (non-conjugates)	5.8[1]	0.001	42	0.34	4.4	0.003	4.6	0.006
Acid-released (0.1 n), methylene chloride/ether (aglycones)	-	-	32	026	-	-	20	0.028
Glucosidase-released (aglycones)	-	-	19	0.15	-	-	23	0.032
Residual acid aqueous	-	-	8.1	0.066	22	0.016	31[2]	0.036
Acid-released from PES	48[3]	0.011	3.5 (1.0 organo-soluble)	0.028	13 (4.2% organo-soluble)	0.010	9.9 (2.8% organo-soluble)	0.014
Surfactant-released from PES	-	-	1.6	0.013	4.7	0.004	5.1	0.007
Total released residue	48		87		44		71	

[1]Methanol/water extract
[2]1 N HCl treatment of the residual 0.25 N aqueous fraction generated an additional 7.9% of the TRR (0.011mg/kg) of organosoluble residue
[3] <1% partitioned into methylene chloride.

The organosoluble fractions from the forage and silage, i.e. the methylene chloride/ether extracts of the acidified methanol/water extract and of the 0.25 N HCl hydrolysate, were analysed by HPLC, TLC and GC-MS. The methylene chloride/ether extract of the 1 N HCl hydrolysate of silage was also analysed. Because of the relatively low levels of radiolabelled residue, the extracts from the grain and stover were not analysed. The identified compounds are shown in Table 13.

Table 13. Carbofuran and its metabolites in organosoluble extracts of maize silage and forage.

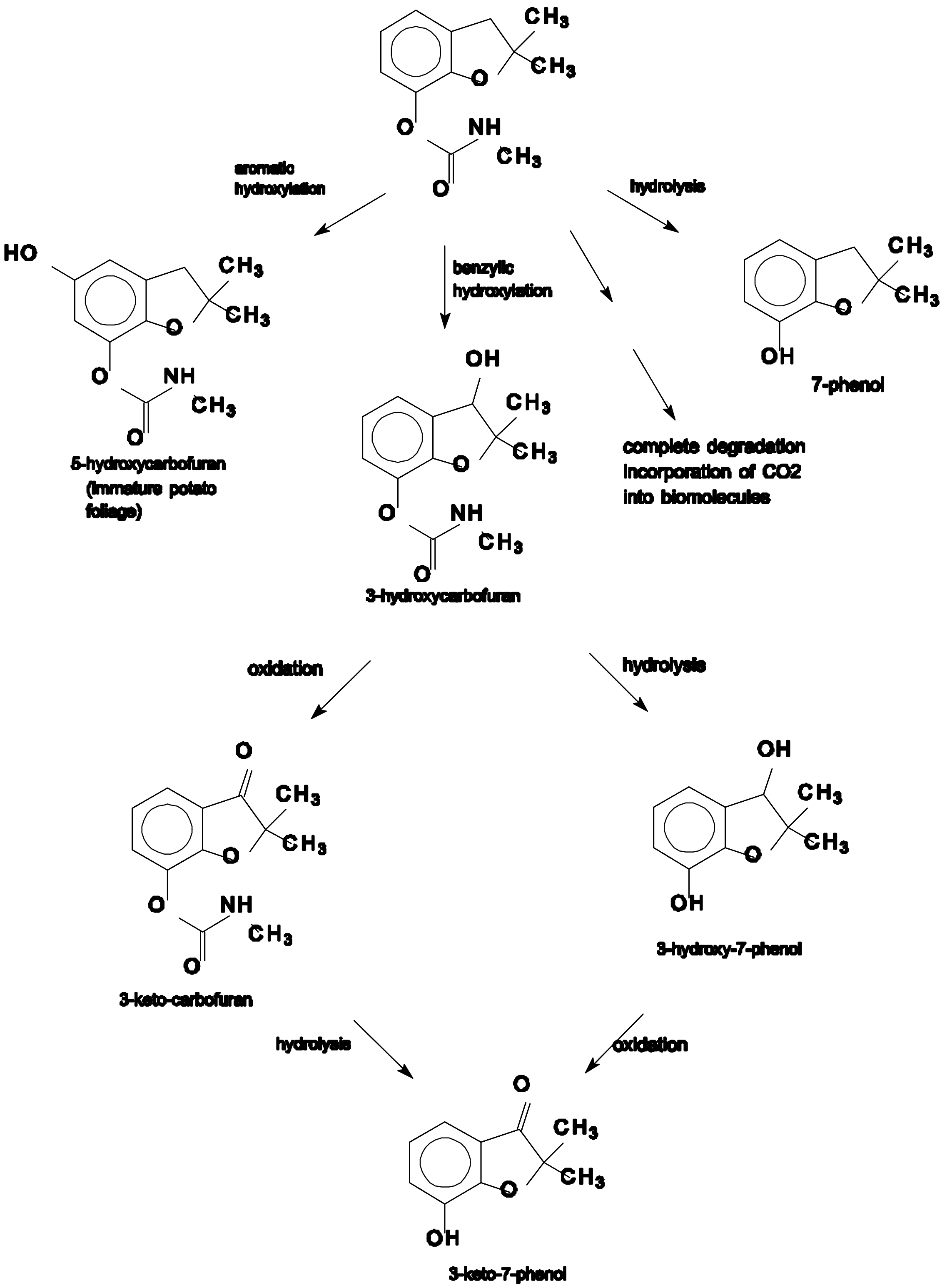
Compound	Forage		Silage	
	% of TRR	mg/kg as carbofuran	% of TRR	mg/kg as carbofuran
carbofuran	14	0.11	0.18	<0.001
carbofuran aglycone	2.4	0.019	2.1	0.003
3-keto-carbofuran	1.6	0.013	-	-
3-keto-carbofuran aglycone	0.28	0.003	0.91	0.001
3-hydroxy-carbofuran	13	0.11	1.3	0.002
3-hydroxy-carbofuran aglycone	9.7	0.078	7.9	0.011
7-phenol	0.47	0.004	0.088	<0.001

Compound	Forage		Silage	
	% of TRR	mg/kg as carbofuran	% of TRR	mg/kg as carbofuran
7-phenol aglycone	7.5	0.060	2.8	<0.001
3-keto-7-phenol	4.8	0.039	1.4	0.002
3-keto-7-phenol aglycone	5.6	0.045	2.4	0.003
3-hydroxy-7-phenol	2.4	0.020	0.88	0.001
3-hydroxy-7-phenol aglycone	3.6	0.029	2.3	0.003
Total	65	0.53	22	0.026

The major components of the radiolabelled residue identified in the forage were carbofuran and 3-hydroxy-carbofuran, free and conjugated and in the silage 3-hydroxy-carbofuran, free and conjugated. The amount of radioactivity that could not extracted with solvent or released by mild acid hydrolysis increased with the PHI, suggesting incorporation of the radiolabel into plant constituents.

The metabolites in the three crops (maize, potatoes and soya beans) are similar and are consistent with metabolism by hydroxylation and oxidation at C-3 and hydrolysis of the carbamate linkage (C-7). Aromatic hydroxylation was seen only in immature potato foliage. The proposed metabolic pathways are shown in Figure 3.

Figure 3. Proposed metabolic pathways of carbofuran in plants.



Environmental fate in soil

The rate and degree of the aerobic degradation of [^{14}C]carbofuran and its metabolites in acid and alkaline soils were determined in a study conducted in accordance with US EPA Guidelines (Saxena *et al*., 1994c). An acidic sandy loam soil (pH 5.7) was collected in Georgia and a portion was made alkaline (pH 7.7) by the addition of lime. The limed soil was incubated for about 2 months at approximately 25°C until the soil pH and microbial population had reached equilibrium before adding [14C]carbofuran uniformly labelled in the phenyl ring.

The test system consisted of approximately 50 g of oven-dried soil in a 250-ml flask. The soil samples were fortified with [14C]carbofuran at a nominal concentration of 3 mg/kg (equivalent to 6.7 kg ai/ha) and incubated at 25 ± 1°C under aerobic conditions in darkness for 365 days. The apparatus included ethylene glycol to trap organic volatiles and sodium hydroxide to trap CO_2.

Duplicate samples were analysed on days 0, 1, 3, 7, 14, 30, 62, 92, 122, 181, 273 and 365, and a third sample was taken at each interval to measure the pH and microbial population. The solutions in the traps were changed and the soil moisture was adjusted periodically. The samples were analysed immediately after collection: the population of aerobic bacteria and the pH were determined, the radioactivity in the traps was counted by LSC, the soils were extracted and analysed by HPLC and the extracted soil was combusted to measure the 14C. Selected extracts were also analysed by TLC to confirm the identity of [14C]carbofuran. Mass spectrometry was used to confirm the identities of degradation products which accounted for >10% of the TRR. More than 90% of the applied radioactivity was accounted for in all the samples. A summary of the results is given in Tables 14 and 15.

Table 14. Aerobic degradation of carbofuran in acidic soil.

Day	Mean % of applied 14C as							
	carbofuran	3-OH-CF	3-K-7-P	3-K-CF	7-phenol	Volatiles	Soil-bound	Total
0	97.49	0.06	0.16	0.18	0.04	ND	0.40	98.32
1	96.10	0.19	0.31	0.31	ND	0.01	2.91	99.82
3	95.06	0.13	ND	0.29	ND	0.03	4.64	100.15
7	92.68	0.32	0.03	0.70	ND	0.05	5.94	99.71
14	88.89	0.15	0.13	2.10	ND	0.09	8.35	99.71
30	84.30	0.56	0.02	2.08	0.02	0.16	10.96	98.10
62	82.72	ND	ND	2.60	ND	0.29	13.01	98.62
92	74.98	0.56	ND	6.36	ND	0.55	15.00	97.45
122	69.86	ND	ND	7.13	ND	0.94	20.89	98.81
181	58.29	ND	ND	12.41	ND	2.52	24.59	97.80
273	53.85	0.55	ND	11.41	ND	3.98	29.28	99.05
365	43.58	0.63	1.91	11.14	0.33	4.96	35.41	97.95

Table 15. Aerobic degradation of carbofuran in alkaline soil.

Day	Mean % or applied 14C as							
	carbofuran	3-OH-	3-K-7-P	3-K-CF	7-phenol	Volatile	Soil-bound	Total
0	96.63	0.36	0.11	0.12	ND	ND	0.52	97.73
1	93.22	0.18	0.33	0.09	0.03	0.01	3.23	97.08
3	91.73	0.10	0.16	0.05	ND	0.02	7.18	99.23
7	87.73	0.79	0.37	0.07	0.28	0.11	9.98	99.32
14	83.00	0.92	0.77	0.13	0.05	0.25	12.98	98.08
30	77.39	0.33	0.20	0.02	ND	0.61	18.00	96.54
62	66.53	0.14	0.12	0.17	ND	1.67	27.48	96.11
92	59.65	ND	ND	ND	0.59	3.18	29.60	93.01
122	25.07	1.32	0.84	0.31	0.32	8.31	55.62	91.78
181	27.14	1.32	0.14	0.22	0.38	10.97	55.95	96.10
273	23.27	0.36	0.24	ND	1.08	14.11	59.23	98.27
365	20.96	0.56	0.26	0.14	0.36	16.60	57.83	96.71

3-0H-CF: 3-hydroxy-carbofuran
3-K-7-P: 3-keto-7-phenol
3-K-CF: 3-keto-carbofuran

The pH of the acidic soil samples showed no significant change during the study and ranged from 5.2 to 5.8. The pH of the alkaline samples remained between 7.4 and 8.0 in most samples but was 7.0 on day 181 and 6.6 on day 273. The microbial population remained viable and stable during the one-year period in both soils.

The only major degradation product (>10% of the applied radioactivity) in the acidic soil extracts was 3-keto-carbofuran, which reached a maximum of 12.41% of the applied radioactivity by day 181 and then decreased to 11.14% by day 365. The structure of 3-keto-carbofuran was confirmed by mass spectrometry and the structure of carbofuran was confirmed by two-dimensional TLC. Radioactivity from the alkaline soil in the NaOH traps was confirmed to be due to $^{14}CO_2$ by barium chloride precipitation. No degradation products exceeding 10% of the applied radioactivity were detected in the alkaline soil extracts. The other major products of degradation were soil-bound residues in both soils. A maximum of 35.41% (on day 365) and 59.23% (on day 273) of the applied radioactivity was incorporated in bound residues in the extracted acidic and alkaline soils respectively. Fractionation of the bound residues into humic acid, fulvic acid and humin indicated the presence of radioactivity in all three fractions.

The [^{14}C]carbofuran decreased from 97.49% at day 0 to 43.58% on day 365 in the acidic and from 96.63% on day 0 to 20.96% on day 365 in the alkaline samples. The half-life of [^{14}C]carbofuran in the test system calculated according to a first-order rate constant was 321 days and 149 days in the acidic and alkaline soils respectively.

<u>The photodegradation</u> of [^{14}C]carbofuran labelled in the phenyl ring, was studied in accordance with US EPA Guidelines under natural sunlight on a sandy loam soil at a field application rate of 1.7 kg ai/ha at approximately 22°C (McGovern and Shepler, 1989). The soil was sieved (2 mm) and sterilized before treatment. The control soil samples were covered to prevent exposure to light. All samples were placed in temperature-controlled chambers. Ethylene glycol and 10% NaOH were used to trap volatile organic compounds and CO_2 respectively, and air was drawn through both the irradiated and control chambers into separate sets of traps. Duplicate irradiated and control samples were analysed at 0, 3, 8, 15, 22 and 30 days after treatment.

The soil samples were extracted with methanol/water and the extracts assayed by LSC and analysed by HPLC. The remaining soil was combusted and assayed by LSC. More than 90% of the ^{14}C was recovered from all the samples. The ^{14}C from carbofuran decreased to 77% of the applied activity by day 30. The degradation products were the phenol, 3-hydroxy-carbofuran, the 3-hydroxy-7-phenol and CO_2, each <10% of the applied ^{14}C. Carbofuran was also found to be degraded to the 7-phenol in the dark, showing that the showing that 7-phenol is not (all) photochemically derived. The calculated photolysis half-life of carbofuran was 78 days and the half-life of carbofuran in the dark was 720 days.

Three <u>terrestrial field dissipation studies</u> were in accordance with the US Environmental Agency Pesticides Assessment Guidelines in vineyards in California. In all three Furadan 4F was incorporated into plots of soil at the maximum use rate of 11.2 kg ai/ha. Triplicate soil cores were taken from treated and control plots before and immediately after application, and then at intervals for about a year. The cores were to a depth of 120 cm for the first 14 days and 240 cm thereafter. Each core was divided into 15 cm sections which were composited in groups of three to provide triplicate samples at each 15 cm depth, which were analysed for carbofuran, 3-hydroxy-carbofuran and 3-keto-carbofuran.

The first study was in Napa in 1987 (Daly and Tanner, 1988). The treated plot was approximately 30 x 30 m (11 rows of 10 vines) and the control plot was 72 m from the treated plot. The soil cores were taken before and immediately after incorporation and on days 3, 7, 14, 40, 90, 120, 150, 180, 304, 335 and 360. The soil was classified as a loam with the following characteristics.

pH	6.6
% Sand	41
% Silt	13
% Clay	46
CEC (meq/l)	28.7
Bulk density (g/cc)	1.4

The analytical limit of detection was 0.01 mg/kg and the limit of quantification 0.05 mg/kg. More than 77% of the residue in the soil was carbofuran. Residues of 3-keto-carbofuran and 3-hydroxy-carbofuran increased in the 0-15 cm soils for 30 days then decreased to <0.05 mg/kg by 108 days. The average total carbamate residue in the top 15 cm ranged from 3.16 to 6.66 mg/kg during the first 30 days. Quantifiable residues were not detected below the 105-120 cm. depths at any time except on day 30 at a level of 0.73 mg/kg at 120-135 cm. After 181 days the residues were below the limit of quantification at all depths. The first-order half-life calculated from the 0-15 cm depth was 43 days. A summary of the results is given in Table 16.

Table 16. Carbamate residues in soil dissipation study (Napa, 1987-88).

Mean total carbamate residue, mg/kg											
Depth,	Days after application										
inches	0	5	7	14	30	108	119	150	181	282	388
0-6	3.16	4.65	4.07	6.66	4.45	0.55	0.26	0.15	0.14	(0.02)	(0.04)
6-12	0.29	0.34	0.14	0.05	0.12	0.28	0.10	(0.04)	(0.03)	ND	ND
12-18	0.13	0.07	0.11	(0.02)	ND	0.36	0.19	0.05	(0.02)	ND	ND
18-24	0.09	0.06	0.07	(0.01)	(0.02)	0.11	0.25	0.05	0.05	(0.01)	ND
24-30	0.11	0.08	0.06	(0.02)	ND	0.08	0.16	(0.02)	0.09	(0.01)	ND
30-36	0.08	0.07	0.06	(0.01)	ND	(0.04)	0.10	(0.02)	0.06	(0.01)	ND
36-42	0.07	0.09	(0.04)	ND	(0.01)	(0.02)	0.06	(0.01)	(0.02)	(0.01)	ND
42-48	0.10	0.08	0.06	(0.03)	(0.04)	(0.01)	(0.01)	ND	(0.03)	ND	ND
48-54	NS	NS	ND	NS	0.73	ND	ND	ND	(0.04)	ND	ND
54-60	NS	NS	NS	NS	ND	ND	ND	ND	(0.03)	ND	ND
60-66	NS	NS	NS	NS	ND	ND	ND	ND	(0.03)	ND	ND
66-72	NS	NS	NS	NS	ND	ND	ND	ND	ND	ND	ND
72-78	NS	NS	NS	NS	(0.01)	ND	ND	ND	ND	ND	ND
78-84	NS	NS	NS	NS	(0.02)	ND	ND	ND	ND	ND	ND
84-90	NS	NS	NS	NS	ND	ND	ND	ND	ND	ND	ND
90-96	NS	NS	NS	NS	ND	ND	ND	(0.02)	ND	ND	ND

NS: no soil core sample was taken ND: undetectable (<0.01 mg/kg)
Values in parenthesis are estimated, below the limit of quantification (0.05 mg/kg) but above the limit of detection (0.01 mg/kg)

A second study was in Farmersville in 1988-1989 (Herbert, 1989). The treated plot was 36 x 24 m (10 rows of 10 vines) and the control plot was 72 m from the treated plot. Core samples were taken before and immediately after incorporation and on days 3, 7, 14, 40, 90, 120, 150, 180, 304, 335 and 360. The soil was classified as a loam with characteristics shown in Table 17.

Table 17. Soil characteristics, Farmersville dissipation study.

{PRIVATE }Soil depth (inches)	0-12	12-24	24-36	36-48	48-60	60-72	72-84	84-96
PH	7.49	7.55	7.54	7.51	7.55	7.48	7.99	8.01
% Sand	51.4	66.8	69.8	70.8	70.8	68.6	70.6	71.6
% Silt	32.0	18.4	13.4	12.4	11.4	18.2	19.2	17.2
% Clay	16.6	14.8	16.8	16.8	17.8	13.2	10.2	11.2
CEC (meq/l)	10.1	5.1	5.5	5.4	5.5	7.7	8	12.4
% organic matter	1.04	0.34	0.07	0.04	0.06	0.20	0.09	0.10
Bulk density (g/cc)	1.32	1.37	1.40	1.35	1.38	1.47	1.49	1.56

The limit of detection of the analyses was 0.02 and the limit of quantification 0.05 mg/kg. More than 80% of the total residue in the soil was carbofuran. The average total carbamate residue in the 0-15 cm depth ranged from 4.41 to 6.07 mg/kg during he first 14 days, with minimal leaching. The dissipation of residues in this layer in 360 days was significant. Quantifiable residues were not found below 45 cm at any time except on day 40 at a level of 0.08 mg/kg at 120-135 cm and on day 304 at 0.25 mg/kg at 105-120 cm. The first-order half-life calculated from the 0-15 cm soil depth was 23 days. The results are given in Table 18.

Table 18. Carbamate residues in soil dissipation study (Farmersville, 1988-89).

{PRIVATE }	Mean total carbamate residue, mg/kg											
Depth	Days after application											
Inches	0	3	7	14	40	90	120	150	180	304	335	360
0-6	5.91	6.07	5.74	4.41	0.92	0.18	(0.02)	0.05	ND	ND	(0.03)	(0.03)
6-12	0.48	0.86	0.81	1.07	1.13	(0.02)	ND	(0.04)	ND	ND	ND	ND
12-18	ND	ND	ND	ND	0.51	(0.02)	ND	0.05	ND	ND	ND	ND
18-24	ND	ND	ND	ND	(0.02)	ND	ND	(0.03)	ND	ND	ND	ND
24-30	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
30-36	ND	ND	ND	ND	ND	ND	ND	(0.02)	ND	0.09	ND	ND
36-42	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.26	(0.02)	ND
42-48	ND	ND	ND	ND	ND	(0.02)	ND	ND	ND	0.25	ND	ND
48-54	NS	NS	NS	NS	0.08	ND	ND	(0.04)	ND	(0.02)	ND	ND
54-60	NS	NS	NS	NS	(0.04)	ND	ND	ND	ND	ND	ND	ND
60-66	NS	NS	NS	NS	ND	ND	ND	ND	ND	ND	ND	ND
66-72	NS	NS	NS	NS	ND	ND	ND	ND	ND	ND	ND	ND
72-78	NS	NS	NS	NS	ND	ND	ND	ND	ND	ND	(0.04)	ND
78-84	NS	NS	NS	NS	ND	ND	ND	ND	ND	ND	ND	ND
84-90	NS	NS	NS	NS	ND	ND	ND	ND	ND	ND	ND	ND
90-96	NS	NS	NS	NS	ND	ND	ND	ND	ND	ND	ND	ND

NS: no soil core sample was taken
ND: undetectable (<0.02 mg/kg)
Values in parenthesis are estimated, below the limit of quantification (0.05 mg/kg) but above the limit of detection (0.02 mg/kg)

A third study was in conducted in Porterville 1988-1989 (Leppert, 1989). The treated plot was 36 x 24 m (10 rows of 10 vines) and the control plot was 48 m from the treated plot. Core samples were taken before application and on days 0, 3, 7, 14, 50, 61, 90, 120, 150, 180, 307 and 387. The soil was classified as a sandy loam. Its characteristics are shown in Table 19.

Table 19. Soil characteristics, Porterville dissipation study.

Soil Depth (inches)	0-4"	4-8"	8-12"	12-24"	24-36"	36-48"	48-60"	60-72"	72-84"	84-96"
pH	7.27	7.36	7.51	7.52	7.51	7.45	7.41	6.94	6.81	7.24
% sand	64.6	68.8	71.6	75.6	76.2	77.2	66.2	24.2	28.0	32.0

% silt	22.4	18.2	16.4	12.2	12.8	10.8	17.8	47.8	40.0	31.0
% clay	13.0	13.0	12.0	12.2	11.0	12.0	16.0	28.0	32.0	37.0
CEC (meq/l)	4.1	4.0	4.1	5.0	4.4	4.3	6.0	17.3	15.8	17.6
% organic matter	0.38	0.26	0.13	0.2	0.13	0.1	0.2	0.42	0.37	0.15
Bulk density (g/cc)	0.92	0.95	1.05	1.35	1.3	1.32	1.29	1.25	1.32	1.38

The limit of detection was 0.02 mg/kg and the limit of quantification 0.05 mg/kg. More than 80% of the total residue in the soil was carbofuran. The levels of 3-hydroxy-carbofuran did not increase, and it was undetectable by day 50. Residues of 3-keto-carbofuran increased for 14 days in the 15 cm layer, then decreased to undetectable levels by 50 days. The average total carbamate residue in the top 0-15 cm ranged from 4.05 to 5.19 mg/kg during the first 14 days, with minimal leaching. During this period there was little or no rainfall. At the next sampling, day 50, the residues had almost disappeared from the top 15 cm layer with movement of low levels into the lower depths. By 61 days, corresponding to the start of rainfall and irrigation, the residues had permeated the soil strata from 0-240 cm. The levels found, however, were much lower than the original 0-15 cm residues suggesting that various factors such as soil microbial activity, pH and sorption reduced the movement, particularly of oxidized carbamates, through the soil. Quantifiable residues were not found below the 150 cm depth at any time except on days 61 and 150 when residues were found at low levels (0.19 mg/kg) in the 225-240 cm layer. No residues were detectable at any depth after 150 days. A half-life of 13 days was calculated from all of the 0-15 cm residues.

A summary of the results is shown in Table 20.

Table 20. Carbamate residues in soil dissipation study (Porterville, 1988-89).

Mean total carbamate residue, mg/kg													
Depth	Days after application												
Inches	-1	0	3	7	14	50	61	90	120	150	180	307	387
0-6	ND	5.19	4.36	4.17	4.05	(0.04)	0.06	ND	ND	0.07	ND	ND	ND
6-12	ND	0.75	1.01	0.94	1.40	0.05	ND	ND	ND	0.08	ND	ND	ND
12-18	ND	ND	ND	ND	0.08	0.08	ND	ND	ND	0.06	ND	ND	ND
18-24	ND	ND	ND	ND	ND	0.09	0.05	ND	ND	0.05	ND	ND	ND
24-30	ND	ND	ND	ND	ND	0.08	0.11	ND	ND	(0.04)	ND	ND	ND
30-36	ND	ND	ND	ND	ND	(0.4)	0.15	ND	ND	0.05	ND	ND	ND
36-42	ND	ND	ND	ND	0.11	0.08	0.48	0.07	ND	ND	ND	ND	ND
42-48	ND	ND	ND	ND	0.71	0.12	0.64	0.08	ND	ND	ND	ND	ND
48-54[1]	ND	NS	NS	NS	NS	(0.04)	0.24	0.07	ND	0.14	ND	ND	ND
54-60	ND	NS	NS	NS	NS	(0.02)	0.22	0.09	ND	0.17	ND	ND	ND
60-66	ND	NS	NS	NS	NS	ND	0.20	(0.02)	ND	0.32	ND	ND	ND
66-72	ND	NS	NS	NS	NS	ND	0.21	ND	ND	0.28	ND	ND	ND
72-78	ND	NS	NS	NS	NS	ND	0.19	(0.02)	ND	0.18	ND	ND	ND
78-84	ND	NS	NS	NS	NS	(0.04)	0.21	(0.04)	ND	0.21	ND	ND	ND
84-90	ND	NS	NS	NS	NS	0.05	0.06	(0.02)	ND	0.19	ND	ND	ND
90-96	ND	NS	NS	NS	NS	(0.02)	0.19	(0.04)	ND	0.17	ND	ND	ND

NS: no soil core sample was taken.
ND: undetectable (<0.02 mg/kg)
Values in parenthesis are estimated, below the limit of quantification (0.05 mg/kg) but above the limit of detection (0.02 mg/kg)
[1]The sampling equipment may have caused contamination of the cores below 120 cm. The equipment could take only a 120 cm core, so to take a deeper core the probe had to be reinserted into the same hole after the 0-120 cm core had been taken. In some cases, leaf debris and root fragments were found where there should have been none

Adsorption/desorption was studied in a 0.01 M $CaCl_2$ solution with $[^{14}C]$carbofuran labelled in the phenyl ring (Leppert, 1989). The nominal test concentrations were 0, 0.5, 1.5 and 10 mg/kg. The samples were maintained in an environmentally-controlled chamber at approximately $25^{\circ}C$. Two soils, a silt loam and a sandy loam, were used. Concentrations of carbofuran were estimated in the aqueous phase by LSC and in the soils by combustion followed by LSC after the desorption phase. Adsorption and desorption constants were determined for the silt loam, but desorption could not be accurately determined for the sandy loam owing to the small amount of test material adsorbed during the adsorption phase. The mass balances for the silt loam and sandy loam were 106% and 104% respectively. The average K_{oc} of 24.7 indicates that carbofuran has the potential to be mobile in the two soils tested. The results are summarized below.

Soil type	% organic carbon	pH	Adsorption K_d	Adsorption K_{oc}	Desorption K_d	Desorption K_{oc}
Silt loam	1.2%	7.1	0.246	20.5	0.243	20.3
Sandy loam	0.4%	6.5	0.115	28.9		

<u>Column leaching study</u> was conducted to determine the mobility of [*phenyl*-^{14}C]carbofuran and its degradation products in four agricultural soils (Saxena *et al*., 1994). The soils were a sandy loam from Georgia (GA), a clay loam and a loam from Ohio, and a sandy loam from California (CA). The CA sandy loam had an organic matter content of less than 1%. The characteristics of the soils are tabulated below.

Source	Type [USDA]	pH	% OM	CEC meq/ 100g	Sand, %	Silt, %	Clay, %	Bulk density, g/cm^3
Georgia	sandy loam	5.7	1.2	4.3	73	16	11	1.42
Ohio	clay loam	5.8	4.6	24.7	23	38	39	1.13
California	sandy loam	6.8	0.6	5.0	65	28	7	1.39
Ohio	loam	7.6	1.5	13.9	29	46	25	1.12

OM: organic matter
CEC: cation exchange capacity

A preliminary study was conducted to determine the rate of degradation of [^{14}C]carbofuran on each soil type, and hence the sampling intervals and the length of time for the aerobic aging-phase in the definitive study (one half-life or 30 days, whichever was shorter). The four soils were fortified with [^{14}C]carbofuran at a concentration of 3.2 mg/kg and incubated at 25 ± 1°C under aerobic conditions for 15 days. The soil moisture was maintained at approximately 75% of field capacity throughout the preliminary study. Duplicate soil samples were collected on days 0, 5, 10 and 15. The samples from days 5 and 10 were frozen upon collection. The samples from days 0 and 15 were extracted immediately after collection and the extracts analysed by HPLC. Since less than 20% of the carbofuran in the two sandy loams and the clay loam was degraded by day 15, the sampling points for these soils in the definitive study were days 0, 15, 22 and 30. In the loam soil approximately 41% of the carbofuran was degraded by day 15 so the samples from days 5 and 10 were extracted and analysed to determine the half-life of carbofuran, which was calculated to be 21.9 days. Samples in the definitive study were therefore taken on days 0, 9, 15, 20 and 23.

In the major study each of the four soils was fortified with [*phenyl*-^{14}C]carbofuran at a concentration of 3.2 mg/kg (equivalent to 6.7 kg ai/ha, which represents the highest single application for row crops) and incubated aerobically at 25 ± 1°C. The soil moisture was maintained at approximately 75% field capacity throughout the study. $^{14}CO_2$ and other volatile products were trapped and quantified. Duplicate soil samples were extracted immediately upon collection and analysed by HPLC.

The mean recoveries of the applied radioactivity and the half-life of carbofuran in each soil are shown in Table 21.

Table 21. Recoveries of ^{14}C and half-life of carbofuran in four soils.

Soil	^{14}C recovered, %				Half-life, days
	Extracted	Volatile	Bound	Total	
GA sandy loam	87.6	0.1	9.9	97.6	90.8
clay loam	73.1	2.7	22.0	97.8	53.0
CA sandy loam	86.2	2.4	9.2	97.8	99.9
loam	49.4	4.1	42.4	95.9	21.9

The proportion of the recovered radioactivity associated with each of the compounds determined by HPLC is shown in Table 22.

Table 22. Distribution of recovered ^{14}C.

Soil	^{14}C, % of recovered and mg/kg as carbofuran		
	Carbofuran, %	3-OH-carbofuran (% mg/kg)	3-keto-carbofuran (%, mg/kg)
GA sandy loam, day 30	81.6	2.1 (0.07)	1.6 (0.05)
Clay loam, day 30	67.1	0.2 (<0.01)	2.8 (0.09)
CA sandy loam, day 30	79.1	0.8 (0.03)	3.4 (0.11)
Loam, day 23	46.1	0.1 (<0.01)	0.1 (<0.01)

The remaining radioactivity was distributed among soil-bound residues and $^{14}CO_2$. The identification of 3-ket°Carbofuran was confirmed by LC-MS.

The remaining soil samples of each soil from the definitive study were combined and used as the aged soil in the leaching study. The soils were packed in columns to a height of 30 cm and the aged soil containing the [^{14}C]carbofuran residues at a nominal concentration of 6.7 kg ai/ha was applied to each column. The columns were maintained at approximately 25 ± 1°C and leached with 50 column cm of 0.01 N $CaCl_2$ at an approximate rate of 1.5 cm per hour. Four fractions of leachate (approximately 12.5 cm or 600 ml each) were collected from each column. The leachates and the soils in the columns were assayed for radioactivity. The proportion of the applied radioactivity in the leachates and soil sections were as follows.

% of radioactivity applied to column

	Georgia sandy loam	Clay loam	California sandy loam	Loam
Aged soil layer	42.5	31.1	13.7	49.2
Total in six soil sections	9.4	26.5	2.8	9.0
Total in leachates	53.4	40.9	78.2	33.2
Mass balance	105.3	98.5	94.7	91.4
K_d	0.73	*	0.25	*

*less than 50% of the applied radioactivity was in the leachate

Leachate fractions that contained >10% of the applied ^{14}C were analysed by HPLC. The identity of carbofuran was also confirmed by two-dimensional TLC. More than 94% of the ^{14}C from all four soils was due to [^{14}C]carbofuran. Minor components (less than 1% of the applied radioactivity) detected in the leachates and/or aged layer sections included 3-keto-carbofuran (0.8%) the 7-phenol (0.2% in GA sandy loam, 0.2% in clay loam, 0.5% in loam), the 3-keto-7-phenol (0.2%) and 3-keto-carbofuran (91.8% in CA sandy loam).

The proportion of leached radioactivity was greatest in the CA sandy loam, followed by GA sandy loam, clay loam and loam. The results indicate that carbofuran and its degradation products have the potential to be mobile in all four soils under the "worst-case" conditions of applying 50 column cm of water.

Environmental fate in water/sediment systems

Cook (1974) studied the hydrolysis of [*phenyl*^{14}C]carbofuran in aqueous solutions buffered to pH 5, 7 and 9 at a concentration of 2 mg/l. At room temperature (28°C), carbofuran was hydrolytically stable over the 28-day test period at pH 5 and was slowly hydrolysed at pH 7 with a calculated half-life of 26 days. At pH 9 only 20% of the carbofuran remained after 1 day at 26°C and the half-life was 12 hours. At 5°C the half-life was 1.5 days. The hydrolysis product was the 7-phenol.

Degradation in water/sediment systems

In a study in accordance with US EPA Guidelines (Saxena *et al.*, 1994b) the rate and degree of the anaerobic aquatic degradation of [^{14}C]carbofuran was determined in acidic pond water plus sediment systems (approximate pH 5.4) consisting of approximately 82 g wet sediment, equivalent to 50 g oven-dried sediment and 100 ml of pond water in sealed bottles. Test systems were prepared and incubated under anaerobic conditions in the dark at approximately 25°C for at least 30 days (pre-anaerobic incubation) before adding [^{14}C]carbofuran uniformly labelled in the phenyl ring at a nominal concentration of approximately 3 mg/kg (equivalent to 6.7 kg ai/ha) and incubating at 25°C under anaerobic conditions for 12 months in the dark. The test systems were incubated in a “static” anaerobic apparatus that permitted the trapping of organic volatiles and $^{14}CO_2$. Duplicate samples of sediment plus water were collected immediately after dosing (day 0) and after 1, 3, 7, 14, 31, 60, 98, 122, 183, 273 and 365 days. The samples were flushed with nitrogen to collect organic volatiles, $^{14}CO_2$ and [^{14}C]methane and the dissolved oxygen content, pH and redox potential of the water samples were determined. The populations of aerobic and anaerobic microbes were also measured. The sediment and water were extracted and analysed by HPLC. The identities of compounds that accounted for more than 10% of the applied radioactivity were confirmed by liquid chromatography-mass spectrometry (LC-MS). The extracted sediment was combusted to determine the amount of bound radioactivity.

More than >90% of the ^{14}C was accounted for in all the samples. Volatile radioactivity was negligible and reached a maximum of 0.5% by day 273. [^{14}C]carbofuran decreased from 96.2% at day 0 to 24.9% by day 365. One major product, the 7-phenol, reached a maximum of 53.7% by day 365, when the maximum level of 20.4% of the applied radioactivity was observed. Fractionation of the bound residues into humic acid, fulvic acid and humin indicated the presence of radioactivity in all three fractions. The observed redox potential and dissolved oxygen values indicated that anaerobic conditions were maintained. A summary of the results is given in Table 23.

Table 23. Anaerobic degradation in a water/sediment system.

Day	% of ^{14}C as					
	carbofuran	3-keto-7-phenol	7-phenol	Trapped volatiles	Bound residues	Total
0	96.2	ND	0.4	NA	0.3	96.8
1	91.1	ND	0.4	0.1	0.8	92.3
3	90.9	0.1	0.5	0.1	1.1	92.6
7	90.4	0.3	0.9	0.3	3.0	94.8
14	85.9	0.1	0.8	0.2	7.8	94.7
31	75.9	0.4	9.6	0.2	11.8	97.8
60	64.7	0.4	17.9	0.3	11.7	94.9
98	55.5	ND	26.6	0.3	14.3	96.7
122	47.0	ND	30.9	0.3	16.1	94.3
183	41.9	ND	39.3	0.3	16.7	98.2
273	34.0	ND	45.3	0.5	18.8	98.6
365	24.9	ND	53.7	0.2	20.4	99.1

The average half-life of [^{14}C]carbofuran in the test system, assuming first order kinetics, was approximately 189 days.

The rate and degree of aerobic degradation of [^{14}C]carbofuran uniformly labelled in the phenyl ring was determined in an acidic pond water/sediment system (approximate pH 5.4) by Saxena and Marengo (1994). Each vessel contained 50 g of pond sediment and 100 ml of pond water fortified with [^{14}C]carbofuran at a concentration of 3.05 mg/kg (equivalent to 6.7 kg ai/ha which represents the highest single application for row crops) and incubated at 25 ± 1°C under aerobic conditions for 30 days in darkness. The vessels were connected in pairs to a set of traps (ethylene glycol for organic volatiles, sodium hydroxide for CO_2) and CO_2-scrubbed humidified air was bubbled through the overlying water of the first vessel of each pair into the water of the second and then into the traps.

Pairs of sediment/water vessels were taken on days 0, 1, 3, 7, 10, 20 and 30 and the contents analysed immediately upon collection. The population of aerobic bacteria, pH, dissolved oxygen content and redox potential of the test system were determined, the radioactivity in the traps was counted by LSC, that in the sediment and water was extracted and the extracts analysed by HPLC and the extracted sediment was combusted to determine bound ^{14}C. Selected extracts were also analysed by TLC to confirm the products detected by HPLC. The identities of compounds accounting for more than 10% of the applied radioactivity were confirmed by LC-MS.

The recovery of applied radioactivity from individual samples was >90% at all times. The distribution of the radioactivity at 0 and 30 days was as follows.

Mean % of applied radioactivity

Day	Water layer	Sediment extract	Extracted sediment	Traps	Total
0	71.07	24.81	3.92	0	99.8
30	15.29	45.86	32.78	1.87	95.8

The first sample in each pair remained acidic throughout the 30-day study with a pH of about 5, and the second sample remained acidic on days 0-10. A shift in the pH of the overlying water in the second vessel of the pair to about 8 was observed at days 20 and 30. A significant difference between the two samples in the degradation of carbofuran was caused by the shift in pH (carbofuran is known to be hydrolysed rapidly at an alkaline pH to the 7-phenol).

The 7-phenol was a major product in the two alkaline samples on days 20 and 30 (23.74 and 17.30% of the applied radioactivity respectively, compared with 0.61 and 1.89% in the first vessels). The other major degradation products were soil-bound residues which accumulated to an average of 32.67% by day 30. Fractionation of the 30-day sediments into humic acid, fulvic acid and humin indicated the presence of radioactivity in all three fractions. Carbofuran decreased to an average of 39.65% by day 30. Minor amounts (<1%) of 3-hydroxy-carbofuran, the 3-keto-7-phenol and 3-keto-carbofuran were detected in all the samples. No unidentified compounds were detected.

An additional study was conducted to determine whether the original arrangements of the vessels in pairs caused the second samples to become alkaline and hence the differences on days 20 and 30. Each vessel, with the same contents as before, was now connected to its own set of traps. Duplicate vessels were collected on days 0, 10, 20 and 30. The contents of the day 0 and day 30 vessels were analysed immediately upon collection as before. The pH, dissolved oxygen and redox potential were measured at days 10 and 20 and the samples were then stored frozen without further analysis.

The mass balance was >90% of the applied radioactivity in the day 0 and day 30 samples. A slight increase in the pH of the overlying water with time was observed in the individual vessels. The pH of the individual water samples at day 0 ranged from 5.23 to 5.50 and a maximum pH of 6.44 was observed in any individual sample during incubation. The 7-phenol was detected at a maximum level of 2.88% and a mean of 2.84% by day 30. No unidentified compounds were detected, the analyses of the duplicate samples at each time interval agreed, and the water samples remained acidic throughout the study. Evidently the connection of the vessels in sequence in the main study caused the pH change to alkaline and the differences between "replicates" on days 20 and 30.

The half-life of carbofuran was calculated by linear regression to be approximately 41 days.

Aqueous photolysis was studied with [^{14}C]carbofuran labelled in the phenyl ring at a concentration of 20 mg/l in a sterile buffer solution at pH 5 (McGovern and Shepler, 1989a). The samples were exposed to natural sunlight in a water bath at approximately 25°C together with control samples wrapped in aluminum foil. Duplicate irradiated and control samples were analysed 0, 3, 6, 12, 20 and 31 days after treatment. Ethylene glycol and 10% NaOH were used to trap volatile organic compounds and CO_2 respectively. Air was drawn through both the irradiated and control sample tubes into separate sets of traps. All samples were analysed directly by LSC and HPLC. The average recovery of ^{14}C from all samples was 97.1%. The 7-phenol and CO_2 were the only degradation products observed. The 7-phenol reached a maximum of 3.7% and CO_2 a maximum of 0.3% of the applied ^{14}C in the irradiated samples. The extrapolated half-life for photolysis was 1200 days, implying a half-life of 450 days in summer daylight conditions. Carbofuran was also found to be degraded slowly in the dark to the 7-phenol and CO_2 with a half-life of 2100 days, showing that the 7-phenol is not photochemically derived wholly from photolysis.

An aquatic field dissipation study was conducted in the USA in Louisiana and California (Novak, 1987a,b) to determine the distribution of carbofuran and its metabolites in soil, water and rice. Each site consisted of two 20 x 30 m rice plots, one control and one treated, surrounded by a levee. At the Louisiana site, the rice plots were flooded to a depth of 10 cm when the rice plants had reached this height, and the depth of the flood water was maintained between 8 and 21 cm until the rice was mature. The Louisiana plot was treated with Furadan 3G at 0.67 kg ai/ha 19 days after permanent flooding of the planted rice. The California plot was treated with Furadan 5G at 0.56 kg ai/ha immediately after sowing the rice seed before flooding. The plots were flooded to a depth of 20 cm and maintained at 15 to 20 cm. At both sites, an additional plot was planted with crops and irrigated with water from the treated rice plot.

At the Louisiana site, soil core samples were taken from the treated plot to a depth of 64 cm before and 24 hours after application, and then at days 3, 7, 14, 21, 30, 60 and 120. At the California site, soil samples were taken at the same intervals and also at days 162, 196 and 225. The soil cores were taken in the treated plot to a depth of 64 cm during the unflooded phase and 16 cm during the flooded phase. The soil cores were divided and analysed in 8 cm sections. Water samples were taken just before and 8 hours after application at both sites and on days 7, 14, 21, 30, 60 and 99 in Louisiana and 3, 7, 9, 12, 14, 16, 19, 21 and 27 in California. Four 1-l water samples were taken at each sampling at each site.

The soil samples were analysed for carbofuran and 3-hydroxy-carbofuran (Schreier, 1987). The stated limits of determination and detection were 0.1 and 0.02 mg/kg. The analyses showed only low levels (≤0.09 mg/kg) of carbofuran at both sites, and 3-hydroxy-carbofuran was not detected in any of the samples. The maximum residue of carbofuran at the California site, 0.09 mg/kg, occurred 7 days after treatment in the 0-8 cm section. Only the 1-60 days sample at the California site were analysed: both analytes were undetectable to a depth of 16 cm at 60 days. The maximum residue of carbofuran at the Louisiana site was 0.04 mg/kg in the 0-8 cm section after 3 days. Only the 1-21 day samples were analysed because both compounds were undetectable to a depth of 16 cm in the 7-21 day samples. The results are shown in Table 24.

Table 24. Aquatic field dissipation: carbofuran and 3-hydroxy-carbofuran in soil and sample.

Residue, mg/kg		
Site/Interval/Depth	carbofuran	3-hydroxy carbofuran
Louisiana		
Day 1 0-8 cm	(0.04)	ND
Day 1 8-16 cm	ND	ND
Day 3 0-8 cm	(0.04)	ND
Day 3 8-16 cm	ND	ND
Day 7 0-8 cm	ND	ND
Day 7 8-16 cm	ND	ND
Day 14 0-8 cm	ND	ND
Day 14 8-16 cm	ND	ND
Day 21 0-8 cm	ND	ND
Day 21 8-16 cm	ND	ND
California		
Day 1 0-8 cm	(0.05)	ND
Day 1 8-16 cm	ND	ND
Day 3 0-8 cm	(0.06)	ND
Day 3 8-16 cm	ND	ND

Residue, mg/kg		
Site/Interval/Depth	carbofuran	3-hydroxy carbofuran
Day 7 0-8 cm	(0.09)	ND
Day 7 8-16 cm	ND	ND
Day 14 0-8 cm	(0.07)	ND
Day 14 8-16 cm	ND	ND
Day 21 0-8	0.05	ND
Day 21 8-16 cm	ND	ND
Day 30 0-8 cm	(0.07)	ND
Day 30 8-16 cm	ND	ND
Day 60 0.8 cm	ND	ND

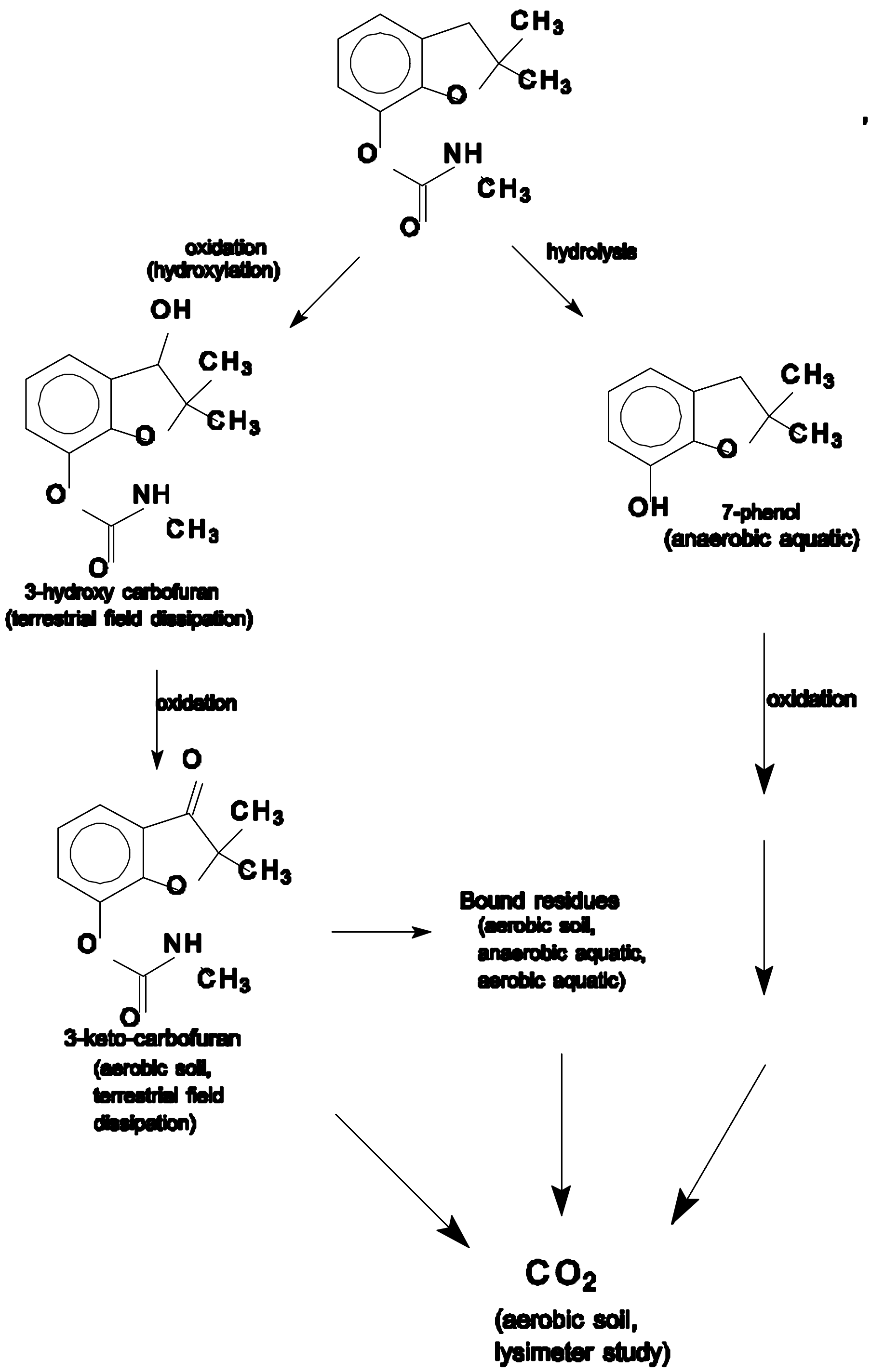
ND: undetectable (<0.02 mg/kg)
Results in parenthesis are estimated levels below the limit of determination (0.1mg/kg) but above the limit of detection (0.02 mg/kg)

Rice paddy water samples were also analysed for carbofuran and 3-hydroxy-carbofuran (Beauchamp, 1987). The limit of determination was 2.5 µk/kg and the limit of detection 1 µk/kg. The water from the Louisiana site contained a mean maximum level of carbofuran of 417 µk/kg after 8 hours, which decreased to 3 µk/kg after 30 days. 3-Hydroxy-carbofuran was detected only in one sample, at 1.0 µk/kg on day 7. The water from the California site contained a mean maximum level of carbofuran of 33 µk/kg after 8 hours which became undetectable by day 27. The half-life of carbofuran in rice paddy water was <10 days at both sites.

Carbofuran dissipated rapidly in soil and water after application to rice plots. Carbofuran was the only residue found in the soil and was undetectable by day 60. In the water, residues of carbofuran were ≤3 mg/kg by day 30 and no residues of 3-hydroxy-carbofuran were found except for a level of 1 µk/kg on day 7. No residues of carbofuran or 3-hydroxy-carbofuran were found in the rice grain or straw.

Proposed degradation pathways of carbofuran in soil and water/sediment systems are shown in Figure 4.

Figure 4. Degradation pathways of carbofuran in soil and water/sediment systems.



Rotational Crops

In a confined crop rotation study [*phenyl*-^{14}C]carbofuran was applied directly to a silt loam soil at an application rate of 3.4 kg ai/ha, based on a 76 cm row space. Wheat, soya beans and sugar beet were seeded into the treated soil 4 and 12 months after treatment and grown to maturity. Wheat forage, straw and grain, soya bean silage, stems, pods and beans and sugar beet tops and roots from both plantings were assayed separately for ^{14}C. Table 25 shows that residues above 0.01 carbofuran equivalents were found in all the samples from both plantings.

Table 25. Total radioactive residues in mature rotational crops, as carbofuran.

Crop	Sample	^{14}C, mg/kg as carbofuran	
		4 months	12 months
Wheat	Forage	-	1.40
	Straw	54.0	0.30
	Grain	0.60	0.04
Soya bean	Silage	16.0	0.50
	Stem	18.0	0.70
	Pod	5.0	0.10
	Bean	1.0	0.08
Sugar beet	Top	0.40	0.05
	Root	0.20	0.05

Subsamples of each plant part were extracted with methanol/water (1:2) and separated into non-polar and polar fractions which were concentrated and analysed separately to determine the nature of the residues. Conjugated metabolites were hydrolysed with 0.25 N hydrochloric acid. Metabolites were identified by TLC, with co-chromatography with reference standards.

The phenolic metabolites (3-hydroxy-7-phenol, the 3-keto-7-phenol and 7-phenol) were the principal degradation products found in the plants. The carbamates (carbofuran, 3-hydroxy-carbofuran and 3-keto-carbofuran) constituted a small proportion of the total radioactive residue; none of them individually exceeded 10% of the TRR in any of the crops sown at 4 or 12 months.

A field rotational crop study was conducted with [*phenyl*-^{14}C]carbofuran applied to the soil at rates of 1.1, 3.4 and 6.7 kg ai/ha. Ten months after treatment, sorghum, soya beans, sugar beet, lettuce, cabbage and wheat were planted in the field and grown to maturity. No ^{14}C was detectable in mature sorghum, soya beans, sugar beet, wheat grain or lettuce from any of the three application rates. Low levels of the total residue (0.01 mg/kg as carbofuran) were observed in mature cabbage harvested from the 3.4 and 6.7 kg ai/ha treatments. Wheat straw and soya bean stems harvested from the 6.7 kg ai/ha treatment contained 0.21 mg/kg and 0.63 mg/kg respectively, but residues were not detectable at the two lower treatment rates. Residues in the immature crops from the 1.1 and 3.4 kg ai/ha treatments harvested 30 and 58 days after planting were generally below the detection limit. Detectable levels of radiocarbon (0.017-0.084 mg/kg as carbofuran) were found in immature crops from the 6.7 kg ai/ha treatment. Low levels of carbofuran (maximum 0.02 mg/kg) remained in the soil ten months after application.

The results indicate that under normal field use conditions the potential for accumulation of carbofuran into ten-month rotational crops is minimal at application rates of 1.1-6.7 kg ai/ha. The residues from all treatment rates were below the limit of detection in all the edible commodities except cabbage where they were at the limit of detection at the two higher treatment rates.

METHODS OF RESIDUE ANALYSIS

Analytical methods

Schreier (1989) provided a GLC method for the determination of carbofuran, 3-hydroxy-carbofuran and 3-keto-carbofuran in green and dry alfalfa, field corn silage and grain, oranges, peanut nutmeat and hulls, potato tubers, sorghum, sugar beet roots and tops and cow milk and muscle. The weighed sample was macerated, hydrolysed by refluxing for one hour with 0.25 N HCL and filtered. The filtrate was partitioned with dichloromethane, transferred to hexane and cleaned up on a Florisil column. Ethyl acetate was used to elute the compounds of interest. The final solutions were analysed on a 10 or 12 m methyl silicone capillary column, either 0.53 mm with direct injection and a nitrogen-phosphorus detector (NPD) or 0.2 mm with splitless injection and a mass-selective detector (MSD). Calibration was with external standards. Chromatograms and raw data were provided for fortified control samples to demonstrate the limits of determination given in Table 26. Limits of determination were also claimed for milk and muscle (0.5 mg/kg for each analyte) and several plant crops, but no chromatograms or data were submitted.

Table 26. Limits of determination of carbofuran and carbamate metabolites by the method of Schreier (1989).

{PRIVATE }Sample	Detector	Limit of determination, mg/kg		
		Carbofuran	3-hydroxy-carbofuran	3-keto-carbofuran
Maize silage	MSD	1.0	3.0	1.0
Peanut kernels	MSD	0.5	0.5	0.5
Peanut hulls	MSD	2.0	2.0	2.0
Potato	MSD	0.5	0.5	0.5
Sorghum	NPD	1.0	1.0	1.0
Sugar beet tops	NPD	1.0	1.0	1.0

Modifications to the Schreier method, e.g. by Brutschy (1984) included ethoxylation of 3-hydroxy-carbofuran and a procedure for the isolation and determination of the phenol metabolites. For the determination of phenol metabolites the hydrolysed and filtered sample was partitioned with methylene chloride/diethyl ether (3:3), butylated hydroxytoluene (1 ml of 10 mg/kg in methylene chloride) and ethanol were added to the extract and the solution was concentrated to remove methylene chloride, acidified, and refluxed for 45 minutes. It was then partitioned into methylene chloride, which was concentrated, cleaned up on a silica gel solid-phase extraction column conditioned with methanol/water (1:1) and eluted with methylene chloride. The eluate was concentrated and analysed by GLC on a cross-linked dimethyl silicone capillary column operated in the splitless mode. Detection was mass-selective with monitoring of the molecular ions of the 7-phenol, 3-keto-7-phenol and 3-ethoxy-7-phenol ($164^{+\cdot}$, $178^{+\cdot}$ and $208^{+\cdot}$). Calibration was with external standards.

Mollhoff (1975a) described a method for the determination of carbofuran, 3-hydroxy-carbofuran and 3-hydroxy-carbofuran glycoside in plants and soil. Plant samples (cereal grains, potatoes) were macerated with methanol and the macerate filtered, concentrated and extracted with chloroform. The residual aqueous fraction was hydrolysed with acid to convert any glycoside conjugate to 3-hydroxy-carbofuran aglycone and extracted with chloroform. Soil samples were macerated with a mixture of methanol, water and hydrochloric acid and extracted with chloroform. Analyses were by GLC on a packed column (Ucon LB 550 X with 0.5% KOH on Chromosorb G AW DMCS). A limit of determination of 0.1 mg/kg claimed and recoveries were reported for several crops, but no data were provided. Recoveries of the conjugate were generally unacceptable at or below 0.6 mg/kg. Several acceptable recoveries of the conjugates were reported at 1.0 mg/kg.

Leppert *et al*. (1983) reported a GLC method for the determination of carbosulfan and carbofuran residues in soil, plants and water. Crops with a high water content, e.g. green alfalfa or citrus, were blended with hexane and 2-propanol (2:1), diluted with water and the hexane fraction retained. Soil and crops with a low water content, e.g. hay and straw were blended with methanol and pH 8 buffer and the filtered solution was extracted with methylene chloride. Oily samples, e.g. citrus oil, were diluted with hexane and extracted with acetonitrile. Water samples were extracted with methylene chloride after salting. Various column clean-up procedures were used, including gel permeation, Darco-Attaclay, aluminum oxide and Florisil. After the Darco-Attaclay fractionation, the ethyl acetate eluate was concentrated and treated with ethanol and concentrated HCl to ethoxylate 3-hydroxy-carbofuran. The final extracts were analysed on a packed column of Chromosorb W-HP with nitrogen-selective detection. A limit of determination of 0.1 mg/kg carbofuran was reported for citrus fruit.

Smith (1991) reported a method for the determination of parts-per-billion levels of carbofuran in water. Samples were concentrated on a C-18 solid-phase extraction column, eluted with acidified methanol and analysed with by HPLC on a cyclohexyl column with a UV detector (220 nm). The mobile-phase was a water/acetonitrile gradient. Adequate resolution and sensitivity were demonstrated for a rice-water sample fortified with carbofuran at 11 µk/kg .

Barros (1995) described a multi-residue method for the determination of carbosulfan and its metabolites in or on oranges. In addition to carbosulfan, the method determines carbofuran, 3-keto-carbofuran, and the 3-hydroxy-carbofuran, 3-keto-7-phenol, 7-phenol and 3-hydroxy-7-phenol.

To determine the carbamates, macerated oranges were hydrolysed with 0.25 N HCl under reflux, the mixture was filtered and an aliquot of the filtrate was loaded onto a C-18 solid-phase extraction cartridge conditioned with methanol and 0.25 N HCl. The compounds of interest were eluted with 1% methanol in methylene chloride and passed through an aminopropyl solid-phase cartridge. The final residue was re-dissolved in acetonitrile and analysed by reverse-phase HPLC (C-18) with a post-column reactor and fluorescence detector. The demonstrated limit of determination was 0.03 for each analyte.

To determine the phenols, a separate aliquot of the original filtrate was loaded onto a C-18 cartridge and the dried cartridge was eluted with 5% ethanol in methylene chloride. The phenols were derivatized with pentafluorobenzyl bromide and the 3-hydroxy-7-phenol derivative was ethylated. A final ethanol solution of the analytes was analysed by gas chromatography with a mass-selective detector. The demonstrated limit of determination was 0.03 mg/kg for each analyte.

Geno (1991) reported validation of the Barros method for maize silage. The independent laboratory validation was in accordance with US EPA PR Notice 88-5, 40 CFR Part 160. Control maize silage was fortified with 0.05 or 0.25 mg/kg each of carbofuran, the 3-keto-carbofuran and 3-hydroxy-carbofuran. Adequate recoveries were demonstrated for all three compounds (83-102%, 95-102% and 96-108% respectively). The method was not validated for the phenol metabolites.

Blass and Philipowski (1992) reported a method for the determination of methylcarbamate residues, including carbofuran, by HPLC with post-column reaction. Samples with little or no fat were extracted with methylene chloride/water and fatty samples with acetonitrile saturated with hexane. The latter extract was washed with hexane, concentrated and extracted with methylene chloride. The final organic extracts were cleaned up on an "Extrelut" cartridge. Aqueous extracts were prepared for the determination of 3-hydroxy- carbofuran. The analytes were separated on a Spherisorb RP 18 column and the eluted methylcarbamates converted in a two-stage reactor to (1-hydroxyethylthio)-2-methylisoindole and the indole measured with a fluorimeter (excitation 340 nm, emission 455 nm). The limit of determination is approximately 0.04 mg/kg for carbofuran and 3-hydroxy-carbofuran. The recoveries given in Table 27 were reported for various samples fortified with carbofuran and 3-hydroxy-carbofuran. Sample chromatograms were provided from barley grain, wheat straw, sugar beet foliage and lettuce.

Table 27. Recoveries of carbofuran and 3-hydroxy-carbofuran by the method of Blass and Philipowski (HPLC with post-column derivatization).

{PRIVATE }Sample	Fortification, mg/kg	Recovery, %	
		Carbofuran	3-hydroxy-carbofuran
Apple	0.04; 1.0	84; 100	
Beet foliage	0.04; 1.0	76; 85	73; 84
Carrot	0.04; 1.0	94; 94	79; 87
Cherry	0.04; 1.0	90; 85	
Maize grain	0.04; 0.1; 1.0	99; 99; 97	100; 98; 95
Lettuce	0.04; 1.0	92; 95	88; 89
Melon	0.04; 1.0	92; 84	78; 75
Pepper	0.04; 1.0	86; 91	78; 80
Potato	0.04; 1.0	79; 97	78; 80
Rice	0.04; 1.0	91; 91	90; 90
Soya beans	0.04; 1.0	96; 91	101; 98
Wheat straw	0.10; 1.0	102; 95	
Sunflower seed	0.04; 0.1; 1.0	92; 94; 97	86; 78; 94
Barley grain	0.04; 1.0	100; 96	
Asparagus	0.04; 1.0	104; 92	80; 84
Bulb onion	0.04; 1.0	82; 90	74; 83
Tomato	0.04; 1.0	94; 93	86; 76
Sugar beet foliage	0.04; 1.0	85; 91	78; 85
Sugar beet root	0.04; 1.0	90; 90	80; 83

The sponsors claim that the Blass method is the official enforcement screening method for use in Europe (see the official multi-residue methods of The Netherlands, below).

A multi-residue method is published in the US Food and Drug Administration (FDA) Pesticide Analytical Manual (PAM) for determining total residues of carbofuran in food for the enforcement of tolerances.

Chen (1995a) described a method for the determination of carbosulfan and its metabolites, including carbofuran and the metabolites of Figure 1, in ruminant commodities. Milk and tissues are extracted with acetone. The acetone extract is centrifuged and cleaned up by a combination of liquid-liquid extraction, solid-phase extraction and/or gel permeation chromatography. Carbofuran and carbamate metabolites are determined by HPLC with a post-column reactor and fluorescence detector. The phenolic metabolites are extracted and analysed by a similar procedure to that of Barros (GC-MS). Limits of determination of 0.025-0.50 mg/kg were demonstrated for carbofuran and the metabolites in milk and tissues. The recoveries from fortified controls are shown in Table 28.

Table 28. Recovery of carbofuran and its carbamate and phenol metabolites from milk and ruminant tissues by the method of Chen (1995a).

Sample	Fortificn., mg/kg	No. of samples	Recovery, %					
			carbofuran	3-K-CF	3-OH-CF	7-phenol	3-K-7-P	3-OH-7-P
Milk	0.025	19	93 ± 13	92 ± 11	84 ± 13			
Milk	0.025	18				88 ± 12	99 ± 11	104 ± 15
Muscle	0.050	4	88 ± 14	97 ± 17	94 ± 14			
Muscle	0.050	2				70	100	80
Kidney	0.050	2	88	102	72			
Kidney	0.050	2				78	103	91
Kidney	0.50	2				87	114	85
Fat	0.050	3	76 ± 1.5	89 ± 9.2	71 ± 8.6			

Abbreviated compound names: see Figure 1, p.

The Netherlands submitted official multi-residue methods for the determination of carbofuran and 3-hydroxy-carbofuran (The Netherlands, 1997). Fruits, vegetables and potatoes were chopped, homogenized and extracted with acetone/methylene chloride/petroleum ether (1:1:1). Nuts, cereals, oil seeds, tropical seeds and dried fruits were extracted with acetone/methylene chloride (1:1). The extracts were analysed without clean-up, by gas chromatography with electron capture or ion trap detection. The limits of determination were stated to be in the range of 0.01-0.05 mg/kg, with recoveries of >80%. A second, HPLC method, consisted in extraction with acetone, partitioning into methylene chloride/petroleum ether and clean-up on a solid-phase extraction cartridge if necessary. The final extract was analysed on a reverse-phase HPLC column, with post-column hydrolysis and derivatization of the resulting amine with *o*-phthaldialdehyde. Detection was by fluorescence at 340 and 455 nm. The stated limit of determination was 0.005 mg/kg. The HPLC method is essentially that of Blass and Philipowski (1992).

The Netherlands also reported two methods for the determination of residues in field trial samples (The Netherlands, 1997). The GLC method is that of Mollhoff (1975a). A limit of determination of 0.1 mg/kg was reported for carbofuran, metabolites and conjugates. This method was used for strawberries, red cabbage, onions, leek, celery, celeriac, cauliflower, carrots and Brussels sprouts. The second method consisted in extraction with methylene chloride, concentration, clean-up on alumina and determination of carbofuran and 3-hydroxy-carbofuran of a reverse-phase HPLC column with a UV detector (220 nm). The limits of determination were 0.02 mg/kg for carbofuran and 0.01 mg/kg for 3-hydroxy-carbofuran. Recoveries were reported to be 95 ± 6% (n = 6) for carbofuran at fortifications of 0.06-1.1 mg/kg and 85 ± 7% (n = ?) for 3-hydroxy-carbofuran at fortifications of 0.05-0.50 mg/kg.

Stability of pesticide residues in stored analytical samples

Storage stability studies were reported for green and dry alfalfa, maize grain and silage, oranges, peanut kernels and hulls, potatoes, sorghum stalks, sugar beet tops and roots and cow milk and muscle (Schreier, 1989b). Fortified control samples were stored at -18°C for about 2 years. Samples were analysed after intervals of 9-11 and 24-26 months by the method of Schreier (1989a). The results are shown in Table 29. CLICK HERE to continue

Table 29. Frozen storage stability of carbofuran and its metabolites added to various commodities.

Sample	Analyte	Fort.[1] mg/kg	Recovery, %	
			9-11 months	24-26 months
Alfalfa, green	carbofuran	1.0	103	90
	3-keto-carbofuran	1.0	105	100
	3-hydroxy-carbofuran	8.0	60	99
Alfalfa, dry	Carbofuran	2.5	86	94
	3-keto-carbofuran	2.5	85	93
	3-hydroxy-carbofuran	20.	80	100
Maize grain	Carbofuran	0.5	94	113
	3-keto-carbofuran	0.5	104	110
	3-hydroxy-carbofuran	0.5	116	115
Maize forage	Carbofuran	1.0	106	105
	3-keto-carbofuran	1.0	113	112
	3-hydroxy-carbofuran	3.0	131	101
Orange (whole)	Carbofuran	0.5	108	107
	3-keto-carbofuran	0.5	104	98
	3-hydroxy-carbofuran	0.5	86	92
Peanut kernels	Carbofuran	0.5	94	83
	3-keto-carbofuran	0.5	90	79
	3-hydroxy-carbofuran	0.5	86	102
Peanut hulls	Carbofuran	2.0	105	116
	3-keto-carbofuran	2.0	105	89
	3-hydroxy-carbofuran	2.0	105	104
Potato, tuber	Carbofuran	0.5	96	86
	3-keto-carbofuran	0.5	104	75
	3-hydroxy-carbofuran	0.5	96	79
Sorghum, stalk	carbofuran	1.0	84	93
	3-keto-carbofuran	1.0	111	87
	3-hydroxy-carbofuran	1.0	78	79
Sugar beet tops	carbofuran	1.0	88	86
	3-keto-carbofuran	1.0	47	46
	3-hydroxy-carbofuran	1.0	85	79
Sugar beet root	carbofuran	0.5	110	99
	3-keto-carbofuran	0.5	94	72
	3-hydroxy-carbofuran	0.5	96	97
Cow milk	carbofuran	0.5	96	97
	3-keto-carbofuran	0.5	106	89
	3-hydroxy-carbofuran	0.5	98	95
Cow muscle	carbofuran	0.5	96	74
	3-keto-carbofuran	0.5	102	72
	3-hydroxy-carbofuran	0.5	90	69

[1]The three analytes were combined in fortified samples

Storage stability studies were also conducted with the processed fractions of maize (Schreier, 1990a) and sugar cane. No loss of carbofuran or 3-hydroxy-carbofuran occurred during more than 2 years of frozen storage.

Definition of the residue

MRLs currently refer to the sum of carbofuran and 3-hydroxy-carbofuran, expressed as carbofuran; 3-keto-carbofuran, the 7-phenol, the 3-keto-7-phenol and 3-hydroxy-7-phenol are excluded. Studies of plant and animal metabolism displayed similar metabolic pathways. In ruminants and poultry the parent carbofuran constitutes less than <1% of the residue. The major carbamate metabolite is 3-

hydroxy-carbofuran, but it is only found in certain animal commodities, e.g. 12% of the TRR in egg yolk and 11% in ruminant kidneys. Carbofuran is found in some plant commodities, e.g. 14% of the TRR in maize forage, but 3-hydroxy-carbofuran is generally the predominant carbamate compound. The metabolite 3-keto-carbofuran is not usually detected and contributes little to the total carbamate residue.

Studies of plant metabolism have shown that conjugate(s) of 3-hydroxy-carbofuran can constitute an appreciable proportion of the total residue. For example in soya bean forage 11% of the TRR was free and 28% of the TRR was conjugated (acid-released) 3-hydroxy-carbofuran. In soya beans, 1.5% was free and 3.2% was conjugated 3-hydroxy-carbofuran (Table 3). As the conjugated form might be released after human ingestion, it must be considered as part of the defined residue.

The residue should be defined both for estimates of dietary intake and compliance with MRLs as carbofuran plus 3-hydroxy-carbofuran, free and conjugated, expressed as carbofuran.

USE PATTERN

Carbofuran is a systemic acaricide, nematicide and insecticide, applied to foliage at 0.25-1.0 kg ai/ha, to the furrow at planting at 0.5-4.0 kg/ha to control soil-dwelling and foliar-feeding insects, or broadcast at 6-10 kg/ha to control nematodes. Information on the use patterns on crops (labels and/or summary tables) provided by the sponsors and the governments of Australia and the UK are summarized in Table 30.

RESIDUES RESULTING FROM SUPERVISED TRIALS ARE SUMMARIZED IN TABLE

Data were supplied on at planting, foliar and directed applications of carbofuran to numerous crops, mainly in Australia, Europe, and North, Central and South America.

Residues judged to be from treatments according to GAP and used to estimate maximum residue levels are underlined. Those resulting from the maximum applications consistent with GAP and used to estimate STMRs are double underlined. All residues have been corrected for the average analytical recovery of the compound determined unless otherwise indicated.

Table 30. Summary of information on supervised trials (not necessarily according to GAP) provided by the sponsors.

Label uses: Crop	Country	Form, type	Application type	Application, kg ai/ha or /kg seed (all metric are g or kg ai)	Volume	Application no.	PHI, days	Remarks
Alfalfa	USA	4 F	foliar	2 pts/A; 1.12	2 GPA - aircraft	1-2/cutting 1.1.2 total	28	
Apple	India	3 G		5 g/tree				
Banana	Brazil	350 SC	immersion of the horn type seedling	1.4 g/100 l		1		
Banana	Brazil	350 ST	Seed treatment	0.14 kg/100 L		1		
Banana	Brazil	5 G		3-80 g/hole		2		4 mo. retreatment

Label uses: Crop	Country	Form, type	Application type	Application, kg ai/ha or /kg seed (all metric are g or kg ai)	Volume	Application no.	PHI, days	Remarks
Banana	Cyprus	10 G		1.5-3 g ai/mat			30	
Banana	Cyprus	10 G		1.5-3 g ai/mat			30	
Banana	Cyprus	5 G		1.5-3 g ai/mat			30	
Banana	Cyprus	75 WP		1.5-3 g ai/mat				
Banana	India	3 G		1.5 g/sucker				
Banana	Kenya	10 G		3 g ai/mat		2		
Banana	Philippines	10 G	at planting + 4 mo	1.25-1.5 g/hole; 5g		2		4 mo interval
Banana	Philippines	10 G	base of the plant	2.5- 3 g/mat		4		established plantations
Banana	Philippines	5 G	at planting	1.25-1.5 g/hole; 5 g/hole			at planting + 4months later	
Banana	Philippines	5 G		3.0 g/mat		4	0	once every 4 months for established plantations
Banana	Spain	20 F		5.6 kg ai/ha			60	
Banana	Spain	5 G		0.6-0.75kgai/ha			60	
Banana	Iv. Coast	10 G		3 g ai/mat				
Banana	Iv. Coast	4 F		1 g ai/mat			21	
Banana	Iv. Coast	5 G		3 g ai/mat				
Banana	Kenya	3 G		100gai/stool				
Banana	Kenya	5 G		3 g ai/mat		2		
Banana	Malaysia	3 G		0.6 g/tree				apply at base of tree at a distance of 1 foot
Barley	India	3 G	broadcast at plant	1.25 kg/ha				
Bean	Brazil	350 SC	at seeding	0.7-1.05 kg/ha	100-300l/ha	1		
Bean	Brazil	5 G	At planting, incorporated	1-2 kg/ha				
Bean	Cz. Republic	5 G		750 g ai/ha				
Beans	Argentina	10 G	at seedling	0.5 kg/ha				
Beets	Cz. Republic	10 G		1-2 kg ai/ha			>28	
Beets	Cz. Republic	350 F		1.05 kg ai/ha				
Beets	Cz. Republic	5 G		.75-1.5 kgai/ha				
Beets	Poland	5 G		.75 kg ai/ha				
Beets/fodder	Cz. Republic	350 ST		1.4/2.6kgai/100				
Beets/sugar	Cz. Republic	350 ST		1.4/2.6kgai/100				

Label uses: Crop	Country	Form, type	Application type	Application, kg ai/ha or /kg seed (all metric are g or kg ai)	Volume	Application no.	PHI, days	Remarks
Cabbage	Brazil	5 G	in furrow or filed hole	2 kg/ha		1		
Cabbage	Cz. Republic	10 G		2 kg ai/ha				
Cabbage	Cz. Republic	350 F		0.7 kg ai/ha				
Cabbage	Cz. Republic	350 F		0.0525 %				
Cabbage	India	3 G	at planting	1.5 kg/ha				
Canola	USA	10 CR		2.5lbs/A; 0.28				
Carrot	Cyprus	10 G		5-8 kg ai/ha			30	
Carrot	Cyprus	5 G		5-8 kg ai/ha			30	
Carrot	Cyprus	75 WP		5-8 kg ai/ha			30	
Coffee	Brazil	350 SC	gound application	0.35 g/hole				
Coffee	Brazil	5 G	at rains start and end of rains	0.5 - 3* g/hole		2		mechanically or manual application
Coffee	Brazil	5 G	incorporation in soil					* rate depends upon the the transplant age and pest infestation
Coffee	Kenya	10 G		2gai/tree x2				
Coffee	Kenya	5 G		4 g ai/tree				
Coffee	Malaysia	3 G		0.9 g /tree				apply around base of tree at one foot
Coffee	USA	10 G	incorporation in soil	1.5 g/tree		2		PR only. 1st application early winter (Jan/Feb); 2nd late June/July.
Corn (see maize also)	Argentina	10 G	in furrow, soil application	1-3.5 kg/ha				
Corn	Argentina	10 G						
Corn	Brazil	310 TS	seed treatment	697.5 g/100 kg seeds		1		
Corn	Brazil	350 SC	in furrow at seeding	1.05 -1.4 kg/ha	100-300l/ha	1		
Corn	Brazil	350 ST	Seed treatment	0.7-1.05 kg/100 kg seeds		1		
Corn	Brazil	5 G	At planting, incorporated	1.5-1.75 kg/ha		1		
Corn	Korea	3 G		3 TO 5		1	45	
Corn	USA	10 G	Post planting, band over row, incorporated	10 lbs/A; 1.1		1		row spacing = 40 inch
Corn	USA	10G	Foliar	10 lbs/A; 1.1		2		aerial application
Corn	USA	4 F	at planting	1 qt/A; 1.1		1	30 (forage)	row spacing=40 inch
Corn	USA	4 F	postplant	1 qt/A; 1.1			30 (forage)	no foliar application if soil application with granule formulation 10lbs/A-10 G; 6.7 lbs/A-15 G

Label uses: Crop	Country	Form, type	Application type	Application, kg ai/ha or /kg seed (all metric are g or kg ai)	Volume	Application no.	PHI, days	Remarks
Corn	USA	4 F	foliar	1/2-2 pints/A; 0.28-1.1		2	30 (forage)	1 only if soil application was made.
Cotton	Brazil	350 SC	furrow application at seeding	0.700-1.05 kg/ha	100-300l/ha	1		
Cotton	Brazil	350 ST	Seed treatment	0.7 kg/100 kg seeds		1		
Cotton	Brazil	5 G	in furrow at planting	1.5-3 kg/ha		1		rate depends upon pest type and infestation
Cotton	Bulgaria	10 G		5 kg ai/ha				
Cotton	Bulgaria	350 F		3 kg/100 kg				
Cotton	Bulgaria	350 ST		1.5 kg ai/100kg				
Cotton	China	3 G	in furrow, incorporated	0.675-0.9 kg/ha			60	
Cotton	India	3 G	at planting	1.0 kg/ha				
Cotton	Malaysia	3G	in hole at planting	0.03 g/hole				
Cotton	Spain	5 G		0.6-0.75kgai/ha			60	
Cotton	USA	10 G	in furrow at planting	10 lbs/A; 1.1		1		row spacing = 40 inch
Cotton	USA	4 F	in seed furrow,at planting	1 qt/A; 1.1		1		row spacing = 40 inch
Cranberries	USA (WA)	15 G	soil with rotary spreader	20 lbs/A; 3.4		1	60	incorporate with water sprinkler; do not use with flooding
Cranberries	USA (WA)	10 G	soil with rotary spreader	20 lbs/A; 2.2		2	60	incorporate with water sprinkler; do not use with flooding
Cucurbits	USA	10 G	soil incorporate	20 lbs/A; 2.2				row spacing = 60 inch
Cucurbit	USA	15 G	band application, incorporated	2.245 lbs /1000 linear feet				cucurbit=cucumber, melon, squash and pumpkins; Federal label
Cucurbit	USA	4 F	in furrow	2.4-3.8 oz/1000 linear feet row				
Cucurbit	Cyprus	10 G		1-1.5 kg ai/ha			30	
Cucurbit	Cyprus	5 G		1-1.5 kg ai/ha			30	
Cucurbit	Cyprus	75 WP		1-1.5 kg ai/ha			30	
Grape	USA	4F	broadcast to soil, incoporate	2.5gal/A, 11.2.; or 1.5gal/A chemigation, 6.72		1	##	
Grape	USA	4F	drip irrigation	075gal/A; 3.4		1	60	prohibited after May 1. Limited to 2.2 if a postharvest application was made in previous yr

Label uses: Crop	Country	Form, type	Application type	Application, kg ai/ha or /kg seed (all metric are g or kg ai)	Volume	Application no.	PHI, days	Remarks
Grape	USA	10 G	over soil between vines, incorporate	100 lbs/A; 11.2		1	##	
Maize	Bulgaria	350 ST		875 g ai/100 kg				
Maize	Cyrpus	10 G		5-8 kg ai/ha			30	
Maize	Cyrpus	10 G		5-8 kg ai/ha			30	
Maize	Cyrpus	10 G		5-8 kg ai/ha			30	
Maize	Cyrpus	5 G		5-8 kg ai/ha			30	
Maize	Cyrpus	5 G		5-8 kg ai/ha			30	
Maize	Cyrpus	75 WP		5-8 kg ai/ha			30	
Maize	India	3 G	at plant	1.0 kg/ha				
Maize	Makedonia	5 G		1-1.5 kg ai/ha			-	
Maize	Pakistan	3 G	at sowing, in furrow; at whorl	0.24 kg/A		2		irrigate immediately after application
Maize	Poland	5 G		.75 kg ai/ha				
Maize	Poland	5 G		.75 kg ai/ha				
Maize	Former Youg.	350 F		1.4-2.1kgaih			-	
Oats/barley	Germany	300 SK	at sowing	4.5			##	
Oats/barley	Argentina	35 TS	seed treatment	0.3 kg/100 kg seeds	134.4/l			diluted
Pea	India	3 G	at planting	1.0 kg/ha				
Peanut	Korea	3 G		5		1	55	
Peanuts	Brazil	350 SC	furrow application at planting	1.4-1.75 kg/ha	100-300l/ha			
Peanuts	Brazil	5 G	at planting, incorporation in soil	2 kg/ha		1		
Peanuts	China	3 G	furrow application	1.35-2.35 kg/ha			60	
Peppers	India	3 G	at planting	0.5 kg/ha				
Peppers	USA	4 F		3 qt/A; 3.4		2	21	first application at-plant. Arizona only.
Peppers	USA	10 G	side dressing	20 lbs/A, 30; 2.2, 3.4		2	21	
Potato	Argentina	10 G	in furrow	1.5-2.5 kg/ha				appl. at seeding or after planting
Potato	Brazil	350 SC	in furrow	3.5 kg/ha	200l/ha			
Potato	Brazil	5 G	At planting, incorporated	1.5-4 kg/ha		1		
Potato	Cyprus	10 G		4-8 kg ai/ha			30	
Potato	Cyprus	5 G		4-8 kg ai/ha			30	

Label uses: Crop	Country	Form, type	Application type	Application, kg ai/ha or /kg seed (all metric are g or kg ai)	Volume	Application no.	PHI, days	Remarks
Potato	Cyprus	75 WP		4-8 kg ai/ha			30	
Potato	Cyprus	75 WP		4-8 kg ai/ha			30	
Potato	Cz. Republic	350 F		240-350 g ai/ha			30	
Potato	Egypt	10 G		3.25 kg ai/ha			14	
Potato	India	3 G	at planting	2.6 kg/ha				
Potato	Korea	3 G		5 kg/10 A		1	45	
Potato	Makedonia	5 G		1-1.5 kg ai/ha			-	
Potato	Poland	5 G		2 kg ai/ha				
Potato	USA	4 F	Foliar	2 pts/A; 1.1	10 gal/A ground; 3 gal/A aerial	8	14	3 lbs ai/A/season if at plant application made
Potato	USA	4F	at-planting, in furrow	3 qt/A; 3.4		1		0.225lb/1000 feet of row
Potato	USA	4 F		1-2 pints/A	3 GPA aircraft	8/season		
Rice	USA	3 G	air/ground equipment	20 lb/A; 0.67		1/season	60	1 d before or up to 21 d after permanent flooding. State label Expires 9/97
Rice	USA	5 G	air/ground equipment	10 lbs/A; 0.56		1/season		1 d before or 2 or 21 days after flooding. Expires 9/97.
Rice	USA	5G or 2G	preplant, soil incorported. ground equip	0.56		1		CA only. Expires 8/97
Rice	USA	5G	postplant to soil. ground or aerial	0.56		1	60	CA only
Rice	Argentina	10 G	broadcast	0.75-1 kg/ha				
Rice	Australia	10 G		1 kg/ha		2/season		application at mid=tillerinf and 30-50 d after panicule initiation
Rice	Brazil	310 TS	seed treatment	527 g/100 kg seeds				
Rice	Brazil	350 SC	furrow application	0.700-1.05 kg/ha	400 ml/ha	1		vs irrigation system
Rice	Brazil	350 ST	Seed treatment	0.525 kg/100 kg seeds		1		
Rice	Brazil	350 ST						
Rice	Brazil	5 G		0.75-1 kg/ha		1		irrigated
Rice	China	3 G	broadcast, at seeding, incroporated	0.9-1.35 kg/ha			60	
Rice	India	3 G	at plant	2.0 kg/ha				
Rice	Japan	3G	broadcast	0.9		3	50	
Rice	Korea	3 G		0.09 - 0.12/10a (?)		2	7	2 applications (control 1st and 2nd generation)
Rice	Pakistan	3 G	nursery	0.3 g/m2				2nd application : 5 d before tansplanting
Rice	Pakistan	3 G		240 g/A		2		1st application 25 - 30 days after transplant. 2nd application 50 - 65 days after transplant
Rice	Philippines	3 G	seedbed	90 g/ha			28	
Rice	Thailand	3 G	seedbed	1.9 kg/ha				10 days after seeding
Rye grass	Argentina	35 TS	seed treatment	1.995 kg/100 kg seeds				undiluted

Label uses: Crop	Country	Form, type	Application type	Application, kg ai/ha or /kg seed (all metric are g or kg ai)	Volume	Application no.	PHI, days	Remarks
Rye/wheat	Argentina	35 TS	seed treatment	497 kg/100 kg seeds	39 g/l			diluted
Small grains	USA	4 F	foliar, before head emerges	1/2 pt/A; 0.28		2/season		do not feed forage
Sorghum	India	3 G	at planting	1.5 kg/ha				
Sorghum	Thailand	3 G	at-planting	1.5 kg/ha				
Sorghum	USA	15 G						
Sorghum	USA	10 G	at-planting	10 lbs/A; 1.1		1		row spacing = 40 inch
Sorghum	USA	4 F		1 pint	20 to 30 GBA	2		State label
Soya bean	Argentina	10 G	in furrow at seedong	1.5 kg/ha				
Soya bean	India	3 G	at planting	2.0 kg/ha				
Soya bean	Philippines	3 G		16.7-33.4 kg product/ha				
Soya bean	USA	10 G	at planting, incorporated	20 lbs/A; 2.2		1		row spacing = 40 inch
Soya beans	USA	4 F	at planting	3-4 pints/A; 1.7-2.2				row spacing=40 inch
Soya beans	USA	4 F	foliar appl.	0.5pint; 0.28	1 1/2 gal/A aerial; 20 gal /A ground	2/season	21	no foliar appl if treatment at planting
Strawberry	USA	4F	postharvest soil band. Ground equipment	2.2		1		limited to OR, MI, MN, MO, TN, WA, CT, NH, OH, VA, VT. May not be used after Sept. 1 or Oct. 1,
Sugar cane	Australia	10 G	band application, incorporated	3 kg/ha				application 3-5 leaf stage
Sugar cane	Brazil	350 SC	in furow along planting stick or in bands or streaks in the cane furoow	1.4-1.75 kg/ha	100-300l/ha			
Sugar cane	Brazil	350 SC						
Sugar cane	Brazil	5 G	application around the plant/ in bands or streaks	1.5-3 kg/ha		1		apllication at cane second harvest
Sugar cane	China	3 G	at planting, band application	1.35-2.25 kg/ha			60	
Sugar cane	Pakistan	3 G	at planting/ 30 d. after planting	0.3-0.45 kg/A				
Sugar cane	Philippines	3G	at planting/30 d aftr planting	0.3-0.45 kg/A				2 nd application at earthing
Sugar cane	Philippines	5 G	at planting, in furrow and furrow ridge	1-2 kg/ha				
Sugar cane	Thailand	3 G	at planting, in the rows	1.9 kg.ha				

Label uses: Crop	Country	Form, type	Application type	Application, kg ai/ha or /kg seed (all metric are g or kg ai)	Volume	Application no.	PHI, days	Remarks
Sugar cane	USA	4 F	after joint formation, foliar	1.5 pts/A; 0.84		2	17	do not use in Hawaii
Sugar cane	USA	10 G	over stubble cane, incorporated	40 lbs/A; 4.5		1		row spacing = 60 inch. Do NOT use in Hawaii
Sugar beet	Bulgaria	350 ST		875 g ai/100 kg				
Sugar beet	Hungary	10 G		1.5-2 kgaiha				
Sugar beet	Poland	350 ST		44.8g/100,000 s				
Sugar beet	Poland	480 FS		58 LITRE/TON				
Sugar beet	Russia	350 ST		8-10 kg ai/ton			-	
Sugar beet	Former Youg.	350 F		1.4-2.1kgaih			-	
Sugar beet	USA	10 G	at planting, incorporated	20 lbs/A; 2.2				row spacing = 22 inch
Sugar beets	USA	4 F		1 to 2 quarts/A				State label
Sunflower	Bulgaria	300 COMBI		1 kg ai/100 kg				
Sunflower	Bulgaria	350 ST		1.5 kg ai/100kg				
Sunflower	Hungary	10 G		1.5-2 kgaiha				
Sunflower	USA	4F	soil band or in-furrow at planting. ground equipment	1.4 qt/acre; 1.6		1		row spacing = 30 inch. 0.16 pt/1000 feet of row
Sunflower	USA	4F	foliar, ground or aerial	1 pint/A; 0.56	2 gal/A aerial; 10 gal/A ground	4	28	
Sweet corn	USA	4F	at planting, 7 inch band over the row or inject on each side of row with water	2 pt/A; 1.12			30	Forage may not be fed within 30 days of application. Use 2.5 fl oz/1000 linear feet of row.
Sweet corn	USA	4F	foliar	1 pt/A; 0.56	10 gallons/A ground; 2 gallons/A aerial	4	7	Limited to machine-harvested corn. 1st application just prior to silking, with 7 day repeat interval.
Tea	India	3 G	at planting	300 mg/plant				
Tobacco	Argentina	10 G	broadcast/in furrow application	3.5-6 kg/ha				
Tobacco	Australia	10 G	in furrow, incorporated	3.5 kg/ha				
Tobacco	Australia	10 G						
Tobacco	Brazil	350 SC	at planting	1.4-1.75 kg/ha	100-300l/ha	1		
Tobacco	Brazil	5 G	At planting/transplanting, incorporated	0.75-4 kg/ha		1		for granule manual or mechanical application
Tobacco	India	3 G	at planting	4 kg/ha				
Tobacco	Korea	3 G	planting	50 kg/10 a		1		

Label uses: Crop	Country	Form, type	Application type	Application, kg ai/ha or /kg seed (all metric are g or kg ai)	Volume	Application no.	PHI, days	Remarks
Tobacco	Pakistan	3 G	nursery, broadcast	0.3 kg/A				15 d before transplanting
Tobacco	Thailand	3 G	at-planting	0.06 g/hill				
Tobacco	USA	4F	pre-transplant, soil incorporated	1.5 gal/A;6.7		1		
Tobacco	USA	10 G	pre-transplant, incorporate	60 lbs/A; 6.7				
Tomato	Argentina	10 G	at transplanting, +1 mo transplanting	1.5 kg/ha		2		
Tomato	Argentina	10 G	in seedbed	3 kg/ha		2		at planting and 15 d before transplanting
Tomato	Brazil	350 SC	in furrow	1.75 kg/ha	200 l/ha	1		
Tomato	Brazil	5 G	at planting/transplanting	0.75-4 kg/ha		1		
Tomato	Brazil	5 G		0.15-0.25 g/ha				
Tomato	India	3 G	at planting	2.5 kg/ha				
Tomato	Malaysia	3 G	at planting, in hole, incorporated	0.09 g /hole		1		
Vege-tables	Cyprus	10 G		4-8 kg ai/ha			30	
Vege-tables	Cyprus	5 G		4-8 kg ai/ha			30	
Vege-tables	Cyprus	75 WP		4-8 kg ai/ha			30	
Vege-tables	Former Youg.	350 F		0.7 kg ai/ha				
Vege-tables	Former Youg.	350 F		0.0525 %				
Wheat	Brazil	350 SC	in furrow at seedling	1.05 -1.4 kg/ha	100-300l/ha			
Wheat	Brazil	5 G	At planting, incorporation in soil	0.75 - 1 kg/ha		1		
Wheat	India	3 G	at planting	3.0 kg/ha				
Wheat	USA	4 F	see small grain					
Wheat/ barley	Australia	360 F	in furrow, at seeding	1.1l/ha	10l/ha			

In the Tables of field trials that follow, a uniform procedure was used to treat residues of the two analytes 3-hydroxy-carbofuran and carbofuran limits at the determination and detection, which may be summarized as follows.

V_1	V_2	Total
<LOd	<LOd	LOd

LOd<V1<LOQ	LOd<V_2<LOQ	V_1 V_2
<LOd	>LOD	V_2
<LOd	LOd<V_2<LOD	V_2

V_1, $V_{2:}$ carbofuran or 3-hydroxy-carbofuran
LOd: limit of detection
LOD: limit of determination

Residues below the limit of determination and above the limit of detection are in parentheses.

Furadan 4F was applied as a foliar spray to alfalfa in 28 supervised field trials in the USA (Singer, 1990a). Three applications were made at 1.12 kg ai/ha and at 0.004-0.006 kg/l. Each application was followed by cutting after 28 days. Both green and field-dried samples of alfalfa were analysed for carbofuran and carbamate and phenol metabolites by the method of Schreier. A limit of determination of 0.5 mg/kg was demonstrated for carbofuran, 3-hydroxy-carbofuran, 3-ket°Carbofuran, 7-phenol, 3-hydroxy-7-phenol and 3-keto-7-phenol. The limit of detection was estimated to be 0.1 mg/kg. The results are shown in Table 31. The water contents of the dried alfalfa samples were not determined.

The US GAP conditions are 2 applications, total 1.1 kg ai/ha, with the second application not exceeding 0.56 kg ai/ha. The PHIs are 28 days after a 1.1 kg ai/ha application , 14 days after 0.56 kg ai/ha, and 7 days after 0.28 kg ai/ha.

Table 31. Residues of carbofuran and its metabolites in or on green and dried alfalfa cut 28 days after foliar applications of carbofuran at 1.12 kg ai/ha in the USA.

Location, Year	No. appl[1]	Sample	Carbofuran	Residues, mg/kg						
				3-OH-CF	3-K-CF	Total carb-amates	7-Phenol	3-K-7-P	3-OH-7-P	Total phenols
California, 1986	1	green forage	(0.35)	1.3	<0.1	1.7	(0.17)	0.54	(0.25)	0.96
				Residues, mg/kg						
	1	dried fodder	1.4	6.2	(0.15)	7.6	0.60	1.3	0.90	2.8
	2	green	(0.17)	1.4	<0.1	1.6	(0.15)	0.50	(0.20)	0.85
	2	dried	(0.38)	4.3	<0.1	4.7	0.45	1.2	0.76	2.4
	3	green	(0.10)	1.2	<0.1	1.3	(0.19)	0.68	0.46	1.3
	3	dried	(0.24)	2.8	<0.1	3.0	(0.30)	0.65	0.58	1.5
Pennsylvania, 1987	1	green	<0.1	<0.1	<0.1	<0.1	<0.1	(0.13)	<0.1	(0.13)
	1	dried	<0.1	1.2	<0.1	1.2	<0.1	(0.25)	(0.19)	(0.44)
	2	green	<0.1	0.52	<0.1	0.52	<0.1	<0.1	<0.1	<0.1
	2	dried	<0.1	0.90	<0.1	0.90	<0.1	(0.42)	(0.24)	0.66
	3	green	<0.1	1.2	<0.1	1.2	<0.1	(0.34)	(0.25)	0.59
	3	dried	<0.1	1.4	(0.12)	1.4	<0.1	0.99	0.58	1.6
Ohio, 1987	1	green	<0.1	1.6	<0.1	1.6	(0.14)	0.52	(0.32)	0.98
	1	dried	<0.1	4.5	<0.1	4.5	(0.41)	1.1	1.2	2.7
	2	green	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
	2	dried	<0.1	(0.32)	<0.1	(0.32)	<0.1	(0.22)	<0.1	(0.22)
	3	green	<0.5	(0.13)	<0.5	(0.13)	<0.1	<0.1	<0.1	(0.13)
	3	dried	<0.1	(0.28)	<0.1	(0.28)	<0.1	<0.1	<0.1	<0.5
California, 1988	1	green	<0.1	1.8	<0.1	1.8				
	1	dried	(0.41)	2.4	(0.34)	2.8	0.65	1.9	1.2	3.8
	2	green	<0.1	0.92	<0.1	0.92	<0.1	0.55	0.32	0.87
	2	dried	(0.37)	3.0	(0.23)	3.4	0.55	2.0	1.8	4.4
	3	green	(0.34)	1.9	<0.1	2.2	(0.28)	0.62	0.52	1.4
	3	dried	(0.40)	2.2	<0.1	2.6	0.64	1.2	1.3	3.1
Pennsylvania, 1987	1	green	<0.1	1.4	<0.1	1.4	(0.12)	0.50	0.62	1.2

Location, Year	No. appl[1]	Sample	Carbofuran	Residues, mg/kg						
				3-OH-CF	3-K-CF	Total carb-amates	7-Phenol	3-K-7-P	3-OH-7-P	Total phenols
California, 1986	1	green forage	(0.35)	1.3	<0.1	1.7	(0.17)	0.54	(0.25)	0.96
	1	dried	<0.1	5.2	<0.1	5.2	0.55	1.4	0.56	2.5
	2	green	<0.1	1.2	<0.1	1.2	<0.1	(0.46)	<0.1	(0.46)
	2	dried	<0.1	3.8	<0.1	3.8	0.36	1.0	0.72	2.1
	3	green	<0.1	4.3	0.90	4.3	<0.1	1.4	(0.30)	1.7
	3	dried	<0.1	4.2	<0.1	4.2				
Wisconsin,1986	1	green	<0.1	(0.29)	<0.1	(0.29)	<0.1	<0.1	<0.29	<0.5
	1	dried	<0.1	0.90	<0.1	0.90	(0.17)	(0.30)	(0.26)	0.73
	2	green	<0.1	1.2	<0.1	1.2	<0.1	0.13	(0.32)	.(0.45)
	2	dried	<0.1	4.6	(0.28)	4.6	(0.45)	0.96	1.6	3.0
	3	green					<0.1	<0.1	<0.1	<0.1
	3	dried					<0.1	0.59	(0.27)	0.86
Minnesota, 1985	1	green	<0.1	(0.34)	<0.1	(0.34)	<0.1	<0.1	<0.1	<0.1
	1	dried	<0.1	0.64	<0.1	0.64	<0.1	(0.22)	(0.16)	(0.38)
	2	green	<0.1	<0.1	<0.1	≤0.1	<0.1	<0.1	<0.1	<0.1
	2	dried	<0.1	<0.1	<0.1	≤0.1	<0.1	<0.1	<0.1	<0.1
	3	green					<0.1	<0.1	<0.1	<0.1
	3	dried					<0.1	(0.21)	<0.1	(0.21)
Iowa, 1984	1	green	<0.1	(0.38)	<0.1	(0.38)	<0.1	<0.1	<0.1	<0.1
	1	dried	<0.1	0.74	<0.1	0.74	<0.1	(0.23)	<0.1	(0.23)
	2	green	<0.1	<0.1	<0.1	≤0.1	<0.1	<0.1	<0.1	<0.1
	2	dried	<0.1	<0.1	<0.1	≤0.1	<0.1	<0.1	<0.1	<0.1
	3	green	<0.1	<0.1	<0.1	≤0.1				
	3	dried	(0.30)	0.57	<0.1	0.87	<0.1	(0.18)	<0.1	0.18
Nebraska,1986	1	green	<0.1	0.94	<0.1	0.94	<0.1	(0.36)	(0.37)	0.74
	1	dried	<0.1	1.6	<0.1	1.6	(0.38)	0.90	(0.41)	1.7
	2	green	<0.1	(0.30)	<0.1	(0.30)	<0.1	<0.1	<0.1	<0.1

Location, Year	No. appl[1]	Sample	Carbofuran	Residues, mg/kg						
				3-OH-CF	3-K-CF	Total carb-amates	7-Phenol	3-K-7-P	3-OH-7-P	Total phenols
California, 1986	1	green forage	(0.35)	1.3	<0.1	1.7	(0.17)	0.54	(0.25)	0.96
	2	dried	<0.1	1.2	(0.28)	1.2	(0.28)	(0.48)	(0.32)	1.1

Abbreviated compound names: see Figure 1
Limits of detection, determination 0.1 , 0.5 mg/kg for each analyte
[1]The alfalfa was cut 28 days after each application and before the subsequent application

In a separate trial in the USA (Leppert, 1986a), alfalfa in California was treated twice at rates of 0.56 and 0.28 kg ai/acre with Furadan 4F applied as an aerial foliar spray. The PHI was 4 days. Samples of green hay, field-dried hay, meal and finished meal pellets were prepared and analysed by the method of Schreier. Limits of determination were established by determination of recoveries from fortified control samples. The recoveries listed in Table 32 were reported and some supporting chromatograms were included. The trial did not comply with GAP because of the 4-day PHI.

Table 32. Recovery of carbofuran and metabolites from alfalfa by the Schreier Method.

Sample	Spike, mg/kg	Recovery, %					
		Carbofuran	3-OH-CF	3-K-CF	7-Phenol	3-OH-7-P	3-K-7-P
Green hay	0.05			104			
	0.5			69	98	94	102
	2.5	87	105				
Cured hay	0.5			104			
	1.0				71	72	88
	2.5	78	93	109			
Meal	0.2	95	110	90			
	1.0				64	79	83
Pellet	0.2			85			
	1.0				82	87	110
	5.0	85	94	77			

Abbreviated compound names: see Figure 1

Table 33. Residues of carbofuran and metabolites in or on alfalfa harvested 4 days after two applications of Furadan 4F (0.56 and 0.28 kg ai/ha).

Sample	Residue, mg/kg					
	Carbofuran	3-OH-CF	3-K-CF	7-Phenol	3-OH-7-P	3-K-7-P
Green hay	3.6	2.7	0.26	0.31	0.22	0.77
	4.3	4.9	0.63	0.46	0.36	1.4
	3.1	2.4	0.40	0.52	0.45	1.6
Cured hay[1]	19	9.0	0.54	2.2	1.3	1.3
	15	8.9	0.29	1.8	1.2	1.6
	19	8.5	0.62	2.4	2.0	2.2
Meal	18	5.2	0.12	2.9	1.7	1.7
	16	4.5	<0.20	3.8	2.0	1.7
Pellets	14	4.8	0.21	2.8	1.3	1.4
	16	4.8	0.20	2.6	1.3	1.3

Abbreviated compound names: see Figure 1 [1]Moisture content not reported.

Maize. Field trial results were reported from Brazil (Sao Paulo University, 1994), France (Mollhoff, 1974), Germany (Mollhoff, 1974) and the USA (Brooks, 1995; Singer, 1990b). The trials represent various combinations of at planting plus foliar treatments and the findings are shown in Table 34. The reports from Brazil, France and Germany consisted of brief summaries and provided inadequate details. They were not suitable for use in estimating maximum residue levels.

The US trials were of two types: an in-furrow application of a 15G formulation at 1.5 kg ai/ha followed by a foliar whorl application of 15G at 1.1 kg ai/ha, and one or two foliar applications of a 4F formulation at 1.1 kg ai/ha. The GAP label conditions specify a soil band, in-furrow, or injection at planting of the F (not G) formulation at 1.12 kg ai/ha (0.090 kg ai/2.54 m row) with a PHI of 30 days for feeding forage, which may be followed by a soil band, side-dress, or basal spray of the F formulation at 1.12 kg ai/ha (2.24 kg ai/ha in South Carolina) with a 30-day restriction on feeding forage, but no other PHI and no limit to the number of treatments. Additionally, two foliar applications may be made at 1.12 kg ai/ha each, with a 30-day PHI, using ground or aerial equipment. The US trials exceed the initial GAP at planting rate by 34% and use an F formulation, which place the trials on the fringe of acceptability. The use of an F or G formulation appears to have no effect on the residue concentrations (see the trials on sweet corn). Only two trials (in Missouri) are within the GAP window.

The other (5) US trials consisted of foliar spray applications of an F formulation at 1.1 kg ai/ha, with PHIs of 102-145 days, far in excess of the 30-day GAP interval. The data could not be evaluated.

Table 34. Total residues of carbamates and phenols in or on maize treated with carbofuran.

Country, Year	Form.	Application		PHI, days	Residue, mg/kg		Method of analysis
		Method/ timing	kg ai/ha		Carbamates[1]	Phenols[2]	
Brazil 1994	Furadan 350 SC	foliar spray	1.4	30	<0.05 (grain)		Leppert
			2.8	30	<0.05		
Brazil 1993	Furadan 350 TS	seed treatment	1.05 kg ai/100 kg	159	<0.1 (grain)		Leppert
			2.1 kg ai/100 kg	159	<0.1 (grain)		
Brazil 1993	Furadan 50G	in-furrow at-plant	1.75	30[3]	<0.1		Leppert
			3.5	30[3]	<0.1		Leppert
France 1973	Curraterr 5G	in-furrow at sowing	0.60	122	<0.1 (cob with grain)		Mollhoff
				163	<0.1 (grain)		
			1.0	115	<0.1 (grain)		
Germany 1973	Curraterr 5G	in-furrow at sowing	1.0	115	<0.1 (grain)		Mollhoff
Germany 1976	Curraterr 5G	in-furrow at sowing	0.94	125 153	0.7, 0.6, 0.8 (silage) <0.1, <0.1, <0.1 (grain) 0.4, 0.3, 0.9 (fodder)		Mollhoff
Germany 1975	Curraterr 5G	in-furrow at sowing	0.50	123 143	0.1, 0.3, 0.2 (silage) <0.1, <0.1, <0.1 (grain)		Mollhoff
Germany 1985	Curraterr 5G	in-furrow at sowing	0.62	105	<0.1 (cob at milk to dough		Mollhoff

Country, Year	Form.	Application		PHI, days	Residue, mg/kg		Method of analysis
		Method/ timing	kg ai/ha		Carbamates[1]	Phenols[2]	
					stage)		
Germany 1974	Curraterr 5G	in-furrow at sowing	1.0	138	<0.1 (cob) <0.1 (grain)		Mollhoff
USA (OH) 1988	Furadan 15G	in-furrow at planting	1.5				
	Furadan 15G	foliar whorl	1.1				
	Furadan 4F	foliar spray	1.1	69	1.5, <0.1[4] (fodder)	0.62, <0.5[4] (fodder)	Schreier
USA (NC) 1988	Furadan 15G	in-furrow at planting	1.5				
	Furadan 15G	foliar whorl	1.1				
	Furadan 4F	foliar spray	2 x 1.1	65	<0.1 (fodder)	0.70, 0.67 (fodder)	
USA (MO) 1988	Furadan 15G	in-furrow at planting	1.5				
	Furadan 15G	foliar whorl	1.1				
	Furadan 4F	foliar spray	1.1	32	1.1, 1.0 (silage)		
	Furadan 4F	foliar spray	1.1	21	<1.0, 1.2 (silage)		
USA (MN) 1988	Furadan 15G	in-furrow at planting	1.5				
	Furadan 15G	foliar whorl	1.1				
	Furadan 4F	foliar spray	1.1	80	1.5 (silage)	<1.0 (silage)	
	Furadan 4F	foliar spray	1.1	55	1.3 (silage)	<1.0 (silage)	
USA (CA) 1988	Furadan 15G	in-furrow at planting	1.5				
	Furadan 15G	foliar whorl	1.1				
	Furadan 4F	foliar spray	1.1	63	2.0 , 2.2 (fodder)	0.80, 0.66 (fodder)	
	Furadan 4F	foliar spray	1.1	63	2.4, 3.1 (fodder)	0.80, 1.4 (fodder)	
USA (IA) 1994	Furadan 4F	foliar spray	1.1	120	<0.03 (grain) <0.1 (fodder)	<0.03 (grain) <0.50 (fodder)	Schreier
USA (IL) 1994	Furadan 4F	foliar spray	1.1	111	<0.03 (grain) <0.10 (fodder)	<0.03 (grain) <0.50 (fodder)	
USA (NE)	Furadan 4F	foliar spray	1.1	102	<0.03 (grain) <0.10 (fodder)	<0.03 (grain) <0.50 (fodder)	
USA (MN) 1994	Furadan 4F	foliar spray	1.1	143	<0.03 (grain) <0.10 (fodder)	<0.03 (grain) <0.10 (fodder)	
USA (IN)	Furadan 4F	foliar spray	1.1	125	<0.03 (grain) <0.10 (fodder)	<0.03 (grain) <0.50 (fodder)	
USA (OH) 1994	Furadan 4F	foliar spray	1.1	124	<0.03 (grain) <0.10 (fodder)	<0.03 (grain) <0.50 (fodder)	

[1]Carbofuran + 3-keto-carbofuran + 3-hydroxy-carbofuran
[2]7-phenol + 3-hydroxy-7-phenol
[3]30-day period from seed planting to mature crop
[4]Duplicate samples from same plot

Sweet corn (corn-on-the-cob). Field trials in the USA were reported by Martin (1986b, 1987). Thailand reported the GAP conditions used in field trials, but not the results (Thai Industrial Standards Institute, 1997). In 16 side-by-side trials in eight states of the USA, sweet corn was treated at planting with either Furadan granular (15G, 10 G in California) or Furadan flowable, at 3.4 kg ai/ha. Whorl applications were made with 15G (10 G in California) at 1.1 kg ai/ha 3-6 weeks after planting. Four additional foliar applications of the flowable formulation were made at 0.56 kg ai/ha over a period of 2 to 7 weeks. The total seasonal application was 6.7 kg ai/ha. Ears and husks were harvested 0 and 7 days, and stalk (forage) samples 21 days, after the final treatment. The samples were analysed by the method of Schreier. Carbamates were measured by GC/NPD and phenols were measured by GC-MSD. Limits of determination of 0.5 and 0.03 mg/kg were demonstrated for each analyte in forage and corn ears respectively, with corresponding limits of detection of 0.01 mg/kg. The recoveries reported from stalks at a 0.5 mg/kg fortification (n = 13) were carbofuran 84 ± 13%, 3-keto-carbofuran 100 ± 18%, 3-hydroxy-carbofuran 87 ± 20%, 7-phenol 78 ± 14%, the 3-keto-7-phenol 80 ± 17%, 3-hydroxy-7-phenol 82 ± 19%. The recoveries from corn-on-the-cob at 0.03 mg/kg (n = 4 for carbamates, 5 for phenols) were carbofuran 79 ± 10%, 3-keto-carbofuran 84 ± 22%, 3-hydroxy-carbofuran 85 ± 13%, 7-phenol 77 ± 16%, the 3-keto-7-phenol 94 ± 7%, and 3-hydroxy-7-phenol 86 ± 14%. The limit of determination demonstrated by the analysis of fortified control husk samples was 0.5 mg/kg (n = 7 for carbamates, 6 for phenols). Recoveries were carbofuran 82 ± 20%, 3-keto-carbofuran 91 ± 12%, 3-hydroxy-carbofuran 86 ± 16%, 7-phenol 74 ± 5%, 3-keto-7-phenol 102 ± 17%, and 3-hydroxy-7-phenol 84 ± 13%.

The total carbamate residues in corn-on-the-cob 0 and 7 days after the final application were <0.03 (6), 0.03 (4), 0.04 (4), 0.05 and 0.08 mg/kg. The total phenol residues were ≤0.10 mg/kg. No single carbamate or phenol predominated and there were no differences between the residues from the two treatment programmes.

The residues found in the stalks (forage) and husks are shown in Table 35. Again there were no differences between the residues from the flowable and granular formulations. Sweet corn forage is not an animal feed item, although maize forage is.

US GAP for sweet corn permits application at planting of 1.12 kg ai/ha (90 g/2.54 m of row). Forage may not be fed within 30 days of treatment. Additionally, the F formulation may be applied at 0.56 kg/ai ha, maximum 4 applications per season, with a 7-day PHI. These treatments may not be made if the at planting application exceeded 1.12 kg ai/ha. There is a 21-day restriction on harvesting or feeding stalks.

All trials exceeded the maximum at planting application rate by 240%. The data were not acceptable for the estimation of maximum residue levels.

Thailand reported trials according to GAP, but without data on residues (Thai Industrial Standards Institute, 1977).

Table 35. Residues in or on sweet corn stalks and husks from the application of carbofuran, at planting and foliar, 6.7 kg ai/ha total rate, USA, 1985.

Location	PHI, days	Residue, mg/kg					
		Carbofuran	3-K-CF	3-OH-CF	7-Phenol	3-K-7-P	3-OH-7-P
Harvest stalks (forage)							
3.4 kg ai/ha 15G at planting, 1.2 kg ai/ha 15G at whorl, 4 x 0.56 kg ai/ha 4F foliar							
Arkansas	0	8.7	<0.1	(0.3)	0.64	(0.26)	(0.24)
	7	(0.20)	<0.1	(0.39)	(0.20)	(0.24)	(0.29)
	14	<0.1	<0.1	(0.20)	(0.29)	(0.32)	(0.38)
	21	<0.1	<0.1	(0.28)	0.50	<0.1 (0.44)	0.94
California	21	<0.1	<0.1	<0.1	0.70	<0.1	<0.1
Florida	21	<0.1	<0.1	(0.028)	<0.1	<0.1	<0.1
Iowa	21	1.2	<0.1	1.3	1.6	1.3	1.2
Illinois	0	13	<0.1	1.2	1.5	0.74	0.90
	7	<0.1	<0.1	1.5	0.46	1.0	1.2
	14	<0.1	<0.1	1.2	(0.17)	(0.22)	0.50

Location	PHI, days	Residue, mg/kg					
		Carbofuran	3-K-CF	3-OH-CF	7-Phenol	3-K-7-P	3-OH-7-P
	21	<0.1	<0.1	1.2	<0.1	<0.1	<0.1
New York	0	4.7	<0.1	0.90	0.62	(0.26)	(0.40)
	7	3.4	<0.1	1.3	0.60	(0.30)	0.52
	14	(0.38)	<0.1	0.52	(0.20)	(0.150	(0.25)
	21	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Oregon	21	<0.1	<0.1	0.88	(0.30)	0.50	0.56
Wisconsin	0	25 (5.2; 45)	<0.1	1.8	1.8	<0.1	<0.1
	7	0.56	<0.1	0.50	(0.20)	(0.20)	(0.21)
	14	<0.1	<0.1	0.98	(0.24)	(0.28)	(0.31)
	21	<0.1	<0.1	0.45	<0.1	(0.16)	(0.18)
4 kg ai/ha 4F at-3.plant, 1.2 kg ai/ha 15G at whorl, 4 x 0.56 kg ai/ha 4F foliar							
Arkansas	0	8.3	<0.1	0.62	0.82	0.62	0.63
	7	(0.25)	<0.1	0.60	0.54	0.88	0.76
	14	(0.16)	<0.1	0.86	0.52	0.68	1.0
	21	<0.1	<0.1	0.64	(0.38)	0.71	0.91
California	21	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Florida	21	<0.1	<0.1	0.54	<0.1	<0.1	(0.20)
Iowa	21	(0.48)	<0.1	1.7	1.9	0.93	1.2
Illinois	0	15	<0.1	1.6	1.1	(0.34)	0.52
	7	(0.15)	<0.5	1.4	(0.26)	(0.40)	0.63
	14	<0.1	<0.1	1.2	(0.29)	0.69	0.56
	21	<0.1	<0.1	1.2	<0.1 (0.32)	0.62	0.75
New York	0	4.1 (2.6; 5.7)	<0.1	0.92	0.68	<0.1	(0.32)
	7	1.5	<0.1	0.95	(0.28)	(0.20)	(0.30)
	14	(0.28)	<0.1	(0.32)	<0.1	<0.1	<0.1
	21	(0.15)	<0.1	(0.29)	<0.1	<0.1	<0.1
Oregon	21	<0.1	<0.1	0.67	(0.16)	(0.38)	0.63
Wisconsin	0	17	<0.1	(0.28)	0.90	<0.1	<0.1
	7	0.92	<0.1	0.64	(0.26)	(0.32)	0.59
	14	<0.1	<0.1	1.4	<0.1	<0.1	<0.1
	21	<0.1	<0.1	0.64	<0.1	(0.23)	<0.1
Sweet corn husks							
3.4 kg ai/ha 15G at planting, 1.2 kg ai/ha 15G at whorl, 4 x 0.56 kg ai/ha 4F foliar							
Arkansas	0	3.7	<0.5	<0.5	(0.27)		
	7	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
California	7	0.94 (0.181; 1.71)	<0.1	<0.1	<0.1	<0.1	<0.1
Iowa	7	2.1	<0.1	<0.1	0.78	<0.1	<0.1
Illinois	0	0.83	<0.1	<0.1	(0.14)	<0.1	<0.1
	7	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
New York	0	0.76	<0.1	<0.1	<0.1	<0.1	<0.1
	7	0.85	<0.1	(0.28)	<0.1	<0.1	<0.1
Oregon	7	(0.16)	<0.	(0.30)	<0.1	<0.1	(0.16)
Wisconsin	0	2.4	<0.1	(0.14)	(0.22)	<0.1	<0.1
	7	(0.18)	<0.1	<0.1	<0.1	<0.1	<0.1
3.4 kg ai/ha 4F at planting, 1.2 kg ai/ha 15G at whorl, 4 x 0.56 kg ai/ha 4F foliar							
Arkansas	0	1.4	<0.1	(0.0.20)	(0.19)	<0.1	<0.1
	7	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
California	7	0.68	<0.1	<0.1	(0.24)	<0.1	<0.1
Iowa	7	4.9	<0.1	(0.46)	<0.1	<0.1	<0.1
Illinois	0	1.4	<0.1	<0.1	(0.18)	<0.1	<0.1
	7	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
New York	0	0.96	<0.1	<0.1	<0.1	<0.1	<0.1

Location	PHI, days	Residue, mg/kg					
		Carbofuran	3-K-CF	3-OH-CF	7-Phenol	3-K-7-P	3-OH-7-P
	7	(0.37)	<0.1	(0.26)	(0.18)	<0.1	<0.1
Oregon	7	<0.1	<0.1	(0.18)	<0.1	<0.1	<0.1
Wisconsin	0	2.1	<0.1	(0.18)	(0.22)	<0.1	<0.1

Abbreviated compound names: see Figure 1

Oats. Carbofuran (Curraterr 300 SK) was applied at sowing to oats seeds at the rate of 4.5 kg/ha in 1975 in Germany. No residues were found in the grain at the claimed limit of detection (0.10 mg/kg) when the grain was harvested 112 days after treatment. The straw contained 1.4, 0.7 and 1.0 mg/kg total carbamates. Three varieties (Flamingskron, Luxor, Tiger) were planted in single plots. The method of Mollhoff was utilized to analyse the grain for carbofuran and 3-hydroxy-carbofuran. Only a summary of the studies was submitted, without adequate detail to validate the reported results. The results could not be used to estimate maximum residue levels, and three trials are insufficient, unless supported by data on other small grains.

Rice. Field trial reports on the use of carbofuran on rice were submitted from Australia, Brazil, Japan, the Philippines and the USA. Thailand submitted information on GAP field trials, but no report of the residues found (Thai Industrial Standards Institute, 1997). The results are shown in Table 36. The results from Brazil were reported as summaries with no detail and were not suitable for use in estimating maximum residue levels.

According to information supplied by the sponsors, GAP treatment in the Philippines is with 90 g/ha of a 3 G formulation, with a 28-day PHI. GAP in Brazil allows seed treatment with 350 ST at 0.525 kg/100 kg seed and at planting furrow application of 0.70-1.05 kg ai/ha by irrigation. Australian GAP specifies 2 x 1 kg ai/ha of a 10 G formulation, with the final application 30-50 days after panicule initiation. There is no PHI. US GAP specifies a pre-plant soil incorporation (before flooding) of a 2G or 5G formulation, at 0.56 kg ai/ha (California only). An additional 0.56 kg ai/ha may be applied after planting, with a 60-day PHI. Outside California a 3% G formulation may be applied after flooding to the water at 0.67 kg ai/ha, or the 5% G formulation may be applied at 0.56 kg ai/ha. A PHI is not specified. Most US uses are temporary. Japanese GAP was not available, but that for China specifies 1.35 kg ai/ha of a 3 G formulation broadcast at seeding, with a 60-day PHI.

The US trials were at twice the GAP application rate. The Japanese trials did not compare well with Chinese GAP: the single application rate was 67% of that specified and/or the PHI was 20-40 days (67%) longer. The Philippine trials were at 33 times the GAP application rate and the PHI was excessive, 49-62 days compared with the specified 28 days.

In one of the three Australian trials, the 2 x 1 kg ai/ha GAP treatment is approximated by a single application of 2 kg ai/ha, with a 58-day PHI.

One trial is insufficient for the estimation of a maximum residue level.

Table 36. Total residues of carbamates and phenolic metabolites from the application of carbofuran to rice.

f	Form./ Appln. method	Rate		PHI, days	Residue, mg/kg		Analytical method & LOD, mg/kg	Ref.
		kg ai/ha	kg ai/hl		Carbamates	Phenol		

f	Form./ Appln. method	Rate		PHI, days	Residue, mg/kg		Analytical method & LOD, mg/kg	Ref.
		kg ai/ha	kg ai/hl		Carbamates	Phenol		
Australia (Queensland), 1982/ Starbonnet	Furadan 10G/Broad-cast	2.0	-	58	≤0.05 (grain) 0.58 (hulls)		Mollhoff 0.05	Stearns,1982
Australia (Queensland), 1982/ Starbonnet	Furadan 10G/Broad-cast	2 x 0.5	-	57	<0.02 (grain) <0.02 (hulls)		Mollhoff 0.05	Stearns,1982
Australia (Queensland), 1982/ Starbonnet	Furadan 10G/Broad-cast	1.0	-	95	<0.02 (grain) <0.02 (hulls)		Mollhoff 0.05	Stearns,1982
Brazil, 1994/ BR-IRGA 409	Furadan 50G/Broad-cast	1	-	30	<0.02 (grain)		Leppert 0.05[1]	Sao Paolo U., 1994
Brazil, 1994/ BR-IRGA 409	Furadan 50G/Broad-cast	2	-	30	<0.02 (grain)		Leppert 0.05[1]	
Brazil, 1993/ Araguaia	Furadan 350 SC/ pulveri-zation in furrow at planting[2]	1.05	Not spec.	156	<0.05 (grain)		Leppert 0.1[1]	
Brazil, 1993/ Araguaia	Furadan 350 SC/ pulveri-zation in furrow at planting[2]	2.1	Not spec.	156	<0.05 (grain)		Leppert 0.1[1]	
Japan, 1974/ Honenwase	Curraterr 3G/ Broadcast	0.9	-	101	<0.01[3] (grain)		GLC (EC) (no detail) 0.01	Mollhoff,1974
		2 x 0.9	-	82	<0.01[3] (grain) <0.02[3] (straw)		GLC (EC) 0.01 0.02	Mollhoff,1974
		3 x 0.9	-	52	<0.01[3] (grain) <0.02[3] (straw)		GLC (EC) 0.01 0.02	Mollhoff. 1974
Japan, 1974/ Harebarc	Curraterr 3G/ Broadcast	0.9	-	110	<0.01[1] (grain) <0.02[4] (straw)		GLC (EC) 0.01 0.02	Mollhof, 1974
		2 x 0.9	-	95	<0.01 (grain) <0.02[4] (straw)		GLC (EC) 0.01 0.02	Mollhoff, 1974
		3 x 0.9	-	43	(0.01)[4] (grain) (0.04))[4] (straw)		GLC (EC) 0.01 0.02	Mollhoff, 1974
Philippines, 1971/ Miracle IR-8	Curraterr 3G/ Broadcast	0.99	-	62	<0.1 (grain)		Mollhoff 0.1[1]	Mollhoff, 1972
		2 x 1.5	-	60	<0.1 (grain)		Mollhoff 0.1[1]	Mollhoff, 1972
		2 x 1.5	-	49	<0.1 (grain)		Mollhoff 0.1[1]	Mollhoff, 1972
		2 x 1.5	-	62	<0.1 (grain)		Mollhoff 0.1[1]	Mollhoff, 1972
USA (Arkansas),	Furadan 4F/	1.1	0.012	117	<0.02	<0.02	Barros	Shevchuk,

f	Form./ Appln. method	Rate		PHI, days	Residue, mg/kg		Analytical method & LOD, mg/kg	Ref.
		kg ai/ha	kg ai/hl		Carbamates	Phenol		
1992/Katy	Broadcast spray, pre-flood				(grain) <0.02 (straw)	(grain) <0.02 (straw)	0.05	1993a
USA (California), 1992/M-201	Furadan 4F/ Broadcast spray, 5 days preplant	1.1	0.012	151	<0.02 (grain) <0.02 (straw	<0.02 (grain) 0.20 (straw)	Barros 0.05	Shevchuk, 1993a
USA/ (California)/M-201	Furadan 4F/ Broadcast spray, 1 day preplant	1.1	0.006	128	<0.02 (grain) 0.07 (straw)	<0.02 (grain) 0.98[5] (straw)	Barros 0.05	Shevchuk, 1993a
USA (Louisiana), 1992/ Lemont	Furadan 4F Broadcast spray, pre-flood	1.1	0.012	96	<0.02 (grain) 0.02 (straw)	0.13 (grain) 0.32	Barros 0.05	Shevchuk, 1993a
USA (Texas), 1992/ Gulfmont	Furadan 4F Broadcast spray, pre-flood.	1.1	0.008	72	<0.02 (grain) 0.06 (straw)	0.08 (grain) 0.30[6] (straw)	Barros 0.05	Shevchuk, 1993a

[1]No data were provided to validate the claimed limit of determination
[2]The method of application is not consistent with the formulation
[3]Limits of determination of 0.01 and 0.02 mg/kg for grain and straw respectively are claimed, but recovery is not reported below 0.2 mg/kg
[4]Limits of determination of 0.01 and 0.02 mg/kg for grain and straw respectively are claimed, but recovery is not reported below 0.05 mg/kg
[5]74% 7-phenol
[6]About 50% 7-phenol and 50% 3-hydroxy-7-phenol

Sorghum. Six trials were in the USA, where one application at planting was followed by two foliar applications (Shevchuk, 1994a). Adequate recoveries were demonstrated at 0.03 mg/kg from grain and at 0.1 mg/kg from all substrates. At a single location in India two varieties of sorghum seed were treated with carbofuran and in a separate trial the soil was treated after planting (Rallies, 1981). The results of the trials are shown in Table 37.

Information on GAP was not available for India or a neighbouring nation, so the data from India could not be evaluated. In the USA, GAP conditions include soil-band, in-furrow, or injection application at planting of the F formulation at 1.12 kg ai/ha (1.12 kg ai/3960 m row) in Arizona, Louisiana, Mississippi and Texas. An in-furrow application at 2.8 kg ai/ha may be made in Kansa, Missouri and Nebraska, when grazing or cutting for silage or forage within 75 days of planting is prohibited. Additionally, 2 applications of 0.56 kg ai/ha may be made as a post-emergence foliar directed spray before the head emerges from the boot. Grazing treated fields is prohibited (Louisiana), or there is a 30- or 75-day restriction (Kansa, Nebraska, Mississippi and Texas).

Table 37. Total residues of carbofuran and 3-hydroxy-carbofuran in or on sorghum.

Country, Year	Form.	Application		PHI, days	Residue, mg/kg[1]	Analytical method
		Method, timing	kg ai/ha			
India 1981	Furadan 40F	to seed, 4 days before planting	2.5% 5.0% 10% (w/w)	45	0.14 0.18 0.27 (forage)	Cook (colorime-tric)[3]
			2.5 5.0	62	0.076 0.12	

Country, Year	Form.	Application		PHI, days	Residue, mg/kg[1]	Analytical method
		Method, timing	kg ai/ha			
			10% (w/w)		0.18 (forage)	
			2.5 5.0 10% (w/w)	76	0.048 0.068 0.072 (forage)	
	Furadan 3G	soil incorporation 2 days after planting	1.0	45	0.14 (forage)	
				62	0.086 (forage)	
				76	0.046 (forage)	
USA 1993	Furadan 4F	in-furrow at planting	1.1	44 Texas 40 Kansas 39 Nebraska 37 Missouri 37 Oklahoma 60 S Dakota	1.2 <0.05 <0.05 0.11 0.13 <0.05 (forage)	Barros
	Furadan 4F	foliar	2 x 0.6[3]	29 Texas 57 Kansas 53 Nebraska 58 Missouri 21 Oklahoma 39 S Dakota	0.06 <0.05 <0.05 <0.05 0.26 0.11 (forage)	Barros
				63 Texas 79 Kansas 69 Nebraska 80 Missouri 59 Oklahoma 91 S. Dakota	(0.06) (fodder) <0.01 (grain) <0.10 (fodder) <0.01 (grain) (0.07) (forage) <0.01 (grain) <0.10 (fodder) <0.01 (grain) 0.20 (fodder) <0.01 (grain) 0.19 (forage)	Barros

[1]Carbofuran + 3-keto-carbofuran + 3-hydroxy-carbofuran
[2]No validation or limit of determination data were presented
[3]The two foliar applications are in addition to the one at-plant application at 1.1 kg ai/ha

Wheat. Supervised field trial results were reported from South Africa and the USA.

In the South African trials (Anon., 1985a) Curraterr 10G or Curraterr 9G (Curraterr 7% + Volaton 2%) were applied to the soil at the time of planting. The application rates were given as 0.03-0.06 g ai/linear metre. Grain samples (180 days PHI) were analysed for carbofuran and 3-hydroxy-carbofuran by an HPLC method. Neither was found at the stated limit of determination, 0.05 mg/kg. The limit of detection was not stated nor were any sample chromatograms supplied. Was not reported for South Africa or neighbouring countries, so the data could not be evaluated. The South African submission was rudimentary and did not contain necessary details.

The US trials (Stearns, 1986a) were conducted at six locations (South Dakota, Colorado, Oregon, Illinois, Washington and Arizona). Two foliar applications were made, one pre-boot and the second 21 days before harvest. Both were with Furadan 4F at 0.28 kg ai/ha. The volume of spray applied per ha was not stated. The mature grain samples were analysed for carbamates and phenolic metabolites by GC-MS (Schreier, 1989a). A limit of determination of 0.05 mg/kg was demonstrated for carbofuran and each of the metabolites. The limit of detection was estimated as 0.02 mg/kg. The 3-hydroxy-7-phenol was found in two trials (Colorado 0.05 mg/kg; Arizona 0.11 mg/kg) but was undetectable in the other four. All the other analytes (carbofuran, 3-keto-carbofuran, 3-hydroxy-carbofuran, 7-phenol) were below the limit of determination in all six locations. Thus, the residues from the six independent trials were (0.04), (0.04), (0.04), (0.04),<0.02 and <0.02 mg/kg.

US trials (Martin, 1985) were also conducted in Illinois with carbofuran as a seed treatment or at planting treatment. Immature spring wheat seedlings were collected 10, 20, 30, 45 and 60 days after emergence and analysed for carbofuran and 3-hydroxy-carbofuran by the method of Schreier. The demonstrated limit of determination was 0.1 mg/kg. The limit of detection was estimated to be 0.02 mg/kg for each analyte. The results are shown in Table 38.

US GAP specifies 2 post-emergence ground or aerial applications of an F formulation at 0.28 kg ai/ha, made before the heads emerge from the boot. Treated forage may not be fed. GAP limited to Nebraska, South Dakota and Wyoming allows the application of the 4F formulation in-furrow at planting to small grains (including barley, oats and wheat) at 1.5 g ai/cm row, with a 15-cm minimum row spacing. The feeding of treated forage is prohibited.

Table 38. Residues of carbofuran and 3-hydroxy-carbofuran in or on immature wheat plants[1] following seed treatment or at planting treatment with carbofuran at 1.0 kg/ha.

-Treatment/Procedure	Days after emergence	Residue, mg/kg	
		Carbofuran	3-OH-CF
Furadan 25 ST, applied as 1% ai to seed	10 (22-day PHI)	3.3	5.6
	20	1.1	5.0
	30	<0.02	0.62
	45	(0.07)	0.68
	60	<0.02	0.46
Furadan 4F, microtube to soil in furrow	10 (22-day PHI)	1.5	1.2
	20	0.60	2.4
	30	<0.02	0.52
	45	<0.02	(0.15)
	60	<0.02	(0.22)
Furadan 5G, in-furrow at planting	10 (22-day PHI)	2.1	2.8
	20	0.58	2.4

-Treatment/Procedure	Days after emergence	Residue, mg/kg	
		Carbofuran	3-OH-CF
	30	<0.1	0.38
	45	<0.1 (0.05)	0.30
	60	<0.1 (0.03)	0.78

[1]Not a food or feed item.

Legume Vegetables

Soya beans. Trials were carried out in Brazil, France and the USA. Residues of carbofuran plus 3-hydroxy-carbofuran were below the limits of determination of the methods. Thailand submitted information on GAP for soya beans, but no data from the field trials (Thai Industrial Standard Institute, 1997). The results are given in Table 39.

GAP in Argentina, which can be used to evaluate the Brazilian trials, calls for application of the 10 G formulation in-furrow at planting at 1.5 kg ai/ha. In US GAP the 4F formulation is applied at planting at 1.7-2.0 kg ai/ha with a 100 cm row spacing or, if not used at planting, twice as a foliar spray at 0.28-0.56 kg ai/ha. No PHI is specified. GAP for France is 0.4 kg/ha of a 5% G formulation, but the data presented lacked detail.

Table 39. Total residues of carbofuran and 3-hydroxy-carbofuran in or on soya bean seeds.[1]

Country, year, variety	Form.	Application		PHI, days	Residue, mg/kg	Method of analysis, LOD	Ref.
		Method, timing	kg ai/ha)				
Brazil, 1994/ Engopa 201- Gold	5% G	in-furrow at planting	2	75	<0.05	Leppert, 0.1	Anon., 1997 FMC
			4	75	<0.05	Leppert, 0.1	Anon., 1997 FMC
France, 1988/ King	5% G	Soil at planting	0.60	150	<0.04	Blass, 0.04	Anon., 1988
USA (NE) , 1979	Furadan 4F	foliar spray at pod set	0.28	63	<0.05	Mollhoff, 0.1	Cook, 1978
		foliar spray at pod set and at pod maturity	2 x 0.28	36	0.10	Mollhoff, 0.1	Cook, 1978

[1]No data were submitted for forage or fodder

Yard-long beans. Thailand submitted information on GAP, but no data on residues (Thai Industrial Standards Institute, 1997).

Root and tuber vegetables

Carrots. The Netherlands submitted summary reports of field trials with Curraterr 200 SC applied to soil in 1980 before sowing carrot seeds (The Netherlands, 1997). The soluble concentrate was applied at 3.6-3.7 kg ai/ha and 3.6-7.5 g ai/l. Samples were analysed by the HPLC method of The Netherlands. No recovery data or storage periods from harvest to analysis were reported. The findings are shown in Table 40. Multiple results are from field replicates.

No GAP was available for The Netherlands or Europe. The data could not be evaluated for the estimation of a maximum residue level.

Table 40. Residues of carbofuran and 3-hydroxy-carbofuran in or on carrot roots from the application of Curraterr 200 SC to the soil at the time of sowing in The Netherlands.

Location/ Year	Rate		PHI, days	Residues, mg/kg		Method of analysis
	kg ai/ha	g ai/l		Carbofuran + 3-OH-CF	3-OH-CF conjugates	
Alkmaar, 1977	3.6	3.6	95	0.05; 0.08; 0.14; 0.26 (mean 0.14)	<0.01; <0.01; <0.01	Netherlands GLC (Mollhoff, 1979a)
Nooruyk-erhouk, 1977	3.6	6.0	118			
Metevik/ 1978	3.7	6.2	102	<0.01 <0.01 < 0.01 (mean <0.01)	<0.01 <0.01 <0.01	
Wageningen, 1980	3.7	7.5	111	<0.02 <0.02 <0.02 <0.02	0.05 0.05 0.06 0.05 (mean 0.05)	Netherlands HPLC
Zaltbommel	3.7	7.5	145	<0.02 <0.02 <0.02 <0.02	<0.01 <0.01 <0.01 <0.01	Netherlands HPLC

Abbreviated compound names: see Figure 1

Celeriac. The Netherlands submitted a summary report of one field trial with Curraterr 200 SC applied to the soil before planting celeriac in 1978. The application rate was 3 kg ai/ha and 5 g ai/l. Mature roots were harvested 158 days after the application and samples were analysed by the method of Molhoff (The Netherlands GLC method). The combined residue of carbofuran and 3-hydroxy-carbofuran was 0.05 mg/kg and the residue of 3-hydroxy-carbofuran conjugates <0.1 mg/kg. The stated limits of determination were 0.05 mg/kg for carbofuran plus 3-hydroxy-carbofuran and 0.1 mg/kg for 3-hydroxy-carbofuran conjugates. No GAP was available for The Netherlands or Europe.

Potatoes. Field trials were reported from Colombia, France, the UK and the USA. The tubers were treated at planting or post-emergence. Results are shown in Table 41.

GAP for Poland (2 kg ai/ha of 5 G) may be used for the evaluation of trials in France and the UK. GAP for Colombia or a neighbour was not available and the data from Colombia could not be considered for the estimation of a maximum residue level. US GAP requires an in-furrow application at planting of a 4F formulation at 3.4 kg ai/ha (Delaware, Pennsylvania, Virginia only). The same formulation may also be used post-emergence at 3.4 kg ai/ha in a shank or band application up to a four-inch rosette potato size. Up to 8 applications of the 4F formulation may be made at 1.1 kg ai/ha with a PHI of 14 days. The maximum foliar application is 3.4 kg ai/ha after an at-plant application, but no foliar applications may be made after a shank or band application.

Table 41. Residues of carbamates and phenolic metabolites in or on white potato tubers from the application of carbofuran.

Country, year, variety	Form./ Applin.	Rate		PHI, days	Residue, mg/kg		Method of analysis, LOD	Ref.
		kg ai/ha	kg ai/l		Carbamates	Phenols		

Country, year, variety	Form./ Applin.	Rate		PHI, days	Residue, mg/kg		Method of analysis, LOD	Ref.
		kg ai/ha	kg ai/l		Carbamates	Phenols		
France, 1973/ Bintje	Curraterr 5 G in-furrow at planting	1.5	-	154	<0.05[1]	-	Mollhoff, 0.1	Bayer 1975 Bayer 7155-75
UK, 1977/ Maris Piper	Yaltox 5G in-furrow at planting	5.0	-	101	0.03[2]	-	Mollhoff, 0.01	Bayer 1977 Bayer TCR 155/20-77
Columbia 1984/	Furadan 3G at planting and band. Furadan 3F foliar	3 x 1.0 2 x 1.3 (5.6 total)	 not stated	18	<0.02,[3] 0.07, 0.06	-	Schreier, 0.05	Martin 1985 FMC P-1316
Columbia, 1984	Furadan 3G at planting and band. Furadan 3F foliar	2 x 1.0 1 x 1.3 (3.3 total)	 not stated	69	0.05, 0.06, <0.02	-	Schreier, 0.05	Martin 1985 FMC P-1316
Columbia, 1984	Furadan 3G	3 x 1.0	-	134	0.06, <0.02, <0.02	-	Schreier, 0.05	Martin 1985 FMC P-1316
Columbia, 1984	Furadan 3F	0.8	not stated	171	<0.02, <0.02, <0.02	-	Schreier, 0.05	Martin 1985 FMC P-1316
Columbia, 1984	Furadan 3G at planting Furadan 3G band post-emergence Furadan 3F band	0.42 2 x 0.18 1.0 (1.78 total)	- - not stated	28	0.10, 0.06, 0.06	-	Schreier, 0.05	Martin 1985 FMC P-1316
Columbia, 1985	Furadan 3G at planting Furadan 3F foliar	1.0 2 x 1.0 (3.0 total)	 not stated	132	<0.02, <0.02, <0.02, <0.02	-	Schreier, 0.05	Martin 1985 FMC P-1316
Columbia, 1985	Furadan 3G at planting Furadan 3F foliar	1.0 2 x 1.0	 not stated	92	<0.02, <0.02, <0.02: <0.02	-	Schreier, 0.05	Martin 1985 FMC P-1316
Columbia, 1985	Furadan 3F foliar	3 x 1.0	not stated	92	<0.02, <0.02, <0.02, <0.02	-	Schreier, 0.05	Martin 1985 FMC P-1316

Country, year, variety	Form./ Applin.	Rate		PHI, days	Residue, mg/kg		Method of analysis, LOD	Ref.
		kg ai/ha	kg ai/l		Carbamates	Phenols		
Columbia/ 1985	Furadan 3F foliar	3 x 1.0	not stated	66	0.07, <0.02, <0.02, <0.02	-	Schreier, 0.05	Martin 1985 FMC P-1316
Columbia/ 1985	Furadan 3F foliar	4 x 1.0	not stated	35	<0.02, <0.02, 0.06, <0.02	-	Schreier, 0.05	Martin 1985 FMC P-1316
USA (ID)/ 1991/ Russet Burbank	Furadan 4F banded at hill-up	3.4	0.036	105	0.04, 0.04 (3-OH-CF)	0.23, 0.21	Barros, 0.03	Singer, 1992a
USA (ID)/ 1991/ Russet Burbank	Furadan 4F banded at hill-up	3.4	0.020	105	<0.01, <0.01	0.09, 0.09	Barros, 0.03	Singer, 1992a
USA (ND)/ 1991/ Norchip	Furadan 4F banded at hill-up	3.4	0.036	74	<0.01, <0.01	0.07, 0.07	Barros, 0.03	Singer, 1992a FMC P-2682
USA (OR)/ 1991/ Manona	Furadan 4F banded at hill-up	3.4	0.036	88	<0.01, <0.01	0.11, 0.15	Barros, 0.03	Singer, 1992a FMC P-2682
USA (PA)/ 1991/ Katahdin	Furadan 4F banded at hill-up	3.9	0.042	119	<0.03, <0.03	0.26, 0.31	Barros, 0.03	Singer, 1992a
USA (WA), 1991/ Russet Burbank	Furadan 4F Banded at hill-up	3.4	0.037	126	0.03, 0.03 (3-OH-CF)	0.48, 0.47 (7-P, 3-K-7-P)	Barros, 0.03	Singer, 1992a

[1]Carbofuran + 3-hydroxy-carbofuran + conjugates
[2]Carbofuran + 3-hydroxy-carbofuran
[3]Carbofuran + 3-hydroxy-carbofuran + 3-keto-carbofuran

No data were submitted on trials with multiple foliar post emergence applications.

Sugar beet. Field trials were conducted in France, Italy, Germany, the UK and the USA. Applications were at planting and/or foliar and only carbamate residues were determined except in the US trials where the phenol metabolites were also determined.

The European trials were evaluated against the GAP for Hungary (1.5-2 kg ai/ha of 10 G) and Bulgaria (875 g ai/100 kg ST). US GAP is a soil band treatment with the 4F formulation at 2.2 kg ai/ha with a 90-day PHI in Idaho, Oregon, Texas only and a soil band at planting through the six-leaf stage of the 4F formulation at 0.01 g ai/cm of row with a 90-day PHI in Nebraska only. The results are shown in Table 42.

Table 42. Total residues of carbamates and phenolic metabolites from the application of carbofuran to sugar beet.

Country, Year, Variety	Form./appln.	Rate		PHI, days	Residue, mg/kg		Method of analysis, LOD	Ref.
		kg ai/ha	kg ai/l		Carbamates	Phenols		
France, 1973/?	Curraterr 5G Unknown (?at-plant)	0.66		175	<0.05 (root) <0.05 (foliage)		Mollhoff 0.1	Mollhoff, 1974
France, 1973/?	Curraterr 5G Unknown (?at-plant)	0.68		186	<0.05 (root) <0.05 (foliage)		Mollhoff 0.1	Mollhoff 1974
Germany, 1973/ Poly-Beta	Curraterr 5G In-furrow at-plant	0.50		191	<0.05 (root) <0.05 (foliage)		Mollhoff 0.1	Mollhoff 1974
		1.0		191	<0.05 (root) 0.15 (foliage)			
Germany/ 1984/ Geem 65	500 SC Pelleting (with seed)	0.033 (30 g ai/100,000 pills)		174	<0.05 (root) 0.07 (foliage)		Mollhoff 0.05	Mollhoff 1985
				177	<0.05 (root) <0.05 (foliage)			
Germany/ 1984/ Novadima	500 SC Pelleting (with seed)	0.033 (30 g ai/100,000 pills)		208	<0.05 (root) <0.05 (foliage)			
UK, 1974/ Amono	Curraterr 5G Spreading at planting	0.75		136	<0.05 (root) <0.1 (foliage)		Mollhoff 0.1	Mollhoff 1975
Italy, 1974/Dickman Dima	Curraterr 5G Spreading at planting	0.60		155	<0.05 (root)		Moll-hoof 0.1	Mollhoff 1974
USA (Idaho), 1992/ WS-88	Furadan 4F Banded, postemergence (2-6 leaf)	2.24	0.019	86	0.05[1] (foliage)	0.18[2] (foliage) <0.01 (root)	Barros 0.03	Singer, 1992b
USA (Oregon), 1991/ Great North-western 2905	Furadan 4F Banded, post-emergence	2.24	0.017	92	<0.01 (foliage) <0.01 (roots)	0.03 (foliage) <0.03 (roots)	Barros 0.03	Singer, 1992b
USA (Idaho), 1991/ WS-88	Furadan 4F Banded, post-emergence	2.24	0.028	173	<0.01 (foliage) <0.01 (roots)	<0.03 (foliage) <0.03 (roots)	Barros 0.03	Singer, 1992b

Country, Year, Variety	Form./appln.	Rate		PHI, days	Residue, mg/kg		Method of analysis, LOD	Ref.
		kg ai/ha	kg ai/l		Carbamates	Phenols		
USA (Wy-oming), 1991/ Monohikari	Banded, at planting	2.24	0.034	181	<0.01 (foliage) <0.01 (roots)	<0.03 (foliage) <0.03 (roots)	Barros 0.03	Singer, 1992b

[1]3-OH-CF, 0.03 mg/kg and 0.07 mg/kg total carbofurans
[2]About 50% 3-keto-7-phenol, 0.26 mg/kg and 0.10 mg/kg total phenols

Swedes or turnips. Supervised field trials were conducted in France and Norway. The results are shown in Table 43.

No information on GAP was available so the data could not be evaluated for the estimation of a maximum residue level.

Only carbamate residues were determined. The applications were made at planting or early bulb formation. No measurable residues were found in any of the samples.

Table 43. Residues of carbamates in or on swedes from the application of carbofuran.

Country, Year, Variety	Form./ Application	Rate, kg ai/ha	PHI, days	Residue, mg/kg	Method of analysis, LOD[1]	Ref.
France, 1978/ Croissy	Curraterr 5G at planting	1.0	59	<0.05 (tops) <0.05 (roots)	Mollhoff 0.1	Mollhoff 1979
UK, 1977/ Acme (rutabaga)	Yaltox 5G spread at early to mid bulb formation	1.25	40	<0.01 (root)	Mollhoff 0.01	Anon. 1977
Norway, 1982/ (rutabaga)	Curraterr 5G	1.25	133	<0.05 (root, carbofuran) (0.05) (root, 3-hydroxy-carbofuran)	Mollhoff 0.1	Mollhoff 1983
Norway, 1982/ (rutabaga)	Curraterr 5G post-emergence after thinning	1.25	98	<0.05 (root, carbofuran) (0.05) (root, 3-hydroxy-carbofuran)	Mollhoff 0.1	Mollhoff 1983
`Norway, 1982/ (rutabaga)	Curraterr 5G at planting	2.5	133	<0.05 (root, carbofuran) (0.05) (root, 3-hydroxy-carbofuran)	Mollhoff 0.1	Mollhoff 1983

[1]No data were submitted to support the claimed limits of determination

Cotton seed (SO 691). Field trials were carried out in Brazil and the USA. The Meeting was informed that trials were in progress (1996-1997) in Southern Europe.

The trials in Brazil were with a single post-emergence foliar treatment of cotton plants with Furadan 350 SC at 1.0 or 2.1 kg/ha, both at about 600 l/ha, or a single post-emergence application along the plant rows with Furadan 50G at 2.5 or 5 kg/ha. In all cases the PHI was 45 days. Delinted cotton seeds were analysed by the method of Leppert. It was claimed that the method was validated at 0.1 mg/kg with 81% recovery, but no data were provided. The residues of carbofuran and 3-hydroxy-carbofuran were below the limit of determination, 0.1 mg/kg, in all four trials. The method does not include a hydrolysis step to release conjugated carbamates.

GAP for Brazil specifies an in-furrow treatment at planting with the 5 G formulation at 1.5-3 kg ai/ha or the 350 SC formulation at 0.7-1.05 kg ai/ha. There is also a seed treatment at 0.7 kg ai/100 kg seed with 350 ST formulation. The above trials therefore did not comply with GAP.

In two other trials in Brazil (San Paulo University, 1994) cotton seed (IAC 20) was treated with Furadan 350 TS at rates of 0.70 and 1.4 kg ai/100 kg seed. The seeds were planted in 1994 at an unstated rate of seeding and mature cotton seeds were harvested 154 days after treatment. The delinted seeds were analysed by the method of Leppert. Carbofuran and 3-hydroxy-carbofuran were below the limit of determination, 0.1 mg/kg. These trials complied with GAP.

The US trials (Shevchuk, 1993) were in California, Arizona, Texas, Mississippi and Louisiana with two broadcast foliar applications of Furadan 4F. Carbofuran and its carbamate and phenol metabolites were determined in delinted cotton seed by the method of Barros. A mass-selective detector was used for the phenols. Limits of determination were established for carbofuran at 0.1 mg/kg (76 ± 4% recovery), 3-hydroxy-carbofuran at 0.1 mg/kg (71 ± 1% recovery), 3-keto-carbofuran (77 ± 4% recovery), the 7-phenol at 0.2 mg/kg (85 ± 10% recovery), 3-keto-7-phenol at 0.2 mg/kg (107 ± 13% recovery) and 3-hydroxy-carbofuran at 0.2 mg/kg (73 ± 17% recovery). Recoveries of the carbamates at 0.05 mg/kg showed poor precision. Limits of detection of 0.01 mg/kg for the carbamates and 0.05 mg/kg for the phenols were claimed. The results of the trials are shown in Table 44. No results were reported for cotton fodder.

US GAP requires in-furrow treatment at planting with the 4F formulation at 0.14 or 1.12 kg ai/ha. The feeding of cotton forage is prohibited. The reported trials are not according to GAP as they are post-emergence treatments with PHIs of about 30 days.

Table 44. Residues of carbofuran and its carbamate and phenol metabolites in or on delinted cotton seed from the foliar application of Furadan 4F to cotton plants.

Location, Year, Variety	Rate		PHI, days	Residue, mg/kg							
	kg ai/ha	g ai/l		carbo-furan	3-K-CF	3-OH-CF	total carb-amates	7-phenol	3-K-7-P	3-OH-7-P	total phenols
Louisiana, 1992, Deltapine 5415	2 x 0.28	3	27	<0.05	<0.05	<0.05		<0.1	<0.1	<0.1	<0.1
Mississippi, 1992/ DES 119	2 x 0.28	3	27	<0.05	<0.05	<0.05		<0.1	<0.1	<0.1	<0.1
Texas, 1992/ GSC 71+	2 x 0.28	4	27	<0.05	<0.05	<0.05	<0.05	<0.1	<0.1	<0.1	<0.1
Texas/ 1992/ Paymaster HS 200	0.28 2.8 (3.1 total)	4	27	(0.05)	<0.05	(0.02)	(0.07)	<0.1	<0.1	<0.1	<0.1
Arizona, 1992/ DPL 5461	2 x 0.28	3	27	(0.06)	<0.05	<0.05	(0.06)	<0.1	<0.1	<0.1	<0.1

Location, Year, Variety	Rate		PHI, days	Residue, mg/kg							
	kg ai/ha	g ai/l		carbo-furan	3-K-CF	3-OH-CF	total carb-amates	7-phenol	3-K-7-P	3-OH-7-P	total phenols
California, 1992/ Germains GC-510	2 x 0.28	3	27	<0.05	<0.05	<0.05	<0.05	<0.1	<0.1	<0.1	<0.1

Abbreviated compound names: see Figure 1

Peanuts. Field trials were reported from Brazil and the USA. In Brazil, peanut plants were sprayed 80 days after planting with Furadan 350 SC at 1.75 or 3.0 kg/ha, 2.4 and 4.2 g/l respectively (Sao Paolo University, 1994b]. Peanuts with hulls were harvested 14 days after the treatment. The peanuts were shelled and the carbofuran residues in the kernels determined by the method of Leppert. Recoveries of 78 ± 6% at 0.1 mg/kg were reported, without data. No carbofuran (<0.1 mg/kg) was found in the two samples. The data could not be used for the estimation of a maximum residue level because adequate details were not provided. Brazilian GAP is an application at planting of the 350 SC formulation at 1.4-1.8 kg ai/ha (100-300 l/ha) or the 5 G formulation at 2 kg ai/ha.

Thailand submitted information on GAP, but none on residues (Thai Industrial Standards Institute, 1997).

Fourteen supervised field trials were reported from the USA (Helt, 1980, Nelson, 1981), in which peanut fields were treated at pegging, and in the 1981 trials at planting. Residues of carbofuran and 3-hydroxy-carbofuran were determined in or on kernels and hulls by the method of Schreier, with an NPD only. Limits of determination of 0.05 mg/kg or 0.1 mg/kg for carbofuran and 3-hydroxy-carbofuran on peanut kernels and 0.10 mg/kg or 0.20 mg/kg for carbofuran and 3-hydroxy-carbofuran on hulls were demonstrated by the analysis of fortified controls, with the following recoveries: carbofuran on kernels 88% at 0.05 mg/kg, 78 ± 6% (n = 4) at 0.10 mg/kg; carbofuran on hulls 76% at 0.10 mg/kg, 74% at 0.20 mg/kg; 3-hydroxy-carbofuran on peanuts 72% at 0.05 mg/kg, 83 ± 15% (n = 4) 94% at 0.10 mg/kg, 75% at 0.20 mg/kg. The results of the analyses are shown in Table 45. There are no registered US uses.

Table 45. Residues in or on peanut kernels and hulls from the application of carbofuran at planting and/or postemergence.

Location, Year, Variety	Form.	Application		PHI, days	Sample	Residue, mg/kg	
		kg ai/ha	Timing, method			Carbofuran	3-OH-CF
Alabama, 1979/	Furadan 10 G	2.2	Pegging 30-36 cm band	83	kernels	<0.02	<0.02
					hulls	(0.06)	0.30
Georgia, 1979/	Furadan 10 G	2.2	Pegging 30-36 cm band	92	kernels	<0.02	<0.02
					hulls	(0.03)	(0.08)
North Carolina/ 1979/ Florigiant	Furadan 10 G	2.2	Pegging 30-36 cm band	80	kernels	(0.02)	0.09
					hulls	0.12	0.62
Oklahoma / 1979/ Runner	Furadan 10 G	2.2	Pegging 30-36 cm band	121	kernels	<0.02	<0.02

Location, Year, Variety	Form.	Application		PHI, days	Sample	Residue, mg/kg	
		kg ai/ha	Timing, method			Carbofuran	3-OH-CF
					hulls	(0.04)	<0.05
Oklahoma, 1979/ Runner	Furadan 10 G	2.2	Pegging 30-36 cm band	120	kernels hulls	(0.01) (0.03)	<0.02 0.14
Virginia, 1979/Virginia 61R	Furadan 10 G	2.2	Pegging 30 -36 cm band	64	kernels hulls	<0.02 (0.06)	<0.02 0.10
Virginia, 1979 Florigiant	Furadan 10 G	2.2	Pegging 30-36 cm band	82	kernels hulls	(0.01) (0.03)	<0.02 <0.05
Georgia, 1980/ Florunner	Furadan 10 G	1.1 2.2 2.2 (5.5 total)	In-furrow at plant Band at plant Pegging 30 -36 cm band	60	kernels hulls	<0.02 1.2	0.22 1.8
Georgia, 1980/Florunner	Furadan 10 G	1.1 2.2 2.2 (5.5 total)	In-furrow at plant Band at plant Pegging 30 -36 cm band	50	kernels hulls	<0.02 0.48	(0.06) 0.60
Georgia, 1980/ Florunner	Furadan 10 G	1.1 2.2 2.2 (5.5 total)	In-furrow at plant Band at plant Pegging 30 -36 cm band	60	kernels hulls	(0.08) 0.74	(0.06) 0.35
Georgia, 1980/Runner	Furadan 10 G	1.1 2.2 2.2 (5.5 total)	In-furrow at plant Band at plant Pegging 30 -36 cm band	60	kernels hulls	(0.02) 0.22	(0.08) 0.22
Georgia, 1980/ Runner	Furadan 10 G	1.1 2.2 2.2 (5.5 total)	In-furrow at plant Band at plant Pegging 30 -36 cm band	60	kernels hulls	0.11 2.6	0.42 1.5
North Carolina/ 1980/ Florigiant	Furadan 10 G	1.1 2.2 2.2 (5.5 total)	In-furrow at plant Band at plant Pegging 30 -36 cm band	60	kernels hulls	(0.04) 0.36	0.10 0.40
North Carolina/ 1980/ Florigiant	Furadan 10 G	1.1 2.2 2.2 (5.5 total)	In-furrow at plant Band at plant Pegging 30 -36 cm band	60	kernels hulls	(0.04) 0.32	<0.02 (0.08)
North Carolina/ 1980/ NC 6	Furadan 10 G	1.1 2.2 2.2 (5.5 total)	In-furrow at plant Band at plant Pegging 30 -36	60	kernels hulls	(0.04) (0.12)	<0.05 (0.12)

Location, Year, Variety	Form.	Application		PHI, days	Sample	Residue, mg/kg	
		kg ai/ha	Timing, method			Carbofuran	3-OH-CF
			cm band				

Abbreviated compound names: see Figure 1

Rape (canola). Field trials were carried out in Canada and France. The trials in Canada included seed, at-plant and post-emergence foliar treatments. The treatments in France were at planting.

No GAP was reported for France, other European countries or Canada, but temporary GAP was reported for the USA where the 10% G formulation may be applied at 0.28 kg ai/ha by soil incorporation at planting. The use is limited to Minnesota, Montana, North Dakota and Washington, states bordering on or near Canada.

In Canada, rape seed was treated with Furadan 5G at planting in Manitoba and Alberta at 0.28 kg/ha (Leppert, 1980a). One or two additional applications of Furadan 4.8F were made at various growth stages (2 leaf, 3-4 inch, post-podding, post-flowering) at rates of 0.28 kg/ha for all stages except post-flowering at 0.14 kg/ha. The PHIs ranged from 23 days (post-flowering) to 108 days (at planting only). Rape seed was collected at normal harvest and analysed by the method of Leppert. Limits of determination of 0.09 mg/kg were demonstrated for carbofuran and 3-hydroxy-carbofuran, which were detectable (>0.01 mg/kg) but not measurable in one of eleven trials. The trial was in Manitoba and involved an at-plant application, a foliar application at the 3-4 inch growth stage, and a foliar application after podding, all at 0.28 kg ai/kg, with a 39-day PHI. Carbofuran was detected at an estimated 0.02 mg/kg and 3-hydroxy-carbofuran at an estimated 0.01 mg/kg, the total residue estimated as 0.03 mg/kg.

In Saskatchewan and Manitoba, Canada, rape seeds were treated with carbofuran (Furadan 350 ST) at 12 and 24 g ai/kg seed (Leppert, 1984). In 6 of the 7 trials, the treatment mixture also contained carbendazim and thiram. The seeds were grown to mature plants and the seeds from these were harvested (PHI 79-127 days) and analysed for carbofuran and 3-hydroxy-carbofuran by the Leppert method. A limit of determination of 0.1 mg/kg was demonstrated for each analyte. Carbofuran was detected at 0.01 mg/kg in one trial (127-day PHI). Neither carbofuran nor 3-hydroxy-carbofuran were detected in the remaining 6 trials.

As the Canadian trials did not comply with the temporary US GAP they could not be evaluated for the estimation of a maximum residue level.

In the French trials in 1976 (Ministry of Agriculture, 1977) carbofuran (5% granular) was incorporated into the soil when the seed was sown at rates of 0.68, 0.75 and 1 kg/ha, with PHIs of 275, 300 and 280 days. Seeds were analysed by an undefined semi-quantitative technique. It was claimed that carbofuran and 3-hydroxy-carbofuran were absent at limits of 0.005 mg/kg and 0.010 mg/kg respectively. GAP for France specifies 0.45 kg ai/ha of a 5% G formulation.

In four trials in 1979 in France (Anon., 1997) Curraterr 5G was applied in the furrow at rates of 0.9, 0.95 and 0.95 kg ai/ha. The PHIs were 345-364 days. Rape seed samples and straw were analysed by the method of Mollhoff and limits of determination of 0.2 mg/kg were demonstrated for carbofuran and 3-hydroxy-carbofuran on both seed and straw. The residues were undetectable in the seed and 0.20-0.24 mg/kg in the straw. The moisture content of the straw was not determined.

Sunflower. Field trials were reported from Canada, France and the USA. The Meeting was informed that trials were in progress (1996-1997) in northern and southern Europe.

No GAP was reported for France or Canada, but GAP in the USA, which may be applicable to Canada, specifies an in-furrow application of the 4F formulation at 3.1 kg ai/ha at planting and four foliar applications at 0.56 kg ai/ha with a 28-day PHI.

In six trials in Manitoba and Saskatchewan, Canada (Leppert, 1980b) sunflower plants 30-60 cm in 1-2 ft height were treated with one or two foliar applications of Furadan 4.8F at 0.28 or one at 0.56 kg/ha. The PHIs ranged from 91 to 111 days. Sunflower seeds were harvested at maturity and analysed by the method of Leppert. Limits of determination of 0.05 mg/kg were demonstrated for carbofuran (76% and 86% recovery) and 3-hydroxy-carbofuran (92 and 92%). No carbofuran or 3-hydroxy-carbofuran was found at or above 0.05 mg/kg. Carbofuran was detected in all samples at estimated concentrations of 0.01-0.03 mg/kg and 3-hydroxy-carbofuran was detected at 0.01 mg/kg in a single sample from the application of 0.56 kg/ha to 60 cm high plants after a 101-day PHI.

In France (Anon., 1977) carbofuran (5G) was applied to the seedbed line at the planting of sunflowers. The application rate was 0.40 kg ai/ha and the PHI 135 days. Residues of 0.02 mg/kg carbofuran and <0.05 mg/kg 3-hydroxy-carbofuran were reported, but the method of analysis was described as semi-quantitative and no details of it were provided.

In the USA (Brutschy, 1984) Furadan 4F or 15G applied as a band at cultivation at 1.1 kg ai/ha was followed by four foliar applications of Furadan 4F, each at 0.56 kg ai/ha. Seeds were collected at maturity and analysed by the method of Schreier. The phenol metabolites were determined by GC-MSD. Limits of determination of 0.05 mg/kg were demonstrated for each analyte by the analysis of triplicate fortified control samples, with the following recoveries: carbofuran 66, 50, 64%, 3-hydroxy-carbofuran 68, 62, 68%, the 7-phenol 68, 72, 56%, the 3-keto-7-phenol 98, 86, 80% and the 3-hydroxy-7-phenol 84, 84, 66%. The recoveries of the carbamates were low, about 60%, over the entire tested range of 0.05-0.20 mg/kg. The trial results are shown in Table 46. The maximum total residue was 0.65 mg/kg. Although the trials were according GAP, none were at the maximum at-plant application rate. The later season foliar applications, which were at the maximum rate, are more likely to have contributed most to the carbamate residues.

Table 46. Residues of carbofuran, 3-hydroxy-carbofuran and phenolic metabolites in or on sunflower seeds from the treatment of sunflowers with carbofuran.

Location, Year, Variety	Application		PHI, days	Residue, mg/kg						
	Form.	kg ai/ha		CF	3-OH-CF	Total carb.	7-P	3-K-7-P	3-OH-7-P	Total phenols
Kansas, 1983/ Oil	15G 4F	1 x 1.1 4 x 0.56	61	0.06	0.05	0.11	(0.02)	(0.02)	(0.04)	(0.08)
	4F	1 x 1.1 4 x 0.56	61	0.06	(0.04)	0.10	(0.02)	(0.02)	(0.04)	(0.08)
Arkansas, 1983/ Sunbred 265	15G 4F	1 x 1.1 4 x 0.56	52	(0.04)	(0.02)	(0.06)	(0.02)	(0.02)	(0.04)	(0.08)
	4F	1 x 1.1 4 x 0.56	52	(0.03)	<0.02	(0.03)	<0.02	<0.02	<0.02	<0.02
Minnesota, 1983/ Sigco Dwarf-Oil	15G 4F	1 x 1.1 4 x 0.56	42	0.05	(0.02)	0.07	(0.02)	(0.02)	(0.02)	(0.06)

Location, Year, Variety	Application		PHI, days	Residue, mg/kg						
	Form.	kg ai/ha		CF	3-OH-CF	Total carb.	7-P	3-K-7-P	3-OH-7-P	Total phenols
	4F	1 x 1.1 4 x 0.56	42	(0.04)	(0.02)	(0.06)	<0.02	<0.02	(0.02)	(0.02)
Illinois, 1983/ Oil	15G 4F	1 x 1.1 4 x 0.56	53	0.30	0.12	0.42	0.06	0.06	0.11	0.23
	4F	1 x 1.1 4 x 0.56	53	0.28	0.11	0.39	(0.04)	0.05	0.08	0.17
North Dakota, 1983/ Cargill 205-Oil	15G 4F	1 x 1.1 4 x 0.56	50	0.10	(0.02)	0.12	(0.02)	(0.02)	(0.02)	(0.06)
	4F	1 x 1.1 4 x 0.56	50	0.06	(0.02)	0.08	<0.02	(0.02)	(0.01)	(0.03)
Illinois, 1983/ Confectionery	15G 4F	1 x 1.1 4 x 0.56	53	0.21	0.08	0.29	0.06	0.06	0.08	0.20
	4F	1 x 1.1 4 x 0.56	53	0.18	0.08	0.26	0.05	0.05	0.06	0.16

Abbreviated compound names: see Figure 1

Leeks. The Netherlands provided the results of two field trials, one each in Waandenburg and Huissen, in 1977 (Ministry of Health, Welfare and Sport, 1997). Curraterr 200 SC was applied to the soil before planting at 4.4 kg ai/ha, 7.4 g/l. The mature crop was harvested 118 or 125 days after application. Leek bulbs were analysed by the method of Mollhoff. No limits of determination were stated. Results were reported as the sum of carbofuran, 3-hydroxy-carbofuran and conjugates of 3-hydroxy-carbofuran. In the Huissen trial the residues were <0.1 mg/kg (0.07, 0.07 mg/kg) and in the Waandenburg trial 0.13 mg/kg (0.15, 0.12, 0.12 mg/kg). No GAP was reported for The Netherlands or a neighbouring nation.

Onions. The Netherlands reported the results of three field trials in 1977 and 1978. Mature samples were analysed by the method of Mollhoff and limits of determination of 0.1 mg/kg were claimed for carbofuran plus 3-hydroxy-carbofuran and for conjugates of 3-hydroxy-carbofuran. The results are shown in Table 47. No GAP was reported.

Table 47. Residues of carbamate in onions after application of carbofuran.

Location, Year	Form.	Application			PHI, days	Carbofuran 3-OH-CF, mg/kg	Conjugate of 3-OH-CF, mg/kg) <0.1
		Timing	Rate, kg ai/ha	Rate, kg ai/l			
Nieuu Vossemeir, 1978	Curraterr 5G	after sowing	1.5	-	176	<0.1	<0.1
Willemstad, 1978	Curraterr 5G	after sowing	1.5	-	158	<0.1	<0.1
Zwingelspoon, 1977	Curraterr 200 SC	before sowing	5	8.4	90	<0.1	

Abbreviated compound names: see Figure 1

Celery. The Netherlands provided the results of two trials in 1978 in Berghen (trial 1) and Schayk (trial 2). Curraterr 200 SC was applied at 3.12 kg ai/ha, 5.2 g/l, to the soil one day before planting celery (goudgele relfblekende). Celery was harvested 84 days (trial 1) or 90 days (trial 2) after the treatment and samples were analysed by the method of Mollhoff. No limits of determination were stated. The combined residue of carbofuran and 3-hydroxy-carbofuran averaged 0.21 mg/kg (0.21, 0.18, 0.24 mg/kg) in trial 1 and 0.15 mg/kg (0.21, 0.12, 0.13 mg/kg) in trial 2. In both trials the conjugate of 3-hydroxy-carbofuran was <0.1 mg/kg (not detected). No GAP was reported for celery.

Tomatoes. Field trials were conducted in Brazil (Anon., 1994), Canada (Hawk, 1975), France (Anon., 1986b), Mexico (Shuttleworth, 1975) and the USA (Hawk, 1974). Thailand reported field trials according to GAP but without data or results (Thai Industrial Standards Institute, 1997). The findings are shown in Table 48.

GAP for Brazil specifies the use of the 350 SC formulation in-furrow at planting at 1.75 kg ai/ha or the 5 G at planting or transplanting at 4 kg ai/ha. There is no US GAP. GAP was not reported for Mexico, Canada or France.

Table 48. Residues of carbofuran and 3-hydroxy-carbofuran in or on tomatoes from the application of carbofuran to tomato plants.

Location, Year, Variety	Form.	Application		PHI, days	Residue, mg/kg		Method of analysis	Recovery, mg/kg/%	
		Method	kg/ha		CF	3-OH-CF		CF	3-OH-CF
Brazil, 1993/ Santa Clara	Furadan 350 SC	spray to soil around plant (200 l/ha)	3.5	60	≤0.05		Leppert	0.1/89	
			7.0	60	<0.05				
Brazil, 1993/ Santa Clara	Furadan 50G	broadcast and soil incorporation	2.6	60	<0.05		Leppert	0.1/89	
			5.2	60	<0.05				
Brazil, 1993/ Roma VF	Furadan 50G	broadcast and soil incorporation	4	60	≤0.05		Leppert	0.1/89	
			8	60	<0.05		Leppert	0.1/89	

Location, Year, Variety	Form.	Application		PHI, days	Residue, mg/kg		Method of analysis	Recovery, mg/kg/%	
		Method	kg/ha		CF	3-OH-CF		CF	3-OH-CF
Canada (Ontario), 1974/	Furadan 4.8F	foliar spray	0.56	83	<0.05	<0.05	Schreier	0.1/70, 85	0.1/93
		foliar spray	2 x 0.27	1	0.25	<0.05			
				3	<0.05	<0.05			
				6	<0.05	<0.05			
				10	<0.05	<0.05			
				27	<0.05	<0.05			
France, 1986/ Cam-Root	Curraterr MG (5%)	spread on seed beds	1.5	116	<0.05	<0.25 (conjugate)	Mollhoff		
France, 1986/ Lerica, F1 hybrid	Curraterr MG (5%)	spread on ground with 2-4 leaves on plants	0.75	98	<0.05	<0.25	Mollhoff		
France, 1986/ Variety ACE SS	Curraterr MG (5%)	Spread on ground before fruit formation	1	47	<0.05	<0.25	Mollhoff		
France, 1986/ Arimex	Curraterr MG (5%)	Spreading on 20 cm strips, at 2nd cluster, L 25 stage	1	29	<0.05	<0.25 (conjugate)	Mollhoff		
Mexico (Valle de Culia-can)/ 1975/	Furadan 3G Furadan 75WP	Spread, sidedress incorporated Foliar	2 3 x 1 (total 5)	7	<0.05 (9 plots) 0.056 (1 plot)	<0.05	Schreier	0.05/90	0.1/96, 80
				14	<0.05 (1 detect, 9 no detects)	<0.05 (10 no detects)			
USA/ (Florida), 1971/	Furadan 4F	Foliar spray	5 x 1.2	7	<0.05	<0.05	Schreier	0.1/100, 94, 80	0.1/109, 94, 64
USA / (Virginia, Maryland, Ohio), 1971	Furadan 10G	Banded at transplant	1 x 1.1	57 (VA) 77 (OH) 133 (MD)	<0.105	<0.05	Schreier	0.1/100, 94, 80	0.1/109, 94, 64

Abbreviated compound names: see Figure 1

Peppers, Chilli. Two trials were conducted, one each in California and New Mexico (Kim, 1995a). Furadan 4F was applied to the soil immediately before planting peppers at 1.2 kg ai/ha (5.9 g ai/l) in California (variety Jalapeno M) and at 1.1 kg ai/ha (3.5 g ai/ha) in New Mexico (variety NM 64). After approximately 2 months in California and 4 months in New Mexico, Furadan 4F was applied as a side-dressing (directed spray). The application rates were 1.7 kg/ha (9.0 g ai/l) in California and 1.7 kg/ha (14 g ai/l) in New Mexico. Mature peppers were harvested after a PHI of 28 days in both states.

The trials reflect the maximum US GAP.

The samples were analysed by the method of Barros. Limits of determination of 0.05 mg/kg were demonstrated for all the analytes, with the following recoveries from fortified control samples: carbofuran 94%, 3-keto-carbofuran 82%, 3-hydroxy-carbofuran 94%, 7-phenol 69%, 3-keto-7-phenol 91% and 3-hydroxy-7-phenol 96%. No carbamates were detected in any sample. The 7-phenol was detected below the limit of determination in both the California and New Mexico samples, at estimated levels of 0.02 and 0.04 mg/kg respectively. The 3-keto-7-phenol and the 3-hydroxy-7-phenol were also detected below the limits of determination in the New Mexico samples, both at 0.02 mg/kg.

Peppers, Sweet. Pepper plants were treated in 1974 with Furadan 4F 5 x 0.56 kg ai/ha in Ontario, Canada (Bednar and Stanovick, 1974). The PHI varied from 1 to 3 days. The samples were analysed by the method of Schreier, with limits of determination of 0.1 mg/kg for both carbofuran (78% recovery) and 3-hydroxy-carbofuran (97% recovery). At PHIs of 1, 2 and 3 days the carbofuran concentration was 0.26 mg/kg maximum (0.18 mg/kg average), 0.20 mg/kg maximum (0.18 mg/kg average) and 0.17 mg/kg maximum (0.15 mg/kg average) respectively. Residues of 3-hydroxy-carbofuran were <0.10 mg/kg at all PHIs.

Pepper plants in the USA (Kim, 1995b) were treated in 1994 either with Furadan 4F at 1.1 kg ai/ha at transplanting in-furrow or 4 weeks after transplanting as a side-dress. A second application was made after an interval of 1-3 months with side-dressing at 1.7 kg ai/ha (2.0 kg ai/ha in Florida). Bell peppers were collected at maturity 28 days after the final treatment from four plots in Florida, California, Texas and New Jersey and analysed by the method of Barros. The limit of determination was 0.05 mg/kg for all analytes as shown by the following recoveries from fortified control samples: carbofuran 84 ± 9%, 3-ket°Carbofuran 77 ± 3%, 3-hydroxy-carbofuran 78 ± 7%, 7-phenol 90%, 3-keto-7-phenol 111% and 3-hydroxy-7-phenol 94%.

Canadian GAP was not reported and US GAP does not reflect the conditions of either the Canadian or US trials. US GAP specifies two applications at 3.4 kg ai/ha, 1 at planting and a second 3-4 weeks later as a side-dress. The PHI is 21 days and the use is restricted to Arizona. The trials did not comply with GAP and two trials are inadequate to estimate a maximum residue level.

The LOD for all analytes was 0.05 mg/kg, with an estimated limit of detection of 0.01 mg/kg. The carbamate metabolites 3-keto-carbofuran and 3-hydroxy-carbofuran were not detected in any sample, and carbofuran was detected only in the California sample, at an estimated 0.01 mg/kg. The phenols were detected in all samples, but only the 7-phenol was quantifiable, in one Florida sample at 0.10 mg/kg. The maximum total residue was 0.21 mg/kg (Florida, in-furrow + side-dress). All others were ≤0.05 mg/kg.

Cucumbers. Twenty eight supervised field trials were conducted in the USA in Florida, Illinois, Virginia, Michigan, New York, Arkansas and California (Grigor and Tegris, 1987a). Furadan 15G was applied as an in-furrow treatment at planting (1.1 kg ai/ha or 3.4 kg ai/ha), or Furadan 4F was applied to the soil as a band at planting. Cucumbers were collected at maturity, at PHIs ranging from 44 to 67 days, and analysed by the method of Schreier, with the method extended to the determination of the phenol metabolites by GC-MSD. Limits of quantification of 0.05 mg/kg for each analyte were established by the analysis of replicate fortified controls. The recoveries (mean of 8) were as follows: carbofuran 93 ± 15%, 3-keto-carbofuran, 104 ± 16%, 3-hydroxy-carbofuran, 74 ± 1%, 7-phenol, 86 ± 19, 3-keto-7-phenol, 101 ± 16%, and 3-hydroxy-7-phenol, 84 ± 15%. The limits of detection were estimated as 0.02 mg/kg for the carbamates and 0.01 mg/kg for the phenols. Total residues as high as 0.6 mg/kg were found in one of two duplicate samples from Illinois. The results are shown in Table 49.

US GAP for cucumbers specifies application of the 10 or 15G formulation at planting, soil band incorporated at 2.2 kg ai/ha, or of the 4F formulation at 1.7 kg ai/ha. No PHI is specified. Although the trials were not conducted at the maximum GAP rates, they were at rates above and below the maximum with similar results. It can be concluded that residues from treatments according to GAP would be similar.

Thailand submitted information on trials according to GAP, but without data (Thai Industrial Standards Institute, 1997).

Table 49. Residues of carbofuran and carbamate and phenol metabolites in or on cucumbers following at planting treatment with Furadan 15G or Furadan 4F.

Location, Year, Variety	Form.	Rate, kg ai/ha	PHI, days	Residue, mg/kg					
				Carbo-furan	3-K-CF	3-OH-CF	7-phenol	3-K-7-P	3-OH-7-P
Florida, 1984/ Poinsetta 76	4F	1.1	67	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
	4F	3.4	67	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
	15G	1.1	67	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
	15G	3.4	67	(0.023)	<0.02	<0.02	<0.02	<0.02	<0.02
Illinois, 1984/ SMR 58	4F	1.1	53	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
	4F	3.4	53	<0.02	<0.02	<0.02	<0.02	0.02	<0.02
	15G	1.1	53	<0.02	<0.02	<0.02	<0.02	0.027)	<0.02
	15G	3.4	53	0.17 (0.27, 0.071)	<0.02	<0.02	(0.024)	(0.24, 0.11)	(0.018)
Virginia, 1984/	4F	1.1	65	0.13	<0.02	<0.02	<0.02	0.07	<0.02
	4F	3.4	65	0.21	<0.02	0.02	<0.02	0.092	<0.02
	15G	1.1	65	(0.030)	<0.02	<0.02	<0.02	<0.02	<0.02
	15G	3.4	65	0.12	<0.02	<0.02	<0.02	0.054	<0.02
Michigan, 1984/ Chicago Pickling	4F	1.1	52	0.15	<0.02	<0.02	<0.02	O.17 (0.22, 0.12)	<0.02
	4F	3.4	52	0.12 (0.091, 0.16)	<0.02	<0.02	<0.02	0.13 (0.088, 0.18)	<0.02
	15G	1.1	52	0.1	<0.02	<0.02	<0.02	(0.044)	<0.02
	15G	3.4	52	0.12 (0.086, 0.15)	<0.02	<0.02	<0.02	0.14	<0.02

Location, Year, Variety	Form.	Rate, kg ai/ha	PHI, days	Residue, mg/kg					
				Carbo-furan	3-K-CF	3-OH-CF	7-phenol	3-K-7-P	3-OH-7-P
New York, 1984/ Victory	4F	1.1	66	(0.028)	<0.02	<0.02	<0.02	<0.02	<0.02
	4F	3.4	66	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
	15G	1.1	66	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
	15G	3.4	66	0.060	<0.02	<0.02	<0.02	<0.02	<0.02
Arkansas, 1984/ Green Star	4F	1.1	44	0.05	<0.02	<0.02	<0.02	0.022)	<0.02
	4F	3.4	44	0.12	<0.02	<0.02	<0.02	0.076	<0.02
	15G	1.1	44	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
	15G	3.4	44	0.098	(0.026)	<0.02	<0.02	0.081 (0.062, 0.10)	<0.02
California, 1984/ Poinsetta 76	4F	1	52	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
	4F	3.4	52	<0.02	0.002	<0.02	<0.02	<0.02	<0.02
	15G	1	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
	15G	3.4	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02

[1]Furadan 4F was applied as an 18 cm band to the soil at planting. Concentration (kg ai/hl) not stated. Furadan 15G was applied to the furrow at planting

Cantaloupes. In supervised field trials reported from the USA in Florida, Illinois, Virginia, Michigan, New York, Arkansas and California, ± trials per state, Furadan 15G was applied in-furrow at planting (1.1 kg ai/ha or 3.4 kg ai/ha), or Furadan 4F was applied to the soil as ± band at planting (Grigor and Tegris, 1987b). Samples were collected at maturity, with PHIs of 60 to 92 days, and analysed by the method of Schreier, the phenols by GC-MSD. Limits of determination of 0.05 mg/kg for each analyte were established by the analysis of fortified controls, with recoveries from ± replicates of carbofuran 85 ± 15%, 3-keto-carbofuran 94 ± 15%, 3-hydroxy-carbofuran 68 ± 7%, 7-phenol71 ± 12%, 3-keto-7-phenol 102 ± 12%, and 3-hydroxy-7-phenol 80 ± 13%. Limits of detection were estimated as 0.02 mg/kg for the carbamates and 0.01 mg/kg for the phenols. The maximum total residue encountered was 0.12 mg/kg. No residues were detectable in or on the Florida samples (88-day PHI) and the residues were not quantifiable in or on any of the Arkansas (60-day PHI) or Virginia samples; one Arkansas sample (4F, 3.4 kg ai/ha) showed an estimated residue of 0.04 mg/kg carbofuran, estimated as ≤0.04 mg/kg, and all Virginia samples contained traces of carbofuran, estimated at 0.02 mg/kg. One of the duplicated samples from the 3.4 kg/ha treatments with 15G and 4F in Illinois contained quantifiable residues, 0.11 mg/kg and 0.081 mg/kg respectively. The major component was 3-hydroxy-carbofuran. In Michigan, treatment at 1.1 kg/ha yielded no quantifiable residues (<0.05 mg/kg) and treatment at 3.4 kg/ha yielded total residues of 0.10 mg/kg from the 4F formulation and 0.50 mg/kg in one of the duplicate from the 15G. The major components were 3-hydroxy-carbofuran and the 3-hydroxy-7-phenol. All New York samples contained carbofuran, ranging from 0.05 to 0.095 mg/kg. California samples from the 1.1 kg ai/ha applications (92 days PHI) also contained carbofuran, 0.05-0.11 mg/kg.

US GAP for cantaloupes is the same as for cucumbers: application of the 10 or 15G formulation at planting, soil band incorporated at 2.2 kg ai/ha, or of the 4F formulation at 1.7 kg ai/ha, no PHI specified. Although the trials were not at the maximum GAP rates, they included rates above and below the maximum, with similar results. It can be concluded that the residues from GAP treatments would also be similar.

Thailand submitted information on GAP trials, but without data on residues (Thai Industrial Standards Institute, 1997).

Summer squash. In trials in Florida, Illinois, Indiana, Michigan, New York, Arkansas and California (Grigor and Tegris, 1987c) Furadan 15G was applied in-furrow, or Furadan 4F to the soil as ± band, at planting at 1.1 or 3.4 kg ai/ha. Samples were collected at maturity, at PHIs ranging from 49 to 69 days, and analysed by the method of Schreier, with the same limits of determination (0.05 mg/kg) and detection (0.02 and 0.1 mg/kg) as for cantaloupes. Recoveries from fortified samples (n ± 8) were as follows: carbofuran 86 ± 16%, 3-keto-carbofuran 98 ± 13%, 3-hydroxy-carbofuran 68 ± 9%, 7-phenol 74 ± 6, 3-keto-7-phenol 112 ± 12%, and 3-hydroxy-7-phenol 85 ± 11%.

One Florida sample contained unquantifiable residues (15G, 3.4 kg ai/ha, estimated 0.04 mg/kg carbofuran and 3-keto-7-phenol) and residues were undetectable in the others (52-day PHI). One New York sample (15G, 3.4 kg ai/ha) contained an estimated 0.02 mg/kg of the 3-keto-7-phenol, and the remaining samples had no detectable residues (69-day PHI). Residues were undetectable in all the California samples (49-day PHI). All except one of the samples from Michigan (PHI 61 days, Yellow Crookedneck variety) contained residues of carbofuran only, 0.05-0.09 mg/kg. The exception contained unquantifiable carbofuran and 3-keto-carbofuran. All samples from Indiana (PHI 53 days, President Elite variety) contained detectable residues of carbofuran (estimated as 0.02-0.05 mg/kg) and the samples from the 15G applications contained total residues from 0.10 to 0.20 mg/kg, with each of the three phenols contributing 0.02-0.03 mg/kg and 3-hydroxy-carbofuran 0.07 mg/kg from the 3.4 kg ai/ha treatment and 0.05 mg/kg from 1.1 kg ai/ha. All Illinois samples contained detectable and quantifiable residues, except from the 1.1 kg ai/ha 15G treatment. Residues of the carbamates were 0.06, 0.08 and 0.10 mg/kg. All samples from Arkansas (PHI 37 days, Golden Girl variety) contained residues in the range 0.094-0.26 mg/kg, consisting of carbofuran only, except from the 15G formulation applied at 3.4 kg ai/ha where about 25% of the residue (0.26 mg/kg total) was 3-hydroxy-carbofuran.

US GAP for summer squash is the same as for cucumbers and cantaloupes. Again the squash trials were at rates above and below the maximum GAP rate with similar results, and residues from GAP applications would also be similar.

Thailand again submitted GAP information but no residue data (Thai Industrial Standards Institute, 1997).

Coffee beans. Supervised field trials were reported from Brazil and the USA.

In two trials in 1994-5 (Brooks, 1996c) in major coffee-growing regions of Brazil (Sao Paulo and Minas Gerais, Catuai variety) Furadan 5G was applied twice at 1.5 g ai/bush. The first application was 30-60 days after flowering and the second approximately 6 months later, with a 29-day PHI. The granules were applied as a band round the bases of the coffee bushes. The coffee cherries were harvested at maturity and sun-dried. The green beans were depulped by a commercial process and analysed by the method of Barros. Limits of determination of 0.05 mg/kg were established for carbofuran and the carbamate and phenol metabolites by the determination of recoveries from fortified controls. The average recoveries (means of duplicates) were carbofuran 76%, 3-keto-carbofuran 63%, 3-hydroxy-carbofuran 81%, 7-phenol 79%, 3-keto-7-phenol 99%, 3-hydroxy-7-phenol 73%. The recovery of 3-keto-carbofuran improved at a 0.5 mg/kg to 72%.

Duplicate samples were analysed from each of the two locations. At Sao Paulo the 3-hydroxy-7-phenol was found at 0.08 mg/kg in both samples, the 7-phenol and 3-keto-7-phenol were detected but <0.05 mg/kg, and carbofuran and 3-hydroxy-carbofuran were undetectable (<0.02 mg/kg). In the Minas Gerais samples, the 3-hydroxy-7-phenol was found at 0.16 and 0.13 mg/kg, 3-hydroxy-carbofuran and the 7-phenol were detected at estimated concentrations of 0.03 mg/kg each, and carbofuran was undetectable.

In Brazil, samples of beans from coffee bushes treated with a foliar spray of Furadan 350 SC at 2.1 or 4.2 g ai/bush, 200 l/ha, in 1994 (Sao Paolo University, 1994) were analysed for carbofuran by the method of Leppert. The PHI was 90 days. A recovery of 79 ± 6% was reported at 0.1 mg/kg fortification, but no details were provided. Carbofuran was below the limit of determination (<0.1 mg/kg).

GAP in Brazil specifies application of 0.35 g ai/tree of the 350 SC formulation or application of 0.5-3 g ai/tree of the 5G; the timing is not specified. GAP for foliar treatment was not reported.

Four supervised field trials were conducted on the islands of Kauai, Hawaii and Oahu in the USA (Brooks, 1996a). Two applications of Furadan 5G were made in each trial at 1.5 g ai/tree, the first after flowering and the second after an interval of 5-6 months. For each treatment, approximately 30 g of the granular formulation was applied to the soil near the base of each tree and "minially incorporated (less than 1 cm)". The PHIs were 28-29 days. The treated cherries were harvested at maturity, dried and shelled by the commercial wet method. The beans were analysed by the method of Barros. A limit of determination of 0.05 mg/kg for each analyte was established by the analysis of fortified controls but precision was generally poor. Recoveries were carbofuran (n = 9) 88 ± 16%), 3-keto-carbofuran (n = 8) 105 ± 13%, 3-hydroxy-carbofuran (n = 10) 88 ± 17%, 7-phenol (n = 6) 64 ± 12%, 3-keto-7-phenol (n = 6) 92 ± 14% and 3-hydroxy-7-phenol (n = 6) 72 ± 13%. The results are shown in Table 50.

GAP in the USA specifies the application of a 10% G formulation at 1.7 g ai/tree twice each year to the base of coffee trees in Puerto Rico only. The application rate in the trials was within 10% of the GAP rate and the results could be used to estimate a maximum residue level.

Table 50. Residues of carbamates and phenols in or on green coffee beans from the application of Furadan 5G (2 x 1.5 g ai/tree) to the base of coffee plants, 28-29-day PHI.

Location/Year/ Variety	Residue, mg/kg					
	Carbofuran	3-K-CF	3-OH-CF	7-Phenol	3-K-7-P	3-OH-7-P
Kauai, 1995/ Yellow catuai	<0.02	(0.02)	(0.02)	0.16	(0.03)	0.33
Kauai, 1995/ Yellow catuai	<0.02	<0.02	0.25	0.24	0.10	0.64
Kauai, 1995/ Yellow catuai	<0.02	<0.02	0.79	0.32	0.20	1.08
Hawaii, 1995/ Guatamalan	<0.02	<0.02	0.08	(0.04)	(0.02)	0.22
Hawaii, 1995/ Guatamalan	<0.02	<0.02	0.12	(0.04)	(0.02)	0.28

Location/Year/ Variety	Residue, mg/kg					
	Carbofuran	3-K-CF	3-OH-CF	7-Phenol	3-K-7-P	3-OH-7-P
Oahu, 1995/ Guatamalan	≤0.02	<0.02	≤0.02	(0.01)	<0.052	(0.02)

Abbreviated compound names: see Figure 1

Head cabbage. The Netherlands reported two supervised field trials in which Curraterr 200 SC was applied to head cabbage in 1977 at rates of 0.04 and 0.25 g ai/plant, 0.40 g ai/l water. The mode of application was not described. The PHI was 69 days for the lower application rate and 155 days for the higher. Mature heads were analysed by the method of Mollhoff. All residues were <0.1 mg/kg, the stated limit of determination. GAP was not reported for The Netherlands or a neighbouring country.

Brussels sprouts. The Netherlands reported two supervised field trials with the application of Curraterr 200 EC to Brussels sprouts in 1977 at a rate of 0.025 g ai/plant, 0.25 g ai/l water. Mature sprouts were harvested 126-127 days after the treatment and analysed by the method of Mollhoff. All residues were <0.1 mg/kg, the stated limit of determination. GAP was not reported.

Cauliflower. The Netherlands reported five trials. Curraterr 200 SC was applied to cauliflower plants at rates of 0.025 or 0.038 g ai/plant, and 0.25 or 0.38 g ai/l water. The mode of treatment was not described. Mature crops were harvested 61, 69 or 71 days after the application and analysed by the method of Molhoff. The residues of conjugates of 3-hydroxy-carbofuran were <0.1 mg/kg, the stated limit of determination. The combined residues of carbofuran and 3-hydroxy-carbofuran were 0.2, <0.1, 0.18, 0.22 and <0.1 mg/kg. GAP was not reported.

Kohlrabi. Germany submitted the results of two field trials with single applications of Curraterr-Granulat GR (50 g ai/kg) at a rate of 0.645 g/m. The rate per area and the type of application were not reported. It is implied that residues were determined as carbofuran plus 3-hydroxy-carbofuran and as 3-hydroxy-carbofuran conjugates. Limits of determination and sample chromatograms were not supplied. In the Oldenberburg trial, the application was made 38 days after planting and the carbamate and conjugate residues were <0.1 mg/kg at a 27-day PHI and <0.05 mg/kg after 40 and 54 days. In the Braunschweig trial, the treatment was 52 days after planting and residues of carbofuran plus 3-hydroxy-carbofuran were 2.99 mg/kg at a PHI of 25 days, 0.28 mg/kg at 36 days and 0.17 mg/kg at 52 days. The residues of the conjugates at the same PHIs were 0.7, <0.05 and 0.11 mg/kg. GAP was not reported.

Grapes. Field trials were reported from Germany (Federal Biological Research Centre for Agriculture and Forestry, 1996), Mexico (Fullmer, 1977) and the USA (Pejovich, 1984).

In Germany Curraterr-Granulat GR (50 g ai/kg) was applied to mature grape vines in 1986 at four locations and grapes were harvested at intervals of 0-79 days. The growth stages at treatment were described as stages 27-33 and the treatment was 2 x 0.5 g ai/vine; the rate per area was not reported. The PHIs of the analytical samples are significantly shorter than the crop harvest interval, suggesting that immature crops may have been sampled. Samples were analysed by an undisclosed method and it was not stated whether carbofuran only or carbofuran plus certain metabolites were determined. The results are shown in Table 51. GAP was not reported for Germany or the European Union.

Table 51. Residues in or on grapes resulting from the treatment of vines with a granular formulation of carbofuran in Germany in 1986 at 2 x 0.5 g ai/vine.

Location	PHI, days	Residue[1], mg/kg
Weinsberg	0	0.04
	21	0.08
	42	0.12
Bernkastel-Kues	0	0.09
	22	<0.04
	79	<0.04
Neustadt-W	0	<0.04
	21	>0.04
	35	<0.04
	56	<0.04
	70	0.11
Marienthal	0	<0.04
	21	0.26
	35	0.09
	56	0.12
	70	0.1

[1]The constituents of the residue were not reported

In Mexico, Furadan 5G was applied once to vineyard soil at 10 or 20 kg ai/ha in three trials. Samples of mature grapes were collected 123 days or 43 days after the treatment and analysed by the method of Schreier with limits of determination of 0.1 mg/kg for both carbofuran and 3-hydroxy-carbofuran. The recoveries from single spiked samples were 109% and 70% for carbofuran. The results are shown in Table 52.

GAP for Mexico was not reported, but GAP in the neighbouring USA (California only) requires 11.2 kg ai/ha of the 4F formulation, soil-incorporated after harvest with a PHI of 200 days. Pre-harvest drip irrigation of the same formulation may be made at 3.4 kg ai/ha with a 60-day PHI. The PHI of 123 days in one of the trials was within 40% of the US PHI and the 10 kg ai/ha rate was about 90% of the US rate.

Table 52. Residues of carbofuran in or on grapes from single applications of Furadan 5g to vineyard soil in Torreon, Mexico.

kg ai/ha	PHI, days	carbofuran, mg/kg	3-hydroxy-carbofuran, mg/kg
10	123	<0.1	<0.1
10	43	0.24	0.66

		(0.22; 0.27)	(0.52; 0.80)
20	43	1.1 (1.3; 0.93)	1.5 (1.5; 1.5)

In the USA, vineyards were treated 3 or 5 times by drip irrigation with Furadan 4F at 1.1 or 2.2 kg ai/ha/application at four locations in California in 1983-1984. Grapes taken at normal harvest were analysed by the method of Schreier. An MSD was used for the quantification of the carbamate residues and an NPD for the phenols. Limits of determination were demonstrated at 0.1 mg/kg by the analysis of fortified controls, with recoveries from triplicate analyses of carbofuran 78% ± 4.9%, 3-hydroxy-carbofuran 81% ± 6.2%, 7-phenol 89% ± 4.0%, 3-keto-7-phenol 101% ± 2.1%, and 3-hydroxy-7-phenol 63% ± 4.4%. Recoveries at 0.05 mg/kg were not acceptable and results at such concentrations would be semi-quantitative. The results are shown in Table 53. The maximum total carbamates residue was 0.15 mg/kg and the maximum combined residue of carbamates and phenols was 0.32 mg/kg.

Some trials corresponded to US GAP for post-harvest treatment of vineyards. The maximum total trial rate was 110% of the GAP rate and the PHIs were about 200 days. Some trials also complied with US GAP for pre-harvest treatments (3.4 kg ai/ha, 60-day PHI).

Table 53. Residues in or on grapes from the application of Furadan 4F by drip irrigation to vineyards in California, 1983-4.

Trial No., Location	Variety	Application			PHI, days	Residue, mg/kg				
		kg ai/ha	No.	Total kg ai/ha season		CF	3-OH-CF	7-phenol	3-K-7-P	3-OH-7-P
RRA-038 Thermal	Perlette	4.5 2.2	1 3	11	218	<0.05	<0.05	<0.05	<0.05	<0.05
RRA-041 Thermal	Cardinal	4.5 2.2	1 3	11	209	<0.05	<0.05	<0.05	<0.05	<0.05
RRA-057 Thermal	Thompson Seedless	2.2	3	6.7	238	<0.5	<0.05	<0.05	<0.05	<0.05
RRA-100 Lost Hills	Perlette	2.2	3	6.7	266	<0.05	<0.05	<0.05	<0.05	<0.05
RRA-113 Madera	Thompson Seedless	2.2	3	6.7	256	<0.05	(0.03)	<0.05	<0.05	<0.05
RRA-241 Soledad	Merlot	2.2	6	13	294	<0.05	<0.05	(0.02)	(0.02)	<0.2
RRA-039 Thermal	Perlette	3.4	1	3.4	54	<0.05	0.1	(0.04)	(0.04)	(.07)
RRA-042 Thermal	Cardinal	1.1	3	3.4	54	<0.05	(0.06)	(0.03)	(.03)	(0.03)
RRA-046 Thermal	Perlette	1.1	3	3.4	60	<0.05	(0.06)	(0.04)	(0.04)	<0.1 (0.04)
RRA-048 Thermal	Cardinal	1.1	3	3.4	60	<0.05	<0.05 (0.03)	<0.05 (0.02)	<0.05 (0.02)	<0.1 (0.02)

Trial No., Location	Variety	Application			PHI, days	Residue, mg/kg				
		kg ai/ha	No.	Total kg ai/ha season		CF	3-OH-CF	7-phenol	3-K-7-P	3-OH-7-P
RRA-059 Thermal	Thompson Seedless	1.1	3	3.4	59	<0.05	0.1	(0.06)	(0.05)	0.11
RRA-102 Lost Hills	Perlette	1.1	3	3.4	60	<0.05	0.12	(0.06)	<0.05 (0.04)	<0.1 (0.08)
RRA-111 Madera	Thompson Seedless	1.1	3	3.4	63	(0.02)	(0.08)	(0.04)	<0.05 (0.02)	<0.1 (0.02)
RRA-243 Soledad	Merlot	1.1	3	3.4	139	<0.05	(0.03)	(0.02)	(0.02)	<0.1 (0.02)
RRA-063 Thermal	Thompson Seedless	2.2 1.1	3 2	9	59	<0.05	(0.09)	(0.05)	(0.04)	(0.09)
RRA-105 Lost Hills	Perlette	2.2 1.1	3 2	9	60	<0.05	(0.08)	(0.04)	<0.05 (0.04)	<0.05 (0.05)
RRA-115 Madera	Thompson Seedless	2.2 1.1	3 2	9	63	(0.03)	0.10 (0.12; 0.07)	(0.04)	(0.02)	(0.02)
RRA-248 Soledad	Merlot	2.2 1.1	32	9	153	<0.05	(0.06)	(0.04)	(0.03)	<0.1 (0.05)

Abbreviated compound names: see Figure 1

Strawberries. Supervised field trials were conducted in France, The Netherlands, the UK and the USA.

In France (Anon., 1997) Curraterr 5 MG (microgranulate) was applied once or twice in 1982 to strawberry plants as a band. Ripe fruits were harvested and analysed by the method of Molhoff. Recoveries from fortified control samples were reported without details (3-hydroxy-carbofuran at 0.1 mg/kg, 90% and 99%, carbofuran at 0.05 mg/kg 36%, 41% and 47%; at 0.5 mg/kg 49%, 46% and 46%). A limit of determination was not adequately established for carbofuran and any results for this analyte are semi-quantitative. The results are shown in Table 54.

Table 54. Residues of carbofuran and 3-hydroxy-carbofuran in or on strawberries from the application of Curraterr 5 MG in France, 1982.

Variety	Application			PHI, days	Residue, mg/kg	
	Stage	Method	Kg ai/ha		Carbofuran[1]	3-OH-CF
Red Gauntlet	Before bloom	Band (0.90 m)	1.0	48	(<0.05)	<0.1
Tago	Before bloom Fruit-bearing plants	Band (0.90 m) Band over 2 rows	1.3 0.89	13	(0.4)	0.50 (0.46, 0.54)
Tago	Fruit-bearing plants	Band (0.90 m)	0.89	13	(0.06)	0.20
Tago	Before bloom Fruit-bearing plants	Band (0.90 m) Band over 2 rows	0.89	13	(0.20)	0.26

			0.89			
Tago	Before bloom. Fruit-bearing plants	Band (0.90 m). Band over 2 rows	0.44 0.89	13	(0.08)	0.37 (0.30; 0.43)
Red Gauntlet	Before bloom.	Band (0.90 m).	2.0	48	(0.06)	0.10

Abbreviated compound names: see Figure 1
[1]Limit of determination was not established. Results are estimates only

In three field trials with post-harvest application of carbofuran to strawberry plants in the UK (Bagnall, 1986) Yaltox 5G was applied to three-year-old plants arranged in matted-bed rows with a Horstine Microband Applicator at 1.5 or 2.0 kg ai/ha. Mature strawberries were harvested the following season. The PHIs ranged from 309 to 316 days. The method of Mollhoff was utilized, but the analytes were derivatized to 2,4-dinitrophenyl ethers (Cook *et al.*, 1977). Limits of detection of 0.05 mg/kg for carbofuran and 0.1 mg/kg for 3-hydroxy-carbofuran were claimed, but no recovery data or chromatograms were supplied. The manufacturer's submission listed recovery information (85% for carbofuran, 74% for 3-hydroxy-carbofuran, fortification level not reported), but not in the field trial report. None of the strawberry samples contained detectable residues of carbofuran or 3-hydroxy-carbofuran.

The Netherlands provided a summary report of a field trial conducted in 1977. Curraterr 5G was applied to strawberry plants 2 weeks after transplanting at rates of 5 or 10 kg ai/ha. Mature fruit were harvested 272 days after treatment and analysed by the method of Molhoff. The results are shown in Table 55.

Table 55. Residues of carbofuran and 3-hydroxy-carbofuran in or on strawberries from the application of Curraterr 5G at 5 or 10 kg ai/ha, 272-day PHI. The Netherlands, 1978.

Application, kg ai/ha	carbofuran + 3-OH-CF, mg/kg	Conjugates of 3-OH-CF, mg/kg
5	0.08; 0.13; 0.11 (0.14 mean)	0.13; 0.15; 0.17 (mean 0.15)
10	0.37; 0.18; <0.1 (0.22 mean)	0.34; 0.19; 0.20 (mean 0.24)

Abbreviated compound names: see Figure 1

Three field trials with the post-harvest application of Furadan 4F to strawberry plants as a foliar spray were reported from the USA (Shevchuk, 1995a). The trials were in New York, Michigan and Virginia at 2.2 kg ai/ha, 12 g/l in New York and Virginia and 19 g/l in Michigan. Strawberries were harvested at maturity the following season and analysed by the method of Barros. The PHIs ranged from 225 days to 270 days. The limits of determination were established by the analysis of fortified controls in triplicate. The following recoveries were reported at 0.05 mg/kg: carbofuran 79 ± 3.0%, 3-keto-carbofuran 87 ± 10%, 3-hydroxy-carbofuran 79 ± 6.0%, 7-phenol 109 ± 5.0%, 3-keto-7-phenol 85 ± 1.0%, and 3-hydroxy-7-phenol 57 ± 3%. No analyte was detected (<0.02 mg/kg) in any sample.

GAP was reported only for the USA, where the soil may be treated post-harvest at 2.2 kg ai/ha with the 4F formulation after 1 October. The use is limited to Oregon, Michigan, Minnesota, Missouri, Tennessee and Washington. The US trials reported complied with this GAP.

<u>Bananas</u>. Supervised field trials were reported from Central America, South America and Spain.

Eight supervised field trials were conducted in the 1985-1986 growing season in Costa Rica, Honduras, Mexico, Ecuador and Guatemala (Leppert, 1986b). Furadan 10G (5G in Mexico) was applied twice at rates of 8.1-11 kg ai/ha, except in Costa Rica where the two applications were at 3.8 kg ai/ha, at intervals of about 6 months. Samples of bananas taken at 10, 30, 60, 90 and 120 days after the second application were analysed by the method of Schreier with an NPD for carbamates and an MSD for phenols. The pulp and peel were analysed separately for carbamates but whole fruit were analysed for phenols. The following recoveries were reported, without supporting data, from replicated samples spiked at 0.05 mg/kg, carbofuran (n = 6) 74 ± 10%, 3-keto-carbofuran (n = 6) 86 ± 21%, 3-hydroxy-carbofuran (n = 6) 86 ± 21%, 7-phenol (n = 5) 79 ± 10%, 3-keto-7-phenol (n = 5) 96 ± 17%, 3-hydroxy-7-phenol (n = 5) 78 ± 12%. The precision was unacceptable at 0.05 mg/kg for 3-keto- and 3-hydroxy-carbofuran, but acceptable accuracy and precision were demonstrated at 0.1 mg/kg: 79 ± 10% (n = 10) and 76 ± 8% (n = 11) respectively.

In all eight trials at all PHIs, the carbofuran and metabolite residues were below the limits of determination (<0.05 mg/kg for carbofuran, and the phenols, <0.1 mg/kg for 3-keto-carbofuran and 3-hydroxy-carbofuran). In the Mexico trial, 3-keto-carbofuran and 3-hydroxy-7-phenol were detected at estimated levels of 0.02 and 0.04 mg/kg respectively. In the Costa Rica trial, each of the phenols was detected at one or more PHIs; the estimated maximum total concentration was 0.04 mg/kg.

Summary results (Sao Paolo University, 1994e) were reported from a 1993 field trial in Brazil that involved single applications of Furadan 350SC to the soil round banana plants at rates of 4.2 and 8.4 g ai/plant. Samples were harvested 90 days after treatment and analysed for carbofuran by the method of Leppert. A limit of determination of 0.1 mg/kg was claimed. No residues were found. No details were provided in the very short summary.

Summary results were provided from a field trial in Spain in 1986 (Anon., 1986a). Curraterr 350SC was applied by irrigation to banana plants at 16 kg ai/ha in 50,000 l water/ha. Mature fruits were harvested 61 days after treatment and analysed for carbofuran and 3-hydroxy-carbofuran by the method of Molhoff. A limit of determination of 0.05 mg/kg was claimed. No residues were detected in either pulp or peel.

GAP was reported only for Spain, where 5G is applied at 0.6-0.75 kg ai/ha and 20F at 5.6 kg ai/ha, both with 60-day PHIs. The trial in Spain was at a much higher application rate, but the data could be used because no residues were detected (<0.02 mg/kg).

Thailand submitted GLP field trial information, but no report on residues (Thai Industrial Standards Institute, 1997).

Sugar cane. Four supervised field trials were conducted in Brazil (Sao Paolo University, 1994c) and in the USA (Shevchuk, 1992).

In Brazil separate plots were treated with 3 or 6 kg ai/ha of Furadan 50G (50 g ai/kg) or 1.75 or 3.5 kg ai/ha of Furadan 350SC (350 g ai/l), applied to the soil on the plant row about 5 months after planting. The cane was harvested 90 days after treatment. Samples were analysed by the method of Leppert. A limit of determination of 0.1 mg/kg was claimed, with a recovery of 86 ± 3% at unspecified fortification concentration(s). All samples contained <0.1 mg/kg.

In the USA, Furadan 4F was applied to sugar cane in six trials in three States. The first application was made in-furrow at planting at 1.1 kg ai/ha, 3.9-5.9 g ai/l. Two additional applications were made as aerial foliar sprays after joint formation at 0.84 kg ai/ha (9.0 g ai/l), a total of 2.8 kg ai/ha. The final application was 30 days before harvest. Samples were analysed by the method of Barros. A limit of determination of 0.03 mg/kg was demonstrated for each analyte by the analysis of 10 fortified controls with the following recoveries: carbofuran 91 ± 7%, 3-hydroxy-carbofuran 85 ± 10%, 3-keto-carbofuran 86 ± 10%, 7-phenol 101 ± 15%, 3-hydroxy-7-phenol 104 ± 16%, and 3-keto-7-phenol 102 ± 14%. The results are shown in Table 56.

Table 56 . Residues of carbofuran and metabolites in or on sugar cane from the application of Furadan 4F at planting (1.1 kg ai/ha) and foliar (2 x 0.84 kg ai/ha), 30-day PHI. USA, 1990-91.

State	Residue, µg/kg					
	Carbofuran	3-K-CF	3-OH-CF	7-Phenol	3-K-7-P	3-OH-7-P
Florida[1]	0.05, 0.05	<0.01, <0.01	<0.01, <0.01	(0.01), (0.01)	<0.01, <0.01	<0.01, <0.01
Florida[1]	0.06, 0.06	<0.01 <0.01	<0.01, <0.01	(0.01), <0.01	<0.01 <0.01	<0.01, <0.01
Florida[1]	0.05, 0.05	<0.01, <0.01	<0.01, <0.01	(0.01), (0.02)	<0.01, <0.01	<0.01, <0.01
Florida[1]	0.04, 0.04	<0.01, <0.01	(0.02), <0.01	(0.02), (0.02)	<0.01, <0.01	<0.01, <0.01
Louisiana[2]	(0.02), (0.02)	<0.01, <0.01	<0.01, <0.01	(0.02), (0.02)	<0.01, <0.01	<0.01, <0.01
Louisiana[2]	0.04, 0.06	<0.01, <0.01	<0.01, <0.01	(0.02), (0.02)	<0.01, <0.01	(0.01), (0.01)
Louisiana[3]	(0.02), (0.02)	<0.01, <0.01	<0.01, <0.01	(0.02), (0.02)	<0.01, <0.01	<0.01 <0.01
Louisiana[3]	(0.01), (0.02)	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01	(0.01), (0.01)
Texas[4]	<0.01, <0.01	<0.01, <0.01	<0.01 ,<0.01	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01
Texas[4]	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01	(0.01), <0.01

Abbreviated compound names: see Figure 1
[1]Four trials at same location [2]Two trials at same location [3]Two trials at same location [4]Two trials at separate locations

GAP for sugar cane in Brazil specifies 1.4-1.75 kg ai/ha (100-300 l/ha) of the 350 SC formulation applied in furrow with a planting stick or in bands or 1.5-3 kg/ha of the G formulation at 1.5-3 kg ai/ha applied in bands about the plants at second harvest. In US GAP for the 4F formulation no more than 2 foliar applications of 0.84 kg ai/ha each are made, with the use limited to the mainland, with a 17-day PHI. The 10G formulation may also be applied early in the season at 4.5 kg ai/ha with soil incorporation. None of the US trials complied with GAP, as an additional at-plant application was made at 1.1 kg ai/ha, but the residues were generally at or below the LOD.

Animal transfer studies

In a poultry feeding study in the USA in 1968 (Cook, 1968) three groups of 30 laying pullets were subdivided into three groups of 10 hens each for dosing or feeding at levels equivalent to 0.05, 0.5 or 5.0 ppm in the diet. The first group was fed carbofuran, the second group 3-hydroxy-carbofuran and the third alfalfa that had been treated with carbofuran. The dosing period was 10 consecutive weeks, during which eggs were collected periodically for analysis. At the end of the period, the pullets were killed and tissues taken for analysis. The period of frozen storage before extraction and analysis was not disclosed.

Eggs without shell, muscle, gizzard and liver were extracted with acetone. The extract was concentrated, hydrolysed with 0.25 N HCl and extracted with methylene chloride. The solvent was evaporated and the residue dissolved in acetonitrile and partitioned with hexane. The acetonitrile was evaporated and the residue dissolved in methylene chloride and cleaned up with Nuchar-Attaclay and silica gel. The final extract was analysed by GLC on a packed column with a microcoulometric nitrogen detector.

Fortified control samples were analysed to demonstrate the limits of determination, but the results are not acceptable because extensive corrections were made for column efficiency, typically 60%. The results of dosing with carbofuran and 3-hydroxy-carbofuran at 5 ppm are shown in Table 57. Residues were detected only in the gizzards of hens dosed with carbofuran and were below the LOD.

Table 57. Residues in poultry tissues and eggs from the oral dosing of hens with carbofuran or 3-hydroxy-carbofuran at the equivalent 5 ppm in the diet for 70 consecutive days and the recovery of analytes from control samples fortified at 0.05 mg/kg.

Sample	Compound	Analytical recovery, %[1]	carbofuran, mg/kg	3-hydroxy-carbofuran, mg/kg
Egg	carbofuran	51 58 58	<0.05 (14 and 56 days)	<0.05
	3-hydroxy-carbofuran	38 35 33	<0.05 (14 and 56 days)	<0.05
Muscle	carbofuran	63	<0.05	<0.05
	3-hydroxy-carbofuran	52	<0.05	<0.05
Liver	carbofuran	46	<0.05	<0.05
	3-hydroxy-carbofuran	55	<0.05	<0.05

Sample	Compound	Analytical recovery, %[1]	carbofuran, mg/kg	3-hydroxy-carbofuran, mg/kg
Gizzard	carbofuran	64	<0.05 (0.01)	<0.05 (0.01)
	3-hydroxy-carbofuran	60	<0.05	<0.05

[1]Column efficiency corrections (81% for carbofuran, 59% for 3-hydroxy-carbofuran in eggs) were removed

In a feeding study in the USA with cows in 1994 (Chen, 1995a) carbosulfan (not carbofuran) was fed to lactating dairy cattle for 28 consecutive days at rates equivalent to 1, 3, 10 and 50 mg/kg in the diet. The study is fully described in the monograph on carbosulfan. In summary, carbofuran was not found in any milk, skim milk, cream or tissue samples at any of the 4 feeding concentrations, where the limit of detection was estimated as 0.005 mg/kg for milk and 0.010 mg/kg for tissues and cream. The metabolite 3-keto-carbofuran was detected only in one liver sample at 0.023 mg/kg from the 50 ppm group, and 3-hydroxy-carbofuran was detected in most milk samples from the 50 ppm group, at 0.007-0.030 mg/kg, and in one from the 10 ppm group (day 4, 0.007 mg/kg). Total carbamate residues reached a plateau at about 0.03 mg/kg from days 1 to 21. At the 50 ppm feeding level 3-hydroxy-carbofuran was detected in the kidneys (0.090, 0.13 mg/kg), liver (0.047, 0.060 mg/kg) and muscle (0.020, 0.030 mg/kg), but not in fat. In the 10 ppm group the 7-phenol (0.057 mg/kg) and 3-hydroxy-7-phenol (0.012 mg/kg) were found in the kidneys.

FATE OF RESIDUES IN STORAGE AND PROCESSING

In storage

No data were submitted.

In processing

Sorghum. A processing study on sorghum was reported from the USA (Shevchuk and Singer, 1994). In Texas and Kansas, Furadan 4F was applied in-furrow at planting at 3.6 kg ai/ha and as two broadcast foliar sprays at 2.8 kg ai/ha. The total seasonal application of 9.2 kg ai/ha was four times the GAP limit. The PHI was 63 days in Texas and 87 days in Kansas. Grain samples from both locations were analysed by the method of Barros. The Texas grain sample contained no detectable residue of carbofuran (<0.01 mg/kg). Both 3-hydroxy-carbofuran and the 3-hydroxy-7-phenol were detected at about 0.01 mg/kg. No residues were detected in two of three Kansas grain samples; the 3-hydroxy-7-phenol was detected at about 0.01 mg/kg in one sample. Only the Texas grain was processed.

The grain was dried and cleaned by aspiration and screening, then abrasively milled thereby removing the bran and generating decorticated seeds and grits. Eighteen kg of raw grain yielded 17.1 kg dried grain and 16.8 kg cleaned seed, of which 6.8 kg was dry-milled to 4.2 kg decorticated grain, 1.1 kg bran and 1.3 kg grits. Limits of determination of 0.03 mg/kg were established for carbofuran and each metabolite by the analysis of fortified control samples of grain, decorticated seed, grits and bran. The minimum recovery was 65% of 0.03 mg/kg 7-phenol from grain and the maximum 109% of 0.03 mg/kg 3-hydroxy-carbofuran from bran. No residues were detected (<0.01 mg/kg) in decorticated seed or grits. The 7-phenol and 3-hydroxy-7-phenol were found in bran at estimated concentrations of 0.02 mg/kg each. CLICK HERE to continue

Sugar beet. In a sugar beet processing study in the USA (Stearns, 1986d) Furadan 15G at 4.5 kg ai/ha was applied once to the soil in an 18-cm band at planting in Colorado in 1985. The beets were harvested 176 days after application and samples were analysed by the method of Barros. Limits of determination of 0.05 mg/kg were demonstrated for carbofuran and each metabolite by the analysis of fortified controls. The recoveries at 0.05 mg/kg (single samples) were carbofuran 84%, 3-keto-carbofuran 84%, 3-hydroxy-carbofuran 92%, 7-phenol 56%, 3-keto-7-phenol 100%, and 3-hydroxy-7-phenol 100%. The recovery of the 7-phenol was improved at 0.10 mg/kg to 66%. No carbofuran or metabolite was detected in any sample, where the limit of detection was estimated to be 0.01 mg/kg for the carbamates and 0.02 mg/kg for the phenols. Representative chromatograms were provided.

The beets were commercially processed into cossettes, dehydrated pulp, molasses and sugar. The analysis of fortified controls showed a limit of determination of 0.05 mg/kg for each analyte in each type of sample. The minimum recovery was 62% (7-phenol in sugar) and the maximum 138% (3-keto-7-phenol in molasses). Carbamates and phenols were undetectable (<0.01 mg/kg carbamates, <0.02 mg/kg phenols) in cossettes and dehydrated pulp. The 3-keto-7-phenol was detected in molasses and sugar at an estimated concentration of 0.03 mg/kg. A concentration factor could not be calculated as the raw commodity did not have detectable residues.

Potatoes. In a processing study in the USA (Shevchuk, 1995b) Furadan 4F was applied to a plot in Washington State in 1993, one in-furrow at planting (6.7 kg ai/ha, 36 g/l) and three times as a broadcast spray post-emergence at 2.2 kg ai/ha, 12 g/l. The total application was 13.4 kg ai/ha, twice the GAP rate. The PHI was 21 days. The potatoes were processed in a laboratory-scale simulation of commercial processes into chips and granules (dehydrated potatoes). Samples of tubers, chips, granules, wet peel and dry peel were analysed by the method of Barros, with limits of determination and detection of 0.05 and 0.01 mg/kg for each analyte in each commodity. The limit of detection was corroborated by sample chromatograms. The results are shown in Table 58. The phenol metabolites were concentrated 1.7 times in potato chips and 5 times in both granules and dry peel. Processing factors could not be calculated for the carbamates, because none of the samples contained quantifiable levels.

Table 58. Carbofuran and metabolites in or on processed potato products after treatment of a Washington potato plot at 13.4 kg ai/ha.

Sample	Residue, mg/kg					
	Carbofuran	3-K-CF	3-OH-CF	7-Phenol	3-K 7-P	3-OH-7-P
Tubers	<0.05	<0.05	<0.05	<0.05 (0.03)	<0.05	<0.05
Chips	<0.05	<0.05	<0.05	0.05	<0.05	<0.05 (0.01)
Granules	<0.05	<0.05	<0.05	0.14	(0.01)	<0.05 (0.02)
Wet peel	<0.05	<0.05	<0.05	<0.05 (0.02)	<0.05	<0.05
Dry peel	<0.05	<0.05	<0.05	0.12	<0.05 (0.02)	<0.05 (0.03)

Maize. A processing study on field corn (maize) was reported from the USA (Schreier, 1990b). A plot in Illinois was treated with Furadan 15G in-furrow at planting at 4.4 kg ai/ha. This was followed by one foliar treatment at the whorl stage with Furadan 15G at 3.4 kg ai/ha and two applications of

Furadan 4F at 3.4 kg ai/ha, a total application of 15 kg ai/ha (3 times the GAP rate); PHI was 42 days. The maize was processed by both wet and dry milling procedures and residues were determined in grain, grits, meal, flour, crude oil and refined (edible) oil after dry milling, and in starch, crude oil and refined oil after wet milling. Maize and the dry (non-oily) products were analysed for carbamates by the GLC method of Schreier with an NPD. The method of Leppert with GC-MS was used for the determination of carbamates and phenols in oils and of phenols in dry products. The limit of determination for each analyte in each commodity was shown to be 0.03 mg/kg by the analysis of fortified control samples and the limit of detection was estimated to be 0.01 mg/kg. The recoveries from fortified control samples and the results of the trial are shown in Table 59. No concentration of carbofuran or its metabolites was found in any processed fraction.

Table 59. Carbofuran and metabolites in or on maize and its milling fractions after treatment of a corn field with Furadan 15G and 4F at a total rate of 15 kg ai/ha, PHI 43 days. Illinois, USA.

Sample	Residue, mg/kg Recovery from controls fortified at 0.03 mg/kg					
	Carbofuran	3-K-CF	3-OH-CF	7-Phenol	3-K-7-P	3-OH-7-P
Grain	<0.03 103	<0.03 109	<0.03 109	<0.03 (0.01) 84	<0.03 90	<0.03 (0.03) 77
Dry Milling						
Medium Grits	<0.03 99	<0.03 93	<0.03 94	<0.03 69	<0.03 67	<0.03 (0.02) 62
Meal	<0.03 79	<0.03 104	<0.03 83	<0.03 96	<0.03 98	<0.03 (0.01) 85
Flour	<0.03 115	<0.03 117	<0.03 81	<0.03 60	<0.03 61	<0.03 (0.01) 60
Crude Oil	<0.03 61	<0.03 98	<0.03 96	<0.03 52	<0.03 91	<0.03 91
Refined oil	<0.03 68	<0.03 92	<0.03 63	<0.03 64	<0.03 84	<0.03 76
Wet Milling						
Starch	<0.03 97	<0.03 105	<0.03 76	<0.03 67	<0.03 70	<0.03 66
Crude Oil	<0.03 75	<0.03 99	<0.03 93	<0.03 71	<0.03 96	<0.03 101
Refined oil	<0.03 91	<0.03 111	<0.03 91	<0.03 63	<0.03 89	<0.03 80

In a second processing study (wet milling only) in the USA (Brooks and Arabinick, 1995) Furadan 4F was applied to maize in Iowa in 1994 at planting in-furrow at 4.4 kg ai/ha (81 g/l), at the whorl stage at 3.4 kg ai/ha (32 g/l), and as two broadcast foliar applications, each at 3.4 kg ai/ha (20 g/l). The total application was 15 kg ai/ha (3 times the GAP rate) and the PHI was 67 days. The residues in mature corn and starch were determined by the method of Barros, with limits of determination established at 0.03 mg/kg by the analysis of fortified controls. Recoveries from grain ranged from 72% (0.03 mg/kg 3-hydroxy-7-phenol) to 105% (0.03 mg/kg 3-hydroxy-carbofuran), and from starch from 68% (0.03 mg/kg 7-phenol to 123%; 0.03 mg/kg 3-hydroxy-7-phenol). The grain was found to contain detectable amounts of 3-hydroxy-carbofuran (estimated at 0.01 mg/kg) and quantifiable amounts of 3-hydroxy-7-phenol, 0.03 mg/kg. The starch contained no detectable residues, with the limit of detection estimated to be 0.01 mg/kg for each analyte. Carbamate residues were reduced in processing maize to starch, but a factor could not be determined.

Rice. A processing study was conducted in the USA (Shevchuk, 1995a). Rice in an Arkansas field was treated with Furadan 3G at 3.4 kg ai/ha (5 times the GAP rate) on the first day of permanent flooding, or about one month after planting. Mature rice was harvested 110 days after the treatment and processed by a batch procedure that closely followed standard commercial practice into polished rice, hulls and bran. The treated rough rice (110 kg) was dried (105 kg), aspirated and screened, yielding 23 kg of rough rice. This was dehulled and separated into hulls (3.9 kg), brown rice (18 kg) and unhulled rice (0.41 kg). The brown rice was debranned, yielding bran (2.4 kg) and white milled rice (16 kg). The rough rice and the processed fractions were analysed by the method of Barros. Duplicate control samples fortified at 0.03 mg/kg were analysed to establish the limits of determination. The lowest recovery was 62% for carbofuran in hulls and the highest 124% for 3-hydroxy-7-phenol in grain. Treated grain contained detectable amounts of 3-hydroxy-carbofuran (0.02 mg/kg) and the 3-hydroxy-7-phenol (0.02 mg/kg) and a quantifiable amount of the 7-phenol (0.05 mg/kg). Polished rice contained no detectable residues. Bran contained detectable levels of 3-keto-7-phenol (0.02 mg/kg, 2-fold concentration) and quantifiable levels of 7-phenol (0.42 mg/kg, 8-fold concentration) and 3-hydroxy-7-phenol (0.04 mg/kg, 2-fold concentration). Hulls contained quantifiable concentrations of carbofuran (0.02 mg/kg, 2-fold concentration), 3-keto-carbofuran (0.02 mg/kg, 2-fold concentration), 3-hydroxy-carbofuran (0.05 mg/kg, 4-fold concentration), 7-phenol (0.10 mg/kg, 2-fold concentration), 3-keto-7-phenol (0.03 mg/kg, 3-fold concentration), 3-hydroxy-7-phenol (0.04 mg/kg, 2-fold concentration). Processing factors could not be determined for the carbamates.

Sunflowers. In two processing studies in the USA (Tilka, 1981,1982) plots in North Dakota (Interstate 4 Variety, oil seed type) and Minnesota (Royal Hybrid Variety, confectionary type) were treated at planting with Furadan 10 G at 2.2 kg ai/ha and the plants were treated four times with foliar aerial applications of Furadan 4F at 0.56 kg ai/ha. A total application of 4.5 kg ai/ha. The North Dakota crop was harvested 26 days after the last application and the Minnesota crop after 50 days. The seeds were processed by simulated commercial procedures. Confectionary seeds were cracked and separated into hulls and kernels. Oil seed kernels were extracted to obtain crude oil and extracted meal. The crude oil was refined, bleached and deodorized to yield edible oil and soapstock. The seed and processed fractions were analysed for carbofuran and 3-hydroxy-carbofuran by the method of Schreier with the clean-up procedures of Leppert. Control samples were fortified at 0.05 mg/kg (0.1 mg/kg for confectionary seed) and analysed by the trial method. The limit of detection was estimated as 0.01 mg/kg for carbofuran and 3-hydroxy-carbofuran in all samples except soapstock, where it was 0.02 mg/kg. The results are shown in Table 60. The residue levels increased slightly in hulls and meal only.

Table 60. Carbofuran and 3-hydroxy-carbofuran in processed fractions of sunflower seed, and recoveries from fortified control samples.

Fraction	Residue, mg/kg, and [processing factor]		Recovery, %	
	Carbofuran	3-OH -CF	Carbofuran	3-OH -CF

	Residue, mg/kg, and [processing factor]		Recovery, %	
Oil seed	0.10	0.05	76 (0.05 mg/kg)	68 (0.05 mg/kg)
Edible oil	<0.05 (0.01) [0.1]	<0.05 [1]	88 (0.05 mg/kg)	100 (0.05 mg/kg)
Hulls	0.12 [1.2]	0.05 [1]	80 (0.05 mg/kg)	72 (0.05 mg/kg)
Soapstock	<0.05 [0.2]	<0.05 [1]	58 (0.05 mg/kg)	100 (0.05 mg/kg)
Extracted meal	0.10 [1]	0.09 [1.8]	68 (0.05 mg/kg)	76 (0.05 mg/kg)
Confectionary seed	<0.1 [0.06]	<0.1 [0.02]	93 (0.10 mg/kg)	67 (0.10 mg/kg)
Hulls	0.07 [1.2]	<0.05	-	-

Abbreviated compound names: see Figure 1

Cotton. In a processing study in the USA. (Shevchuk, 1994b) Texas cotton was treated with Furadan 4F in two broadcast foliar applications, each at 1.4 kg ai/ha (19 g/l), a total of 5 times the GAP rate. The cotton was harvested at the bloom and bolls growth stage, 27 days after the second treatment. Ginned and delinted cotton seed, hulls, meal and crude oil were analysed for carbofuran and its carbamate and phenol metabolites by the method of Barros. Soapstock was analysed for the phenol metabolites only. The method was validated at 0.03 mg/kg for each analyte in each sample. The recoveries from duplicate controls fortified at 0.03 mg/kg and the results of the trial are shown in Table 61. Sample chromatograms were included for the phenol but not for the carbamate determinations. There was no concentration of residues except in soapstock, where the 7-phenol was concentrated by a factor of about 7 from an estimated 0.01 mg/kg.

In the processing operation 48.2 kg cotton yielded 15.8 kg kernels and 6.45 kg hulls and the kernels yielded 3.7 kg crude oil and 11.4 kg meal.

Table 61. Carbofuran and metabolites in processed fractions of cotton seed after the foliar application of Furadan 4F (2 x 1.4 kg ai/ha, 27-day PHI), and recoveries from fortified control samples.

Sample	Residue, mg/kg [Processing factor] Recovery, %					
	carbofuran	3-K-CF	3-OH -CF	7-Phenol	3-K 7-P	3-OH-7-P
Ginned cotton seed	0.06 73, 75	<0.03 81, 102	0.19 56, 68	<0.03 (0.01) 106, 106	<0.03 94, 97	<0.03 64, 66
Delinted cotton seed	0.03 [0.5] 84, 88	<0.03 111, 112	<0.03 (0.01) [0.05] 63, 72	<0.03 90, 95	<0.03 103, 113	<0.03 73, 81
Hulls	<0.03 (0.02) [0.4] 72, 82	<0.03 69, 75	<0.03 (0.01) [0.05] 70, 71	<0.03 77, 93	<0.03 96, 101	<0.03 61, 71

Meal	<0.03 (0.02) [0.4] 90, 99	<0.03 85, 86	<0.03 (0.01) [0.05] 68, 71	<0.03 84, 91	<0.03 122, 129	<0.03 122, 128
Crude oil	<0.03 (0.02) [0.4] 90, 101	<0.03 103, 112	<0.03 [0.05] 70, 75	<0.03 (0.01) 60, 61	<0.03 74, 79	<0.03 84, 85
Soapstock	-	-	-	0.07 [7] 57, 64	<0.03 84, 94	<0.03 78, 92

Abbreviated compound names: see Figure 1

Sugar cane. A processing study was carried out in El Salvador (Stearns, 1986c). In the 1985-1986 growing season, ratoon sugar cane was treated twice with Furadan 10G at 2.5 kg ai/ha, as a banded treatment after the 1985 harvest and as a broadcast application about 6 months later, the total application of 5 kg ai/ha being 1.8 times the GAP rate. Mature sugar cane harvested 169 days after the second treatment was processed into brown sugar and molasses, but the process was not described and the molasses were not defined as either blackstrap or edible molasses. Brown sugar was also not defined, but was presumably unrefined sugar, not the commercially available brown sugar. The samples were analysed by the method of Schreier, with an MSD for the phenols and an NPD for the carbamates. Limits of determination were established for the processed fractions but not for the raw cane. Acceptable recoveries were reported at 0.05 mg/kg fortification for the carbamates from brown sugar and molasses and the phenols from molasses and at 0.10 mg/kg for the phenols from brown sugar. Limited chromatographic information was provided. The analyses showed no residues of carbofuran, 3-keto-carbofuran or 3-hydrdoxy-carbofuran in the cane, molasses or brown sugar. The 7-phenol was reported as 0.05 mg/kg in cane, 0.06 mg/kg in molasses (1.2-fold concentration) and 0.12 mg/kg in brown sugar (2.4-fold concentration). The residues of the 3-keto-7-phenol were 0.03 mg/kg in cane, 0.06 mg/kg in molasses (2-fold concentration) and 0.08 mg/kg in brown sugar (2.7-fold concentration), and of the 3-hydroxy-7-phenol 0.05 mg/kg in cane, 0.08 mg/kg (1.6-fold concentration) in both molasses and brown sugar. A limit of detection of 0.02 mg/kg was claimed for all analytes. Processing factors for the carbamate residues could not be estimated as neither the raw nor the processed commodities contained carbamates.

Coffee. In a processing study reported from Minas Gerais, Brazil (Brooks, 1996b) Furadan 5G was applied twice at 3.0 g ai/bush to the soil round coffee plants (Catuai, 1400 cova/ha), the total of 6.0 g ai/bush being twice the GAP rate. The first application (in 1994) was 30 days after flowering and the second (1995) about 6 months later. The PHI was 30 days. The green coffee beans were processed into instant coffee and ground roasted coffee in a laboratory scale operation designed to reflect commercial processing. Green beans (13.8 kg) were roasted (177-221°C hot air for 6 minutes) and a proportion was ground. The remaining beans were brewed and the extract freeze-dried. The spent grounds were press-brewed and the brew added to the extract. The green beans, ground coffee and instant coffee were analysed by the method of Barros. Limits of determination of 0.05 mg/kg for carbofuran and its metabolites on coffee beans only were demonstrated by the analysis of fortified control beans in triplicate. The ranges of recovery were reported as carbofuran 72-94%; 3-keto-carbofuran 64-97%, 3-hydroxy-carbofuran 92-96%, 7-phenol 92-128%, 3-keto-7-phenol 106-138%, and 3-hydroxy-7-phenol 52-82%. The limit of detection was claimed to be 0.01 mg/kg for each analyte. Green bean coffee had a measurable residue of 3-hydroxy-carbofuran, 0.22 mg/kg. Neither instant coffee nor roasted beans contained carbamates (<0.01 mg/kg, processing factor 0.05). Both the green beans and the processed commodities contained all the three phenol metabolites with higher levels in the processed commodities. The phenolic residues are shown in Table 62.

Table 62. Residues of phenol metabolites in or on green beans, roasted coffee and instant coffee from the application of Furadan 5G to the soil around coffee bushes in brazil, 2 x 3 g ai/bush, 30-day PHI.

Commodity	7-phenol		3-keto-7-phenol		3-hydroxy-7-phenol	
	Residue, mg/kg	Processing factor	Residue, mg/kg	Processing factor	Residue, mg/kg	Processing factor
Green beans	0.14	-	0.07	-	0.28	-
Ground roast	0.17	1.2	0.08	1.1	0.45	1.6
Instant	0.47	3.4	0.21	3.0	0.43	1.5

Pimento peppers. In a processing study in the USA (Anon., 1971). Furadan 10 G was applied to pimento pepper plots in Delaware in two side-dress treatments at 2.2 and 3.4 kg ai/ha . Pimentos were harvested at maturity and pickled by an undefined method. Residues of carbofuran and 3-hydroxy-carbofuran were determined by the method of Schreier, using a gas chromatograph equipped with a Coulson nitrogen detection system. The limits of determination were established by the analysis of fortified peppers and pickled peppers. At 0.05 mg/kg the recoveries from peppers were 114 and 132% for carbofuran and 94 and 70% for 3-hydroxy-carbofuran. At 0.20 mg/kg the recoveries from fortified pickled peppers (6 replicates) were 89 $\pm$ 9.5% for carbofuran and 62 $\pm$ 10% for 3-hydroxy-carbofuran. Finite residues were found on the raw peppers which were reduced by pickling. The results are shown in Table 63.

Table 63. Residues of carbofuran and 3-hydroxy-carbofuran in or on pimento peppers and pickled peppers from the application of Furadan 10 G as a banded treatment (2.2 + 3.4 kg ai/ha) in 1971 in Delaware, USA.

PHI, days	Residue, mg/kg			
	Raw peppers		Pickled peppers	
	carbofuran	3-hydroxy-carbofuran	carbofuran	3-hydroxy-carbofuran
21	0.35	0.13	0.19	0.08
	0.35	0.23	0.19	0.08
39	0.10	0.15	<0.2	<0.2

Grapes. Furadan 4F was applied as a broadcast treatment at the GAP rate of 11 kg ai/ha (29 g/l) to grape plants (Pinot Blanc) in California in 1985 (Stearns, 1986b). Grapes were harvested at maturity after a PHI of 198 days and processed into juice and wet and dry pomace. No information was supplied on the processing. The samples were analysed by the method of Schreier, carbamates being determined with an NPD and phenols with an MSD. Limits of determination were demonstrated by the analysis of duplicate control samples of grapes fortified at 0.10 mg/kg and juice and dry pomace at 0.05 mg/kg with carbofuran and each of the carbamate and phenol metabolites. Levels of detection of 0.01 and 0.02 mg/kg were claimed for the carbamates and phenols respectively. The results are shown in Table 64. Residues were not concentrated in the juice but were concentrated in dry pomace (3-hydroxy-carbofuran 2.8-fold and total phenols 3.9-fold).

Table 64. Residues of carbofuran and its metabolites in grapes, juice and pomace from the application of Furadan 4F, 11 kg ai/ha, 198-day PHI and recoveries from fortified control samples.

Commodity	Residues, mg/kg Recoveries, %[1]					
	Carbofuran	3-K-CF	3-OH-CF	7-Phenol	3-K-7-P	3-OH-7-P
Grapes	<0.1 83 (76, 90)	<0.1 98 (78, 117)	0.20 98 (79, 117)	<0.1 (0.07) 87	0.1 93	0.14 0
Juice	<0.05 84 (84, 84)	<0.05 90 (86, 94)	0.18 98 (102, 94)	<0.05 (0.04) 89 (96, 82)	0.07 96 (104, 87)	0.12 89 (97, 81)
Dry Pomace	<0.05 (0.01) 67 (62, 72)	<0.05 (0.02) 68 (66, 70)	0.56 67 (62, 72)	0.32 84 (82, 85)	0.44 104 (103, 105)	0.44 75 (74, 76)

[1]Grapes fortified at 0.1 mg/kg, juice and pomace at 0.05 mg/kg with each analyte

Residues in the edible portion of food commodities

No data were submitted.

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

A farm gate study was submitted from Korea (Chon Chae-gu, 1996) in which seven domestic commodities were analysed for carbofuran in the period April 1995-January 1996. A total of 210 samples of rice, carrots, maize, green onions, potatoes, peanuts and garlic were collected from farms (only brown rice) or markets (all commodities except rice) near the sites of production in various growing regions of the country. Residues were analysed by the method of Leppert, but the ethoxylation step was omitted. A 10 m RSL-300TM or a 25 m methyl silicone capillary column was used with a nitrogen-phosphorus detector. Calibration was by external standards and only carbofuran was determined, although several sample chromatograms showed a peak labelled 3-hydroxy-carbofuran. Results are shown in Table 65.

Table 65. Monitoring of carbofuran residues in seven domestic commodities in Korea, 1995-1996.

Commodity	No. of samples	No. of detections[1]	Detection frequency, %	Carbofuran, mg/kg	Recoveries	
					Fortification, mg/kg	%
Brown rice	60	1	1.7	<0.5 (0.06)	0.5	106, 107, 108
Garlic	40	2	5.0	<0.1 (0.07, 0.13)	0.1	95.5, 91.3, 95.8, 96,1, 97.6, 94.9
Peanuts	20	0	0	<0.1	0.1	98.4, 92.4, 96.2
Potato	10	0	0	<0.1	0.1	100.1, 92.6, 97.3
Green onions	40	0	0	<0.25	0.25	81.9, 83.5, 83.4
Carrots	20	2	10	<0.25 (0.015, 0.015)	0.25	92.0, 92.8, 91.2
Corn (maize)	20	0	0	<0.25	0.25	95.0, 94.5, 94.0

[1] Limit of detection estimated at 0.05 mg/kg for rice, 0.0125 mg/kg for carrots, maize and green onions and 0.02 mg/kg for garlic, peanuts and potatoes. These limits were based on injections of standards and do not reflect the effect of the matrix

NATIONAL MAXIMUM RESIDUE LIMITS

Maximum residue limits, which have been established in 31 countries and the EU are shown below.

Country	Commodity	MRL, mg/kg	Remark
Argentina	Bean	0.1	
	Eggs	0.05	
	Fat	0.05	
	Maize grain	0.1	
	Meat	0.05	
	Meat by-products	0.05	
	Milk	0.05	
	Potato	0.5	
	Sorghum grain	0.1	
	Sweet corn	0.1	
	Tomato	0.1	
Australia			Sum of carbofuran and 3-hydroxy-carbofuran, expressed as carbofuran
	Animal feed	2	
	Banana	0.1	
	Eggs	0.05*	
	Meat	0.05*	
	Meat by-products	0.05*	
	Milk	0.05*	
	Poultry meat	0.05*	
	Rice	0.2	
	Sugar cane	0.1*	
	Wheat	0.2	
Austria			Carbofuran, 3-hydroxy-carbofuran and its conjugates, expressed as carbofuran
	Beet, Sugar	0.2	

Country	Commodity	MRL, mg/kg	Remark
	Coffee		
	Grape	0.2	
	Maize	0.2	
	Meat	0.05	
	Milk	0.05	
	Sunflower seed	0.1	
	Potato	0.5	
	Turnips	1	
Belgium			Carbofuran and 3-hydroxy-carbofuran, expressed as carbofuran
	Plant commodities	0	<0.1 mg/kg
Brazil	Coffee	0.1	
	Cotton seed	0.1	
	Peanut	0.1	
	Rice	0.2	
Canada			Sum of carbofuran and 3-hydroxy-carbofuran, expressed as carbofuran
	Alfalfa	0.1	Negligible tolerance
	Banana	0.1	Negligible tolerance
	Barley	0.1	Negligible tolerance
	Beet, Sugar	0.1	Negligible tolerance
	Clover	0.1	Negligible tolerance
	Coffee	0.1	Negligible tolerance
	Cucumber	0.1	Negligible tolerance
	Eggs	0.1	Negligible tolerance
	Grape	0.1	Negligible tolerance
	Maize	0.1	Negligible tolerance
	Meat	0.1	Negligible tolerance
	Melon	0.1	Negligible tolerance
	Milk	0.1	Negligible tolerance
	Oats	0.1	Negligible tolerance
	Peanut	0.1	Negligible tolerance
	Pepper, Sweet	0.5	
	Potato	0.5	
	Pumpkin	0.1	Negligible tolerance
	Rape seed	0.1	Negligible tolerance
	Rice	0.1	Negligible tolerance
	Rutabaga	0.5	
	Strawberry	0.4	
	Sunflower	0.1	Negligible tolerance
	Tomato	0.1	Negligible tolerance
	Wheat	0.1	Negligible tolerance
Chile			Sum of carbofuran and 3-hydroxy-carbofuran, expressed as carbofuran
	Animal fat	0.05*	
	Barley	0.1*	
	Beat, sugar	0.1*	
	Mammalian, meat	0.05*	
	Milk	0.05*	
	Oilseed	0.1*	
	Potato	0.5	
	Rice, husked	0.2	
	Tomato	0.1*	
	Wheat	0.1*	
Cyprus	Banana	0.1	
	Beets, sugar	0.1	
	Cereals	0.1	

Country	Commodity	MRL, mg/kg	Remark
	Meat	0.05	
	Milk	0.05	
	Potato	0.05	
	Rice	0.02	
	Strawberry	0.1	
	Tomato	0.1	
Denmark			Sum of carbofuran and 3-hydroxy-carbofuran, expressed as carbofuran
	Banana	0.1	
	Potato	0.5	
	Turnips, swedes	0.1	
European Union			Sum of carbofuran and 3-hydroxy-carbofuran, expressed as carbofuran
	Banana	0.1*	T
	Cereals	0.1*	
	Cotton seed	0.1*	T
	Cucurbits	0.1*	
	Egg products	0.1*	
	Eggs	0.1*	T
	Grape	0.1*	T
	Meat	0.1*	T
	Meat by-products	0.1*	
	Meat, preparations of	0.1*	
	Melon	0.1*	T
	Milk	0.1*	
	Milk products	0.1*	
	Oats	0.1*	T
	Peanut	0.1*	T
	Potato	0.1*	T
	Rape seed	0.1*	T
	Rice	0.1*	T
	Rubus species (cane fruit)	0.1*	
	Rutabaga	0.01	T
	Soya	0.1*	T
	Strawberry	0.1*	T
	Sunflower seed	0.1*	T
	Sweet corn	0.1*	T
	Turnip, edible	0.01	T
Finland			Sum of carbofuran and 3-hydroxy-carbo expressed as carbofuran
	Potato	0.5	
France			Sum of carbofuran and 3-hydroxy-carbo expressed as carbofuran
	Maize forage	0.5	T
	Maize grain	0.1	
	Rape	0.5	
	Soya	0.2	
	Strawberry	0.5	
	Sunflower	0.5	
	Sweet corn	0.5	
	Sweet corn, forage	0.5	T
Germany			Carbofuran, 3-hydroxy-carbofuran and its conjugates, expressed as carbofuran
	Animal fat	0.05	
	Beet, Sugar	0.2	
	Egg products	0.05	
	Eggs	0.05	

Country	Commodity	MRL, mg/kg	Remark
	Meat	0.05	
	Meat, preparations of	0.05	
	Milk	0.05	
	Milk products	0.05	
	Other plant commodities	0.1	
	Potato	0.5	
Hungary	Other plant commodities	0.1	
India			Sum of carbofuran and 3-hydroxy-carbofuran, expressed as carbofuran
	Cereal grains	0.1	
	Fruit	0.1	
	Oilseed	0.1	
	Sugar cane	0.1	
	Vegetables	0.1	
	Meat	0.1	Fat basis
	Milk	0.05	Fat basis
	Milk products	0.05	Fat basis
	Poultry	0.1	
Italy	Beet, Sugar	0.1	
	Maize	0.1	
	Potato	0.1	
Kenya	Alfalfa		T
	Alfalfa hay	20	T
	Banana	0.1	T
	Barley	0.1	T
	Beet, Sugar, leaf	0.2	T
	Beet, Sugar, root	0.1	T
	Cattle fat	0.05*	T
	Cattle meat	0.05*	T
	Cattle meat by-products	0.05*	T
	Coffee	0.1	T
	Goat fat	0.05*	T
	Goat meat	0.05*	T
	Goat meat by-products	0.05*	T
	Horse fat	0.05*	T
	Horse meat	0.05*	T
	Horse meat by-products	0.05*	T
	Maize	0.1	T
	Maize, forage		T
	Milk	0.05*	T
	Oats	0.1	T
	Oilseed	0.1	T
	Peanut kernel	0.1	T
	Pig fat	0.05*	T
	Pig meat	0.05*	T
	Pig meat by-products	0.05*	T
	Potato	0.5	T
	Rice, husked	0.2	T
	Sheep fat	0.05*	T
	Sheep meat	0.05*	T
	Sheep meat by-products	0.05*	T
	Sorghum	0.1	T
	Soya	0.2	T
	Strawberry	0.1	T
	Sugar cane	0.1	T
	Sweet corn kernels	0.1	T
	Tomato	0.1	T

Country	Commodity	MRL, mg/kg	Remark
	Wheat	0.1	T
Luxembourg	Maize	0.1	Sum of carbofuran and 3-hydroxy-carbofuran, expressed as carbofuran
Malaysia	Banana	0.1	
	Grape	0.4	
	Strawberry	0.5	
Mexico	Alfalfa	10	
	Alfalfa hay	40	
	Banana	0.1	
	Barley	0.2	
	Coffee	0.1	
	Cucumber	0.4	
	Grape	0.4	
	Maize	0.1	
	Melon	0.4	
	Oats	0.2	
	Peanut	4	
	Pepper, Cayenne	1	
	Pepper, Sweet	1	
	Potato	2	
	Rice	0.2	
	Sorghum	0.1	
	Soya	1	
	Strawberry	0.5	
	Sugar cane	0.1	
	Wheat	0.2	
Netherlands			Sum of carbofuran and 3-hydroxy-carbofuran, expressed as carbofuran
	Potato	0.5	
	Rice	0.2	
	Soya	0.2	
	Strawberry	0.2	
	Other plant commodities	0.1*	
Paraguay	Rice	0.2	
	Tomato	0.1	
Portugal	Potato	0.5	Sum of carbofuran and 3-hydroxy-carbofuran, expressed as carbofuran
South Africa			Sum of carbofuran and 3-hydroxy-carbofuran, expressed as carbofuran
	Cruciferae	0.5	
	Maize	0.1	
	Maize forage	0.2	
	Potato	0.05	
	Sorghum	0.1	
	Sugar cane	0.1	
	Sunflower seed	0.1	
	Wheat	0.1	
South Korea	Banana	0.1	
	Barley	0.1	
	Coffee	0.1	
	Cotton seed	0.1	
	Cucumber	0.5	
	Grape	0.5	
	Maize	0.1	
	Oats	0.1	
	Peanut	0.5	
	Potato	0.5	

Country	Commodity	MRL, mg/kg	Remark
	Pumpkin	0.5	
	Rice	0.2	
	Sorghum grain	0.1	
	Soya	0.2	
	Strawberry	0.1	
	Sunflower seed	0.1	
	Tomato	0.1	
	Wheat	0.1	
Spain			Sum of carbofuran, carbosulfan and 3-hydroxy-carbofuran, expressed as carbofuran
	Sweet corn	0.1*	
	Cotton seed	0.1*	
	Cucurbits with edible peel	0.1*	
	Cucurbits with inedible peel	0.1*	
	Forage crops and straw	0.1*	
	Grape	0.1*	
	Maize forage	2	
	Oats	0.1*	
	Oil seed	0.1*	
	Other pulses	0.1*	
	Peanut	0.1*	
	Potato	0.2	
	Rape seed	0.1*	
	Rice	0.1*	
	Rubus species (cane fruit)	0.1*	
	Rutabaga	0.1*	
	Solanaceae (peppers)	0.1*	
	Sorghum forage	2	
	Soya	0.1*	
	Stimulant plants (coffee)	0.1*	
	Strawberry	0.1*	
	Sugar plants	0.1*	
	Sunflower seed	0.1*	
	Turnip, edible	0.1*	
Sri Lanka	Banana	0.2	
	Gourd	1	
Sweden			Sum of carbofuran and 3-hydroxy-carbofuran, expressed as carbofuran
	Fruit	0.1	
	Potato	0.5	
	Other vegetables	0.1	
Switzerland	Beet, Sugar	0.05*	
	Maize	0.05*	
UK			Sum of carbofuran and 3-hydroxy-carbofuran, expressed as carbofuran
	Banana	0.1*	
	Barley	0.1*	
	Beet, Sugar	0.1*	
	Cereals exc. rice	0.1*	
	Cucumber	0.1*	
	Cucurbits	0.1*	
	Eggs	0.1*	
	Fat	0.1*	
	Grape	0.1*	
	Maize	0.1*	
	Meat	0.1*	
	Meat, preparations of	0.1*	

Country	Commodity	MRL, mg/kg	Remark
	Milk	0.1*	
	Milk products	0.1*	
	Oil seed	0.1*	
	Pepper, Sweet	0.1*	
	Rye	0.1*	
	Squash	0.1*	
	Tomato	0.1*	
	Wheat	0.1*	
	Wine grape	0.1*	
Uruguay	Potato	0.5	
	Rice	0.2	
	Tomato	0.1	
USA			Carbofuran, 3-hydroxy-carbofuran
	Alfalfa		
	Alfalfa hay	20	
	Banana	0.1	
	Barley grain	0.1	
	Barley straw	1	
	Beet, Sugar	0.1	
	Beet, Sugar, top or leaves	1	
	Cattle fat	0.02	
	Cattle meat	0.02	
	Cattle meat by-products	0.02	
	Coffee	0.1	
	Cotton seed	0.2	
	Cucumber	0.2	
	Goat fat	0.02	
	Goat meat	0.02	
	Goat meat by-products	0.02	
	Gourd	0.6	
	Grape	0.2	
	Grape pomace, dried	1.5	F
	Grape, raisin	1	F
	Grape, raisin waste	3	F
	Horse fat	0.02	
	Horse meat	0.02	
	Horse meat by-products	0.02	
	Maize fodder		
	Maize forage		
	Maize grain	0.1	
	Maize, fresh	0.2	
	Melon	0.2	
	Milk	0.02	
	Oat grain	0.1	
	Oat straw	1	
	Peanut	1.5	
	Peanut hull	8	
	Peanut soapstock	3	F
	Pepper, Cayenne	0.2	
	Pepper, Sweet	0.2	
	Pig fat	0.02	
	Pig meat	0.02	
	Pig meat by-products	0.02	
	Popcorn grain	0.1	
	Potato	1	
	Pumpkin	0.6	
	Rape, canola seed	0.2 T	Until 22/02/98

Country	Commodity	MRL, mg/kg	Remark
	Rice	0.2	
	Rice straw	0.2	
	Sheep fat	0.02	
	Sheep meat	0.02	
	Sheep meat by-products	0.02	
	Sorghum fodder	0.5	
	Sorghum forage	0.5	
	Sorghum grain	0.1	
	Soya	0.2	
	Soya forage	20	
	Soya hay	20	
	Soya soapstock	1	F
	Strawberry	0.2	
	Sugar cane	0.1	
	Sunflower meal	0.6	F
	Sunflower seed	0.5	
	Sunflower soapstock	0.5	F
	Sunflower, hull	0.6	F
	Sweet corn, fresh	0.2	
	Wheat grain	0.1	
	Wheat straw	1	

*At or about the limit of determination
F: Food-additive tolerance
T: Temporary

APPRAISAL

Carbofuran, 2,3-dihydro-2,2-dimethylbenzofuran-7-yl methylcarbamate, is a widely used insecticide, nematicide, and acaracide. Its uses include seed treatment, at-plant soil application, and directed or foliar applications. A periodic review of the toxicology of carbofuran was carried out by the 1996 JMPR and the present evaluation is a periodic review of its residue and analytical aspects.

Carbosulfan produces carbofuran as a major metabolite. The periodic review of carbosulfan at the present Meeting includes an evaluation of its use on citrus fruit. In evaluating carbofuran, account was taken of its residues arising from the use of carbosulfan on citrus.

Animal metabolism

Studies were provided by the sponsors on rats, houseflies, laying hens, and lactating goats. The metabolism is similar in all species and consists of oxidation at the C-3 position and hydrolysis of the carbamate ester. The major metabolites observed in the urine from rats treated orally with single doses of carbonyl- or phenyl-labelled [^{14}C]carbofuran were 3-hydroxycarbofuran (14%), 3-ketocarbofuran (48%), the 7-phenol (20%), and the 3-hydroxy-7-phenol (1.4%). The major compounds found from the topical treatment of houseflies with radiolabelled carbofuran were carbofuran (12% internal), 3-hydroxycarbofuran (6%), and conjugated 3-hydroxycarbofuran (11%).

Hens were given 3 mg of phenyl-labelled [^{14}C]carbofuran for 7 consecutive days, about 2 mg/kg bw/day, equivalent to about 25 ppm in the feed. Eggs and tissues were collected and subjected to a series of extractions and hydrolyses. The residues in muscle and fat were negligible, and radiolabelled residues in the kidneys, liver, and eggs ranged from 0.03 to 0.15 mg/kg expressed as carbofuran. The major metabolite found in eggs was the 3-hydroxy-7-phenol (39% of the TRR).

About 5% of the TRR in the liver and kidneys was identified as the 7-phenol, and significant proportions were characterized as releasable by treatment with protease or strong acid.

[^{14}C]Carbofuran, uniformly labelled in the phenyl ring, was administered orally to goats for 7 consecutive days at a rate equivalent to 25 ppm carbofuran in the diet. Milk and excreta were collected daily, and tissues were taken within 24 hours of the final dosing. The total radioactive residue in the milk remained fairly constant (0.10 mg/kg), and residues in the fat and tissues were negligible (<0.01 mg/kg). The milk and tissues were extracted with a series of solvents and subjected to enzymatic and acid/base hydrolyses. The major metabolites released and subsequently identified in the milk were 3-hydroxycarbofuran (10% of the TRR), the 7-phenol (15% of the TRR), and the 3-keto-7-phenol (32% of the TRR). Protease released 13% and 16% of the TRR from the kidneys and liver respectively. Major metabolites in the kidneys were 3-hydroxycarbofuran (11% of the TRR) and the 3-hydroxy-7-phenol (16% of the TRR, enzyme-released).

Plant metabolism

Studies were reported on potatoes, soya beans, and maize. The major metabolites identified in potato tubers were the 7-phenol (45% of the TRR) and the 3-hydroxy-7-phenol (13%). Immature foliage contained 3-hydroxycarbofuran (23% of the TRR) and a metabolite unique to the potato, 5-hydroxycarbofuran (34%). In soya bean forage (45-day PHI), the major compounds were identified as carbofuran (11% of the TRR) and 3-hydroxycarbofuran (28%). At a longer pre-harvest interval (139 days), the beans showed a substantial residue (40% of the TRR) releasable only by enzymes and acid and base hydrolyses. Only 12-13% the residue in the beans and hay was identified. The major metabolites in the beans were 3-ketocarbofuran (5% of the TRR) and the 3-keto-7-phenol (9%). The main compounds identified in maize forage were carbofuran and 3-hydroxycarbofuran (14% and 13% of the TRR).

The metabolites identified or characterized in the plants are consistent with hydroxylation at C-3 and hydrolysis of the carbamate, as in animals. Substantial conjugation of the metabolites and incorporation of the radiolabel into plant constituents occur.

The Meeting concluded that the animal and plant metabolism studies were fully adequate and showed a common metabolic pathway.

Environmental fate

Studies were reported on aerobic soil degradation, aerobic and anaerobic aquatic degradation, soil photolysis, terrestrial field dissipation, aqueous photolysis, and aquatic field dissipation.

The major pathway of degradation of [^{14}C]carbofuran in aerobic soil was by hydroxylation and oxidation at the C-3 position, yielding 3-hydroxycarbofuran and 3-ketocarbofuran. The half-life of carbofuran was calculated to be 320 days under acidic conditions and 150 days under alkaline conditions.

In an anaerobic water/sediment study more than 50% of the [^{14}C]carbofuran was converted to the 7-phenol, which was also a major product of anaerobic aquatic degradation where the carbofuran half-life was 120 days.

The aerobic aquatic half-life in a water/sediment system at pH 5.4 was 40 days.

The photolysis half-life of carbofuran in soil was about 78 days. Carbofuran is photolytically stable in aqueous solution, with a half-life of 450-1200 days.

From the soil dissipation studies it was determined that the half-life of carbofuran at a 0-6 inch depth was 13-43 days. The aquatic field dissipation study showed a carbofuran half-life of <10 days for carbofuran in rice paddy water. Thus, transfer of carbofuran via irrigation water is not anticipated to be a serious concern.

It was shown that carbofuran can be leached from four different types of soil under vigorous conditions.

The Meeting concluded that carbofuran is readily degraded in aquatic systems and that it is somewhat persistent is soil. Degradation in soil and water involves hydroxylation at the C-3 carbon and hydrolysis of the carbamate.

Methods of residue analysis

The methods of analysis are adequate for monitoring and for use in supervised trials, and at least one multi-residue method exists which is suitable for monitoring and enforcement.

The commonly used HPLC method involves solvent extraction of the homogenized sample, purification on a solid-phase extraction column, and determination on a reverse-phase column. A post-column reactor converts the eluted methylcarbamates to an indole, which is measured fluorimetrically. The method has a demonstrated limit of determination of about 0.05 mg/kg for carbofuran, 3-hydroxycarbofuran, and 3-ketocarbofuran. The limit of determination in milk is 0.025 mg/kg. A variation of the method involves initial hydrolysis of the homogenized sample with 0.25 N HCl to release any conjugates.

Several GLC methods exist for the determination of the carbamate metabolites. A macerated sample is refluxed with 0.25 N HCl, partitioned into methylene chloride, and purified on a Florisil column. A methyl silicone capillary column and a nitrogen-phosphorus or mass spectrometric detector are used. The method may be modified by ethylating the 3-hydroxycarbofuran. Limits of determination of 0.05 to 0.10 mg/kg were demonstrated.

In an older variation of the GLC method the initial extraction of the sample is with methanol/chloroform. The residual aqueous fraction is then hydrolysed with acid. A limit of determination of 0.1 mg/kg is claimed, but recoveries of the conjugate of 3-hydroxycarbofuran were generally unacceptable below 1 mg/kg. A variation of this method did not include acid hydrolysis, and the limit of determination for carbofuran and 3-hydroxycarbofuran was 0.1 mg/kg.

Stability of residues in stored analytical samples

Information was submitted on the stability of carbofuran, 3-hydroxycarbofuran, and 3-ketocarbofuran in or on several diverse raw agricultural commodities. The Meeting concluded that carbofuran and its carbamate metabolites are stable for at least 2 years in or on frozen plant commodities and milk, and for 1 year in meat.

Definition of the residue

The residue is defined for compliance with MRLs as the sum of carbofuran and 3-hydroxycarbofuran, expressed as carbofuran. For the estimation of dietary intake the residue should be defined as the sum of carbofuran, free 3-hydroxycarbofuran and conjugated 3-

hydroxycarbofuran, expressed as carbofuran. The metabolism studies on soya beans and maize showed that the concentration of conjugated 3-hydroxycarbofuran was equal to or greater than that of 3-hydroxycarbofuran. For example, in soya bean forage (63 mg/kg of ^{14}C expressed as carbofuran) the free 3-hydroxycarbofuran was 11% of the TRR and the conjugated (acid-released) 3-hydroxycarbofuran was 17%. In the beans the concentrations were approximately equal. Where the analytical method used for a field trial did not include an acid hydrolysis step (refluxing with 0.1 N HCl) to release conjugates of 3-hydroxycarbofuran, the results were not used in the determination of the STMR levels.

Supervised trials

Residue trials were reported on numerous crops: alfalfa, bananas, Brussels sprouts, cantaloupes, cauliflower, celeriac, celery, coffee, cucumbers, grapes, head cabbages, kohlrabi, leeks, maize, oilseed plants (cotton, sunflower, rape, peanuts), onions, peppers, potatoes, rice, sorghum, soya beans, strawberries, sugar beet, sugar cane, summer squash, sweet corn, tomatoes, turnips, and wheat.

<u>Fruits</u>

<u>Citrus fruits</u>. Residues of carbofuran, 3-hydroxycarbofuran, and 3-ketocarbofuran may occur on citrus from the use of carbosulfan. On the basis of the concurrent review of carbosulfan the Meeting estimated a maximum residue level for carbofuran plus 3-hydroxycarbofuran in oranges of 0.5 mg/kg, and an STMR of 0.1 mg/kg.

<u>Grapes</u>. Field trials in the USA, Germany, and Mexico were reported. The four trials in Germany were not considered because the residue determined and the maturity of the crop samples were not clearly explained; the report consisted only of a simple summary. US GAP was used to evaluate the trials in Mexico and the USA (11.2 kg ai/ha of 4 F formulation, applied after harvest with a PHI of 200 days and soil-incorporated; pre-harvest drip irrigation with 4F at 3.4 kg ai/ha, 60-day PHI). One US and three Mexican trials complied with GAP for the vine treatment after harvest, and one US trial with GAP for the pre-harvest treatment. The residues were <0.05 mg/kg in all five trials, but five trials were considered to be insufficient for the estimation of a maximum residue level.

<u>Strawberries</u>. Supervised field trials were reported from France (0.89-1 kg ai/ha, PHI 13-48 days), the UK (2 kg ai/ha, 300-day PHI), and the USA (2.2 kg ai/ha, 250-day PHI). The results constituted two distinct sets, one for the after-harvest application to vines (UK and USA) where residues were below the limit of determination, 0.05-0.1 mg/kg, and the other with residues from <0.1 to 0.94 mg/kg (France). No information on GAP was provided for France or the UK or a neighbouring nation. The US trials conformed to US GAP, 2.2 kg ai/ha applied post-harvest after 1 October. The residues in the three trials were all 0.02 mg/kg. The results were insufficient to estimate a maximum residue level and the Meeting recommended the withdrawal of the existing CXL (0.1* mg/kg).

<u>Bananas</u>. Field trials in Spain, Central America and South America with the application of carbofuran to banana trees were reported. No residues of carbofuran plus 3-hydroxycarbofuran (<0.02-<0.10 mg/kg, n = 8) were found in any trial. GAP was available only for Spain, where the trial was according to GAP and undetectable residues were <0.02 mg/kg. Because none of the trials, some of which were at higher rates than GAP, yielded detectable residues the Meeting estimated a maximum residue level of 0.1* mg/kg, the same as the existing CXL, and an STMR of 0.1 mg/kg.

<u>Vegetables</u>

Leeks. Curaterr 200 SC was applied to the soil before planting leeks at two locations in The Netherlands. Carbamate residues were above the limit of determination in one trial, with a maximum of 0.15 mg/kg. The number of trials was inadequate to estimate a maximum residue level.

Onions. Curaterr 5G or 200 SC was applied to onions at three locations after or before sowing. The carbamate residues were below the limit of determination. There were too few trials to estimate a maximum residue level. The Meeting recommended withdrawal of the existing CXL for bulb onion (0.1* mg/kg).

Head cabbages. Two supervised field trials were reported for the application of Curaterr 200 SC to head cabbage in The Netherlands. No residues were detected (<0.1 mg/kg). Two trials are too few for the estimation of a maximum residue level and the Meeting recommended the withdrawal of the existing CXL (0.5 mg/kg).

Brussels sprouts. Again only two trials in The Netherlands were reported. The Meeting recommended the withdrawal of the existing CXL (2 mg/kg).

Cauliflower. Five trials were carried out in The Netherlands with Curaterr 200 SC applied to cauliflower plants at 0.038 g ai/plant. The mode and timing of the application were not reported. No GAP was available for The Netherlands or other EU country and the data could not be evaluated. The Meeting recommended the withdrawal of the existing CXL (0.2 mg/kg).

Kohlrabi. Two field trials were carried out in Germany with single applications of a granular formulation at 0.64 g/m 38 and 52 days after planting but no GAP was reported. The Meeting recommended the withdrawal of the existing CXL (0.1* mg/kg).

Cucumbers. Field trials were carried out in the USA. US GAP specifies the at-plant application of 2.2 kg ai/ha of a G formulation or 1.7 kg ai/ha of an F formulation. The trials were conducted at 1.1 and 3.4 kg ai/ha with both the 15 G and 4 F formulations. The lower rate is below maximum GAP and the higher exceeds it. The results from the two rates were comparable and could therefore be used to represent the GAP rate. The residues from the 1.1 kg ai/ha rate were 0.02 (6), 0.04, 0.05, 0.08, 0.09, 0.15 (2), 0.16 and 0.21 mg/kg (n = 14), and those from the 3.4 kg ai/ha rate were 0.02 (4), 0.04 (2), 0.05 (2), 0.13, 0.16, 0.18, 0.21, 0.26 and 0.29 mg/kg, n = 14. The STMR for the 3.4 kg ai/ha rate is 0.05 mg/kg, and that for the 1.1 kg ai/ha rate 0.045 mg/kg. The Meeting estimated a maximum residue level of 0.3 mg/kg and an STMR of 0.05 mg/kg from the combined results.

Cantaloupes. Supervised field trials in the USA were reported, with the application of Furadan 15G or 4F to cantaloupes at planting, with PHIs of 60-92 days. The application rates were 1.1 or 3.4 kg ai/ha. Four trials were conducted in each of seven states. GAP specifies at-plant application of the G formulation at 2.2 kg ai/ha or the F formulation at 1.7 kg ai/ha. Some trials were below and others above maximum GAP. The results from the high and low application rates were similar, and could be used to represent residues resulting from GAP applications. The residues from the 1.1 kg ai/ha rate were 0.02 (8), 0.05 (2), 0.11 (3) and 0.13 mg/kg (n = 14), and those from the higher rate 0.02 (7), 0.05 (5), 0.11 and 0.12 mg/kg (n = 14). The STMR for the 1.1 kg ai/ha rate would be 0.02 mg/kg, and for the 3.4 kg ai/ha rate 0.035 kg ai/ha. Combining the distributions, the STMR is 0.02 mg/kg. The Meeting estimated a maximum residue level of 0.2 mg/kg.

Summer squash. GAP in the USA is the same as for cucumbers and cantaloupes. Supervised field trials were carried out in seven states of the USA with the at-plant application of carbofuran 15G and 4F formulations at 1.1 and 3.4 kg ai/ha, some therefore below and some above maximum GAP. The results from the high and low application rates were similar, and the trials may be taken to represent applications according to GAP. The residues in rank order from 1.1 kg ai/ha were 0.02 (7), 0.05 (2), 0.07, 0.10, 0.11, 0.13 and 0.26 mg/kg (n = 14), and from 3.4 kg ai/ha 0.02 (5), 0.04, 0.06 (3), 0.07, 0.08, 0.09, 0.12 and 0.15 mg/kg (n = 14). The STMR for the 1.1 kg ai/ha rate is 0.035 mg/kg, and for both 3.4 kg ai/ha and for all the trials combined 0.05 mg/kg. The Meeting estimated a maximum residue level of 0.3 mg/kg and an STMR of 0.05 mg/kg.

Peppers (hot). Carbofuran was applied to the soil before planting hot peppers in two trials in the USA, with a second post-emergence side-dress application. The trials were according to, but not at the maximum, US GAP. No maximum residue level could be estimated.

Peppers (sweet). Furadan 4F was applied to sweet peppers in Canada and the USA. GAP was not available for Canada. US GAP specifies two applications of a 4 F formulation, one at-plant and the second as a side-dress, with a 21-day PHI. Each application is 3.4 kg ai/ha. The Canadian applications were in excess of US GAP at 5 x 0.56 kg ai/ha, 1-3-day PHI, and the results were not evaluated. In the US trials the application rate was ≤50% of the maximum GAP rate. The Meeting concluded that the data were insufficient to estimate a maximum residue level.

Tomatoes. Field trials were carried out in Brazil, Canada, France, Mexico, and the USA. The government of Thailand provided information on field trial conditions but did not include any analytical results. Most of the treatments were with a granular formulation applied to the soil round the plants. No GAP was reported for France, Mexico, or Canada, and the results from these countries could not be evaluated. There is no GAP in the USA. Two trials in Brazil which complied with GAP gave results of 0.05 mg/kg, but two samples are not enough to estimate a maximum residue level. The Meeting recommended withdrawal of the existing CXL (5 mg/kg).

Sweet corn (corn-on-the-cob). The findings of sixteen field trials on sweet corn were submitted from the USA. A combination of at-planting, at whorl, and foliar applications were made with granular and flowable formulations in accordance with the current label, at the maximum rate and with a minimum PHI. The commodity analysed was corn and cob, less husk. GAP was followed (1.12 kg ai/ha at-plant, followed by 4 foliar applications, each 0.56 kg ai/ha, 7-day PHI), and the total carbamate residues (carbofuran + 3-hydroxycarbofuran) in rank order were <0.03 (6), 0.03 (4), 0.04 (4), 0.05 and 0.08 mg/kg (n = 16). The Meeting estimated a maximum residue level of 0.1 mg/kg and an STMR of 0.03 mg/kg.

Soya beans. Trials were reported from Brazil, France, and the USA. Brazilian GAP specifies at-plant application of a 10% G at 1.5 kg ai/ha. US GAP allows 2.0 kg ai/ha at-plant or 2 applications at 0.56 kg ai/ha/application. No information on GAP was available for France or a neighbouring country. Only two trials according to GAP were reported, one from Brazil and the other from the USA. The residue in Brazil was below the limit of detection (0.05 mg/kg) and that in the USA was at the limit of determination (0.10 mg/kg). Two results are inadequate to estimate a maximum residue level, and the Meeting recommended withdrawal of the existing CXL for soya bean (dry) of 0.2 mg/kg.

Yard-long beans. The government of Thailand submitted a description of the in-field aspects of trials on yard-long beans but included no residue data. The Meeting took no action.

Carrots. The government of The Netherlands reported six field trials with at-plant application of an SC formulation to carrots. No GAP is available for The Netherlands or an EU country. The Meeting recommended withdrawal of the existing CXL (0.5 mg/kg).

Celeriac. The government of The Netherlands reported the results of one field trial with the application of an SC carbofuran formulation to celeriac. No GAP was reported and one trial is inadequate even for a very minor crop.

Potatoes. Field trials were carried out in Colombia, France, the UK and the USA. Applications according to GAP range from at-planting in Europe to banded treatment at hill-up and multiple foliar sprays in the USA. No GAP was available for Colombia, and the trials there were not evaluated. France and the UK each reported one trial in accordance with GAP. Six trials in the USA complied with the appropriate GAP, 3.4 kg ai/ha at-plant and 8 foliar applications at 1.1 kg ai/ha each, PHI 17 days. The residues in the whole tubers in the eight trials were <0.01 (3), <0.03 (2), 0.03, 0.04 and <0.05 mg/kg. The Meeting estimated a maximum residue level of 0.1 mg/kg and an STMR of 0.03 mg/kg.

Sugar beet. Field trials were carried out in France, Italy, Germany, the UK and the USA. European GAP specifies 2 kg ai/ha at planting, and US GAP early post-emergence foliar treatment (2.2 kg ai/ha, 90-day PHI). Five trials were at the maximum GAP rate and minimum PHI. The residues on the foliage were <0.01 (3), 0.05 and 0.15 mg/kg and in the roots <0.01 (4) mg/kg. The data were insufficient to estimate a maximum residue level. The Meeting recommended withdrawal of the existing CXLs for sugar beet and sugar beet leaves or tops.

Turnips. Five field trials on the application of carbofuran to turnips in France, the UK and Norway were reported. No information on GAP was available, and the Meeting could take no action.

Celery. In two field trials in The Netherlands carbofuran was applied to the soil immediately before planting. The Meeting could not estimate a maximum residue level.

Cereal grains

Maize. Reports of trials in Brazil, France, Germany and the USA were submitted. The trials represented a combination of at-planting (France, USA, Germany) and foliar (USA, Brazil) treatments. The reports from Brazil, France, and Germany were abbreviated summaries and did not provide the detail required to evaluate the trials. The results were not used in attempting to estimate a maximum residue level and an STMR.

Eleven trials were conducted in the USA, but only two residues in silage were from trials according to current GAP. In the trials with at-plant applications the rate was 34% higher than the GAP rate and a granular formulation was used in place of the specified soluble concentrate. All the samples of forage, fodder and grain were harvested well outside the GAP PHI (>30% deviation). The two silage residues (1.1 and 1.2 mg/kg) were insufficient to estimate a maximum residue level or an STMR, nor could the Meeting estimate maximum residue levels for maize, maize fodder or maize forage It therefore recommended withdrawal of the CXLs for maize and maize fodder.

Oats. Field trials on 3 varieties at one location were reported from Germany. The treatment was at-planting, and no residues (<0.10 mg/kg) were found in the oats. The number of trials was inadequate and the report consisted of a short summary that lacked the detail required for evaluation. The Meeting recommended withdrawal of the existing CXL (0.1* mg/kg).

Rice. Field trials in Australia, Brazil, Japan, the Philippines, and the USA were reported. The Brazilian summary report lacked the detail needed to evaluate the trials. The trials in the USA, Japan, and Philippines were not according to GAP. Only one trial in Australia accorded with GAP. The Meeting recommended withdrawal of the existing CXL (0.2 mg/kg).

Sorghum. See Sorghum forage etc., below.

Wheat. Field trials in South Africa and the USA were reported. Information on GAP was not available for the at-plant trials in South Africa. The six US trials were at the maximum GAP rate, with two foliar treatments and a 21-day PHI. The total carbamate residues in the grain in rank order were 0.02, 0.02 and 0.04 (4) mg/kg. The Meeting concluded that six trials were insufficient to estimate a maximum residue level and recommended withdrawal of the existing CXL (0.1* mg/kg).

Other crops

Sugar cane. Supervised field trials with the application of carbofuran to sugar cane were carried out in Brazil and the USA. In Brazil, the carbofuran (G or SC) was applied as a soil treatment about 5 months after planting. The PHI was 90 days. No residues were found (<0.1 mg/kg) in the four trials, two of which complied with GAP and two were at twice the GAP rate. In five trials in three states of the USA with the 4F formulation an in-furrow application at planting (1.1 kg ai/ha) was followed by two aerial foliar applications (2 x 0.84 kg ai/ha), with a 30-day PHI. This was according to GAP, and the maximum carbofuran residue was 0.06 mg/kg. The Meeting estimated a maximum residue level of 0.1* mg/kg, the existing CXL and the practical limit of quantification, and an STMR of 0.1 mg/kg.

Oilseed (cotton, sunflower, peanut, rape). Field trials in the USA and Brazil on cotton were reported, and the sponsor stated that trials were now in progress in southern Europe. The trials in Brazil were with seed treatment or a single post-emergence foliar treatment (2.1 kg ai/ha, 45-day PHI). The US trials involved two foliar applications of a flowable formulation (2 x 0.28 kg ai/ha). Neither set of trials complied with the relevant GAP, which is for at-plant use in both countries.

Trials on peanuts in Brazil and the USA were reported. The government of Thailand submitted information on field trials but no data on residues. Carbofuran was applied to peanut plants in two trials in Brazil as a foliar spray (1.75 or 3 kg ai/ha) with a 14-day PHI. In 14 US trials, peanut fields were treated at pegging. In some cases an initial treatment was also made at planting. The maximum carbamate residue was 0.53 mg/kg. Most of the US trials (80%) showed no quantifiable residues. GAP in Brazil is for at-plant treatment, and the USA has no GAP for the use of carbofuran on peanuts. Neither the Brazilian nor the US results could be used to estimate a maximum residue level.

Field trials on rape (canola) were carried out in Canada (seed treatment, at-plant, post-emergence) and France (at-plant). No GAP was reported for Canada or France, and the Canadian trials did not comply with US GAP. The trials could not be evaluated.

Field trials on sunflowers were carried out in Canada, France and the USA. The trials in France were discounted, because the method of analysis was described as semi-quantitative and was not explained. The US trials were not according to GAP; the PHI was >150% of the GAP PHI of 28 days, and the at-plant application was below the maximum rate. Six trials in Canada complied with maximum US GAP and all the residues were 0.04 mg/kg. The Meeting estimated a maximum residue level for sunflower seed of 0.1* mg/kg and an STMR of 0.1 mg/kg, but concluded that the trials were inadequate to support an MRL for oilseed and recommended withdrawal of the existing CXL (0.1* mg/kg).

Coffee. Two field trials in Brazil and four in the USA, all according to national GAP, on the application of carbofuran to coffee bushes were reported. The use patterns are quite similar in both countries. GAP in Brazil specifies 0.35 g ai/tree of SC formulation or 0.5-3 g ai/tree of G formulation, and US GAP specifies two applications of 1.7 g ai/tree, 10 G formulation. The residues in rank order were 0.02 (3), 0.08, 0.12 and 0.79 mg/kg (n = 6). The Meeting estimated a maximum residue level of 1 mg/kg and an STMR of 0.10 mg/kg. The two Brazilian residues of 0.02 mg/kg were not used for the estimation of the STMR because the analysis did not include a hydrolysis step to release conjugated 3-hydroxycarbofuran.

Alfalfa. Three field trials in each of seven states in the USA were according to the current maximum use rate and minimum PHI. Green forage and fodder were analysed. The carbamate residues in the fodder ranged from the limit of detection (<0.1 mg/kg) to 7.6 mg/kg. The trials involved foliar application of a flowable formulation at 1.12 kg ai/ha with a 28-day PHI. The maximum residues of carbofuran plus 3-hydroxycarbofuran in each trial in rank order were <0.1 (2), 0.28, 0.32, 0.64, 0.74, 0.87, 0.90, 0.92, 1.2, 1.4, 1.5, 1.6, 2.6, 2.8, 3.0, 3.4, 3.8, 4.2, 4.5, 4.6, 4.7, 5.2 and 7.6 mg/kg (n = 24). The Meeting estimated a maximum residue level of 10 mg/kg and an STMR of 1.6 mg/kg. The residues in the green forage in rank order were <0.1 (5), 0.13, 0.29, 0.30, 0.34, 0.38, 0.52, 0.92, 0.9 4, 1.2 (3), 1.3, 1.4, 1.6 (2), 1.7, 1.8, 2.2 and 4.3 mg/kg (n = 24). The Meeting estimated a maximum residue level of 10 mg/kg and an STMR of 0.93 mg/kg.

Sorghum forage (green), sorghum straw and fodder, dry . Six trials in India and six in the USA were reported. The trials in India were with seed treatment or at-plant treatment, whereas the US trials were at-plant plus two foliar applications, with a total rate of 2.3 kg ai/ha. GAP was not available for India or a neighbouring country. In the US trials the residues in rank order were 0.055 (6), 0.06, 0.07, 0.11 (2), 0.13, 0.19, 0.26 and 1.2 mg/kg (n = 14) in sorghum forage (green), 0.05 (2), 0.06 and 0.20 mg/kg (n = 4) in sorghum fodder, and <0.01 (5) mg/kg in sorghum grain. The Meeting estimated maximum residue levels of 2 mg/kg for forage, 0.5 mg/kg for fodder, and 0.1* mg/kg for grain, with respective STMRs of 0.065 mg/kg, 0.055 mg/kg and 0.01 mg/kg. Although no residues were found in the grain at an estimated limit of detection of 0.01 mg/kg, the practical limit of quantification for carbofuran and 3-hydroxycarbofuran individually in plant commodities is 0.1 mg/kg.

Barley, egg plant, hops (dry), mustard seeds, peaches, pears. No trials were reported. The Meeting recommended withdrawal of the existing CXLs.

Feeding studies on poultry and cows were reported. The poultry study was defective because although residues were reported as <0.05 mg/kg from feeding 5 ppm in the diet, the uncertainties surrounding the method of analysis cast doubts on the reliability of the results. A study conducted over 7 days at 25 ppm however showed negligible concentrations of radiolabelled residue (<0.01 mg/kg as carbofuran) in muscle and fat and a residue of 0.15 mg/kg in eggs, of which the carbamate content was below 20%. Potential poultry feed items include small grain (maize, barley, oats, wheat, sorghum, 80% of the diet) and alfalfa meal (10% of the diet). Thus, the diet might contain 80% x the 0.04 mg/kg STMR of maize + 10% x the 1.2 mg/kg STMR of alfalfa hay = 0.15 mg/kg. Note that this includes a commodity (maize) for which the withdrawal of a CXL has been recommended. Residues of carbofuran and its carbamate metabolites in poultry commodities are unlikely from such feeding levels. The Meeting concluded that MRLs are not needed for poultry commodities.

The ruminant feeding study was conducted with carbosulfan, not carbofuran. Carbosulfan is metabolized rapidly to carbofuran in ruminants, and the carbofuran is converted to 3-hydroxycarbofuran and phenol metabolites. Goats were fed carbosulfan at a level of 50 ppm in the diet for 28 days. The milk contained no detectable residues of carbosulfan on days 1-4, but it was present at very low concentrations, 0.005-0.011 mg/kg, from days 7 to 27. Carbofuran was detected on day 4 at a maximum concentration of 0.006 mg/kg and on day 7 at a maximum of 0.008 mg/kg. The carbofuran metabolite 3-hydroxycarbofuran appeared on day 1 (0.022 mg/kg) and continued through day 27 (0.013 mg/kg). The tissues contained no detectable residues of carbosulfan or carbofuran, but 0.060 mg/kg of 3-hydroxycarbofuran was found in the liver and 0.13 mg/kg in kidney. The Meeting concluded that feeding with carbosulfan may be substituted for feeding with carbofuran.

The study of metabolism in goats, conducted for 7 consecutive days with 25 ppm carbofuran in the feed, revealed no radiolabelled residues (<0.01 mg/kg as carbofuran) in the muscles or fat. Significant residues occurred in the milk (0.14 mg/kg) and in the liver and kidneys (0.11, 0.18 mg/kg). About 50% of the TRR in the milk was shown not to include carbamate compounds, and about 11% was carbofuran plus 3-hydroxycarbofuran (0.02 mg/kg). The kidneys and liver each contained <15% carbamates (0.02 mg/kg).

On the basis of the MRLs recommended by the present Meeting, the ruminant diet would contain no more than 2 mg/kg of carbofuran plus 3-hydroxycarbofuran. This is based on a diet containing 80% of alfalfa fodder (0.8 x the STMR of 1.6 mg/kg = 1.3 mg/kg). Owing to the substantial number of MRLs recommended for withdrawal there are few animal feed items. The Meeting estimated a maximum residue level of 0.05* mg/kg and an STMR of 0.05 mg/kg for residues (as defined above) in various animal products and milks.

Processing

Studies were conducted with sorghum, sugar beet, potatoes, maize, rice, sunflowers, cotton seed, sugar cane, coffee, pimento peppers, and grapes. Most of them were of limited value because the raw agricultural commodities contained carbamate residues below the limit of detection or between the limit of detection and the limit of determination. In most cases the same applied to the processed commodities. On the basis of the recommendations of the Meeting, processing studies would be appropriate for coffee, potatoes, sunflowers, and sugar cane. The sugar cane and potato processing studies, with applications at 1.8 times and twice the GAP rate respectively, were inadequate because there were no residues in the raw agricultural commodities. The sunflower processing study was acceptable: the residue was unchanged in the edible oil and increased in the hulls and extracted meal by factors of 1.2 and 1.8 respectively. The coffee processing study showed a reduction factor of approximately 0.05-fold for instant and roast coffee. The value is approximate because the residues in the processed commodities were at the limit of detection.

RECOMMENDATIONS[AFM1]

The Meeting estimated the maximum residues and STMR residues listed below. The maximum residue levels are recommended for use as MRLs.

Pesticide (Codex ref. No.)	ADI (mg/kg bw)	Commodity CCN	Recommended MRL or ERL (mg/kg)		STMR (mg/kg)	
			Name	New	Previous	New
Carbofuran**	0.002	AL 1020	Alfalfa fodder	10	20	1.6
(096)		AL 1021	Alfalfa forage (green)	10	5	0.93
		FI 0327	Banana	0.1*	0.1*	0.1
		GC 0640	Barley	W	0.1*	
		VB 0402	Brussels sprouts	W	2	
		VB 0041	Cabbages, Head	W	0.5	
		VC 4199	Cantaloupe	0.2	-	0.02
		VR 0577	Carrot	W	0.5	
		MF 0812	Cattle fat	0.05*	0.05*	0.05

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[AFM1]Table is from Annex I so needs editing. Text also needed.

Pesticide (Codex ref. No.)	ADI (mg/kg bw)	Commodity CCN	Recommended MRL or ERL (mg/kg)		STMR (mg/kg)	
			Name	New	Previous	New
		VB 0404	Cauliflower	W	0.2	
		DM 0001	Citrus molasses[1]			0.11 P
		AB 0001	Citrus pulp, dry[1]	2	-	0.29
		SB 0716	Coffee beans	1	0.1*	0.1
			Coffee, Instant			0.005 P[1]
		SM 0716	Coffee, Roast			0.005 P
		VC 0424	Cucumber	0.3	-	0.05
		MO 0096	Edible offal of cattle, goats, horses, pigs and sheep	0.05*	0.05*	0.05
		VO 0440	Egg plant	W	0.1*	
		MF 0814	Goat fat	0.05*	0.05*	0.05
		DH 1100	Hops, dry	W	5	
		MF 0816	Horse fat	0.05*	0.05*	0.05
		VB 0405	Kohlrabi	W	0.1*	
		VL 0482	Lettuce, Head	W	0.1*	
		GC 0645	Maize	W	0.1*	
		AS 0645	Maize fodder	W	5	
		MM 0096	Meat of cattle, goats, horses, pigs and sheep	0.05*	0.05*	0.05
		ML 0106	Milks	0.05*	0.05*	0.05
		SO 0090	Mustard seed	W	0.1*	
		GC 0647	Oats	W	0.1*	
		SO 0088	Oilseed	W	0.1*	
		VA 0385	Onion, Bulb	W	0.1*	
		FC 0004	Oranges, Sweet, Sour[1]	0.5	-	0.1
		JF 0004	Orange juice[1]			0.001
		FS 0247	Peach	W	0.1*	
		FP 0230	Pear	W	0.1*	
		MF 0818	Pig fat	0.05*	0.05*	0.05
		VR 0589	Potato	0.1	0.5	0.03
		CM 0649	Rice, Husked	W	0.2	
		MF 0822	Sheep fat	0.05*	0.05*	0.05
		GC 0651	Sorghum	0.1*	0.1*	0.01
		AF 0651	Sorghum forage (green)	2	-	0.065
		AS 0651	Sorghum straw and fodder, dry	0.5	-	0.055
		VD 0541	Soya bean, dry	W	0.2	
		VC 0431	Squash, Summer	0.3	-	0.05
		FB 0275	Strawberry	W	0.1*	
		VR 0596	Sugar beet	W	0.1*	
		AV 0596	Sugar beet leaves or tops	W	0.2	
		GS 0659	Sugar cane	0.1*	0.1*	0.1
		SO 0702	Sunflower seed	0.1*	0.1*[2]	0.1
		VO 1275	Sweet corn (kernels)	W	0.1*	
		VO 0447	Sweet corn (corn-on-the -cob)	0.1	-	0.03
		VO 0448	Tomato	W	0.1*	

Pesticide (Codex ref. No.)	ADI (mg/kg bw)	Commodity CCN	Recommended MRL or ERL (mg/kg)		STMR (mg/kg)	
			Name	New	Previous	New
		GC 0654	Wheat	W	0.1*	

FURTHER WORK OR INFORMATION

Desirable

1. A feeding study with cows fed carbofuran.

2. Processing studies on potatoes and sugar cane. Exaggerated treatment rates (five- to tenfold) should be used to obtain weathered residues in or on the raw agricultural commodities.

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CARBOSULFAN (145)

EXPLANATION

Carbosulfan was first evaluated by the 1984 JMPR which allocated a temporary ADI of 0.005 mg/kg bw and recommended a temporary MRL of 2 mg/kg for the sum of carbosulfan, carbofuran, 3-hydroxy-carbofuran in citrus fruits. The temporary ADI was replaced by an ADI of 0-0.01 mg/kg bw by the 1986 JMPR.

The 1991 JMPR considered a request from the CCPR to harmonize the definition of the residue with that of carbofuran (which did not include 3-keto-carbofuran). It recommended that MRLs for carbosulfan should refer to a residue defined as carbosulfan and that MRLs for carbofuran should be for the sum of carbofuran and 3-hydroxy-carbofuran expressed as carbofuran, whether from the use of carbofuran or carbosulfan, with a clear indication of which use was the basis for the MRL. It further recommended that analyses of samples from supervised trials should continue to include the determination of 3-keto-carbofuran.

Because information required by the 1991 Meeting was not provided, the 1993 JMPR recommended withdrawal of the proposed limits for carbofuran and carbosulfan in citrus fruits. It was informed that additional studies were under way.

Both carbofuran and carbosulfan were considered as candidates for review as part of the CCPR periodic review programme. As a result, the 1996 JMPR estimated an ADI of 0-0.002 mg/kg bw for carbofuran. Carbosulfan has not yet been scheduled for periodic review of its toxicology. The residue and analytical aspects of both carbofuran and carbosulfan were subjected to periodic review at the present Meeting.

IDENTITY

Common name: Carbosulfan (BSI, ANSI, E-ISO, F-ISO)

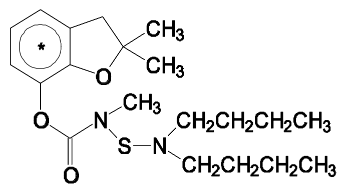
Chemical name:
IUPAC: 2,3-dihydro-2,2-dimethylbenzofuran-7-yl(dibutylaminothio)methylcarbamate
CA: 2,3-dihydro-2,2-dimethyl-7-benzofuranyl [(dibutylamino)thio]methylcarbamate

CAS Registry No.: 55285-14-8

CIPAC No: 417

Synonyms: FMC 35001, Marshal®, Advantage®

Structural formula:



Molecular formula: $C_{20}H_{32}N_20_3S$

Molecular weight: 380.5

Physical and chemical properties (Alvarez,1995)

Pure active ingredient

Vapour pressure: 2.69×10^{-7} mm Hg at 25°C; nitrogen saturation method; 97.1% pure

Melting point: Carbosulfan is a liquid. It decomposes at elevated temperature (boiling point not determinable)

Octanol/water partition coefficient (25°C, pH 9, 97.1% pure):

Log K_{OW} = 5.4; $K_{OW} = 2.8 \times 10^5$
(carbosulfan is hydrolysed at lower pH)

Solubility: available only for technical material

Specific gravity: available only for technical material

Hydrolysis Hydrolyzes at <pH 9
Photolysis: mainly to carbofuran and dibutylamine in aqueous solutions, half-life 1.4 days at pH 7 and 4-8 days in distilled water. Half-life <10 minutes but greater at 70% field moisture (Capps, 1981, in Alvarez, 1995). See also environmental fate in soil and water below.

Technical material

Purity: Not included in cited reference (1984 JMPR reported 93% for technical and 86-91% for manufacturing-use product).

Solubility:

water (pH 9, 25°C) 0.3 mg/l
acetone, acetonitrile, toluene, hexane (25°C) miscible at carbosulfan to solvent weight ratios from 0.02 to 2
Exxon Aromatic 100 (23°C) 1 g completely miscible in 4 ml solvent

Specific gravity (25°C):
Technical 1.054 g/ml
Manufacturing-use product1.052
Melting range: carbosulfan is a liquid. It is degraded at elevated temperature

Stability (10 days at 23 and 50)
Stainless steel and aluminum surfaces: no instability during

Hydrated ferric, manganous and cupric sulfates (10 days)
Manganous sulfate: no significant decrease (92.8% remained)
Ferric sulfate: significant decrease at 23 or 50°C (9.6% remained at 23 , 5.7% at 50°C)
Copper: significant decrease at 50°C (3.75% remained)

Oxidizing and reducing agents: exposure to zinc and potassium permanganate showed no tendency for oxidation or reduction.

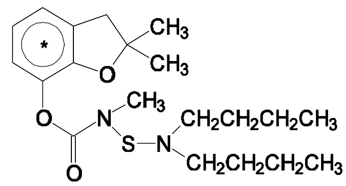
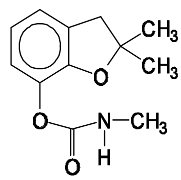
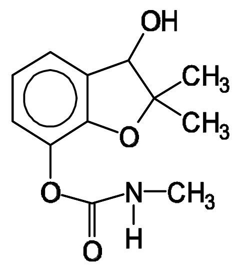
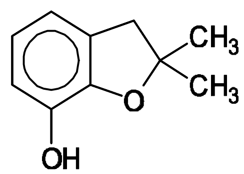
Formulations

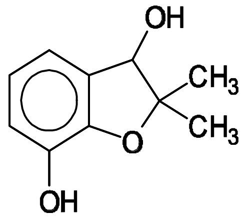
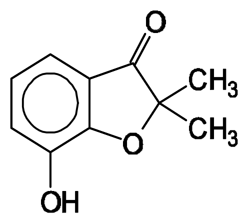
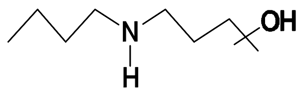
Liquid emulsion 26.1% active ingredient
Liquid emulsion 250 g ai /l

METABOLISM AND ENVIRONMENTAL FATE

The fate of carbosulfan has been investigated in rats, goats and oranges. In each case [^{14}C]carbosulfan was labelled (in separate experiments) both uniformly in the phenyl ring and at the C-1 carbons of the dibutylamine (DBA) group. The structures and chemical names of the major metabolites identified are shown in Table 1.

Table 1. Structures and chemical names of carbosulfan and some major identified metabolites.

Common or abbreviated name Abbreviation FMC no.	Chemical name	Chemical structure
carbosulfan	2,3-dihydro-2,2-dimethylbenzofuran-7-yl (dibutylaminothio)methylcarbamate	
carbofuran CF FMC 10242	2,3-dihydro-2,2-dimethylbenzofurany-7-yl methylcarbamate	
3-hydroxy-carbofuran 3-OH-CF FMC 18209	2,3-dihydro-3-hydroxy-2,2-dimethylbenzofuran-7-yl methylcarbamate	
7-phenol 7-P FMC 10272	2,3-dihydro-2,2-dimethylbenzofuran-7-ol	

Common or abbreviated name Abbreviation FMC no.	Chemical name	Chemical structure
3-hydroxy-7-phenol 3-OH-7-P FMC 16497	2,3-dihydro-2,2-dimethylbenzofuran-3,7-diol	
3-keto-7-phenol 3-K-7-P FMC 16490	2,3-dihydro-2,2-dimethyl-3-oxobenzofuran-7-ol	
Dibutylamine DBA FMC 65387	dibutylamine	$[CH_3(CH_2)_3]_2NH$
Various hydroxylated dibutylamines	Name depends on position of OH substitutent	

Other test substances and reference compounds used but not listed above included 5-hydroxy-carbofuran, *N*-hydroxy-carbofuran, 3-hydroxy-carbosulfan, 3-keto-carbosulfan, carbosulfan sulfone, 3-keto-carbosulfan sulfone, 4-aminobutanol, 1-amino-2-butanol, butylamine, l-henylalanine, l-tyrosine, dl-tryptophan, and C_4 to C18 fatty acids (Curry and Weintraub, 1996).

The major compounds (>10% of the TRR) found in rats, goats and oranges are shown in Table 2..

Table 2. Major components (>10% of the TRR) of the residues from [^{14}C]carbosulfan found in plants and animals.

Compound	RAT		GOAT (MILK)		ORANGE[1]	
	Phenyl label	DBA label	Phenyl label	DBA label	Phenyl label	DBA label
carbosulfan					X	X
carbofuran					X	
3-hydroxy-carbofuran	X		X			
3-keto-7-phenol	X		X			
3-hydroxy-7-phenol			X			
7-phenol	X		X			

Compound	RAT		GOAT (MILK)		ORANGE[1]	
	Phenyl label	DBA label	Phenyl label	DBA label	Phenyl label	DBA label
dibutylamine		X				X
hydroxylated dibutylamines		X				
aminobutanols				X		
Fatty acids				X		
amines				X		

[1] Surface rinses and extracts of peel

Animal metabolism

The fate of carbosulfan has been investigated in rats (Fang and ElNaggar, 1995) and goats (Curry and Weintraub, 1995).

Rats. In the definitive phase of the study, conducted according to US EPA GLP, 60 male or female Hsd:Sprague Dawley rats were dosed orally in groups by syringe with either phenyl- or DBA-labelled [^{14}C]carbosulfan, according to one of three treatment regimens. Four additional animals were used as controls. The dosing solutions (pure radio-labelled standards diluted with unlabelled carbosulfan) were administered at approximately 4.3 mg/kg bw (about 22 uCi/mg specific activity) for low doses and 29 mg/kg (*c.* 3.1 uCi/mg specific activity) for high doses. Dosing was based on animal weights which were about 180 to 240 g for males and 180 to 207 g for females. The dosing regimens (5 female and 5 male rats each for phenyl and DBA labels in each regimen) are designated in Tables 3 and 4 as shown below.

SLD single low dose
MLD multiple low doses (single unlabelled dose followed by labelled dose)
SHD single high dose

Samples of urine, faeces and cage rinses were collected at intervals up to 48 hours and daily thereafter for a total of 168 hours. In the experiment with the DBA label CO_2 and other volatiles were collected at intervals based on expectations from preliminary studies. After 168 hours the rats were killed and samples including fat, muscle, skin, organs and brain were taken for analysis. Solid samples were combusted and ^{14}C measured as carbosulfan equivalents in all samples by liquid scintillation counting (LSC). The distribution and recovery of radioactivity is shown in Table 3.

Table 3. Distribution and total recoveries of administered radioactivity in dosed rats.

Group, Label	Distributions and Total Recoveries of Administered Radioactivity											
	% of Dose											
	Urine		Faeces		CO2		Tissue		Carcase		Total	
	M	F	M	F	M	F	M	F	M	F	M	F
SLD												
Phenyl	76	81	22	16	NA	NA	<0.01	<0.01	0.20	0.79	98	98
DBA	66	65	13	17	12	10	0.20	0.13	1.07	0.98	92	93
MLD												
Phenyl	79	88	15	5	NA	NA	<0.01	<0.01	ND	0.27	93	95
DBA	71	71	8	8	13	15	0.02	0.13	1.01	0.93	94	94
SHD												
Phenyl	83	72	10	17	NA	NA	ND	ND	0.52	1.92	94	91
DBA	66	66	8	12	17	10	0.23	0.09	1.42	1.58	93	90

NA not applicable; ND none detected

About 80-90% of the low dose of both labels was excreted within 24 to 48 hours of dosing by both sexes. With the high dose about 72 hours or longer was required for this level of excretion.

Samples of urine and faeces were analysed separately for the identification and characterization of metabolites mainly by HPLC and TLC with enzymatic hydrolyses as needed. The urine metabolites were 4-9% nonconjugates, 16-27% glucuronide conjugates and 49-57% sulfate conjugates. The identities of the major metabolites were confirmed by electron-impact GC-MS or LC-MS of dansyl, 4-chlorobenzoyl, acetyl or trimethylsilyl (TMS) derivatives. The results are shown in Table 4.

Table 4. Compounds identified in rat excreta (combined faeces and urine).

Compound	Distribution of ^{14}C-Residues as % of Dose					
	SLD		MLD		SHD	
	Male	Female	Male	Female	Male	Female
Phenyl label						
3-keto-7-phenol	20.7	26.7	14.6	20.8	25.9	24.3
3-hydroxy-7-phenol	15.4	13.4	26.3	26	18.5	18.4
7-phenol	23.9	23.1	8.9	11.7	7.4	5.1
3-hydroxy-carbofuran	17.3	13.9	22.1	21.1	17.2	13.4
carbosulfan	2.5	3.4	0.9	0.4	1.7	4.6
Minor metabolites (6)*	6	5.2	6	3.5	6	7.8
Total identified	85.9	85.6	78.8	83.5	76.8	73.6
DBA label						
dibutylamine	38.3	42.8	40.9	44.7	39	50.8
hydroxy-dibutylamine	23.8	22.5	25.8	24.7	23.5	17.1
CO_2	10.7	8.7	12.1	12.5	14.6	9
carbosulfan	6.3	8.3	3.5	4.2	4.1	6.9
Total identified	79.1	82.3	82.2	86.1	81.1	83.8

* Minor metabolites (each <5% of the dose): 5-hydroxy-carbofuran, 3-keto-carbofuran, carbofuran, 3-keto-carbosulfan sulfone, 3-hydroxy-carbosulfan, 3-keto-carbosulfan

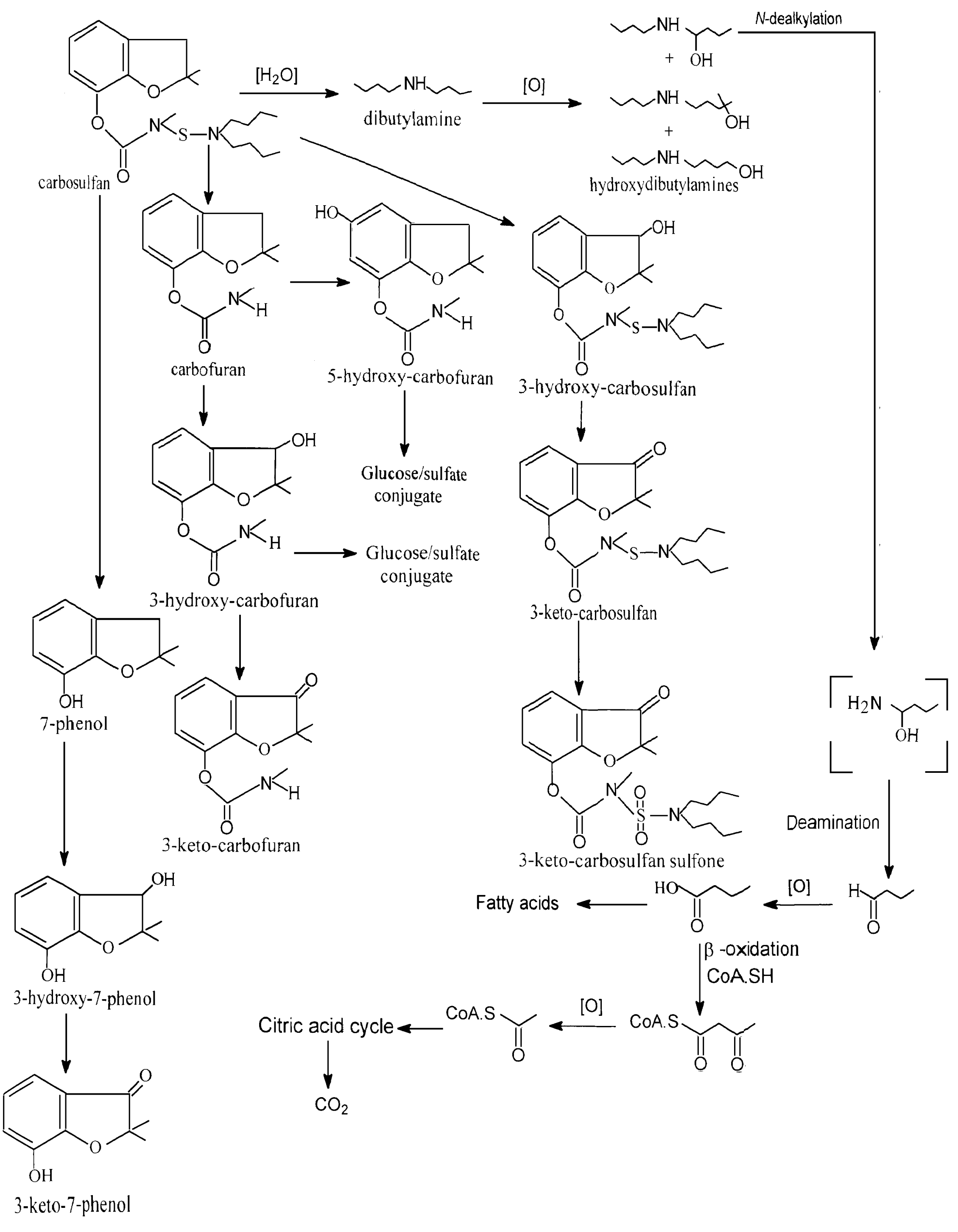
In carcase tissues the radioactivity ranged from undetectable to 1.9% of the applied dose. As an example, the mean residues ^{14}C expressed as mg/kg carbosulfan in the tissues of males rats from single doses were:

	DBA label		Phenyl label
	Low dose	High dose	Low dose
Liver	0.1 ± 0.005	0.87	ND
fat (reproductive)	0.08± 0.002	0.74	ND
skin	0.04± 0.006	0.57	ND
carcass (residual)	0.04± 0.007	0.40	0.008±0.008
kidney	0.03± 0.002	0.34	0.008±0.016
muscle (thigh)	0.01±0.002	0.16	ND

The residue levels from the DBA label were not appreciably different between males and females or between multiple-dose and the single low-dose samples. Residues from single doses of the phenyl label in some tissues of females were 3-4 times as high as in males. The residue levels were too low for further characterization or identification of the tissue residues..

The metabolic pathways proposed for carbosulfan in rats are shown in Figure 1.

Figure 1. Proposed metabolic pathways of carbosulfan in rats (Fang and El Naggar, 1995).



<u>Goats</u>. In this study, again according to US EPA GLP, 4 two year-old lactating Nubian goats were dosed orally once daily with either phenyl- or dibutylamine-labelled carbosulfan by balling gun for 7 consecutive days, two goats dosed with each label and with one control. Doses were prepared from 99.2% pure phenyl- and 98.8% DBA-labelled carbosulfan by dilution with unlabelled material. The phenyl labelled dose was approximately 44.7 mg/goat/day (1.75 mCi/day = 39.2 µCi/mg specific activity) corresponding to approximately 23 ppm in the diet based on average feed consumption of

approximately 1.9 kg/day. The DBA-labelled dose was approximately 40.9 mg/goat/day (1.91 mCi/day = 46.7 μCi/mg specific activity), about 25 ppm in the diet.

Urine and faeces were collected daily and milk in the afternoon and in the morning before dosing. They were stored separately. Blood was sampled just before slaughter 22 hours after the last dose and samples of omental and peripheral fat, liver, kidney and leg and lumbar muscle were taken for analysis. Storage was at ≤-20°C until analysis, generally within 6 months of sample collection.

Organic and aqueous extracts were analysed separately as such or after further clean-up. Some aqueous phases were subjected to enzymatic or acid hydrolysis and further partitions and separations. As an example, milk containing the phenyl label was shaken with acetone and extracted with acetonitrile. The acetonitrile extracts were combined and concentrated, and partitioned with hexane. Aliquots of the acetonitrile fraction were hydrolised with β-glucosidase of sulfatase and cleaned up on a C-18 solid-phase extraction column. The remaining unextractable fractions were analysed by combustion.

Milk samples containing the DBA label were also extracted with acetone and acetonitrile. The combined and concentrated aqueous acetonitrile extract was basified with ammonium hydroxide and partitioned with hexane, yielding a hexane fraction and an aqueous acetonitrile fraction. The hexane fraction after washing with 0.01 N HCl was saponified and partitioned with ethyl ether, and the resulting aqueous fraction acidified and again partitioned with ethyl ether. The acidic hexane wash was saponified and partitioned with ethyl ether, and the resulting aqueous fraction acidified and again partitioned with ethyl ether. The acidic hexane wash was basified and extracted with ethyl ether, to yield acqueous and ethyl ether fractions for analysis.

The aqueous acetonitrile fraction from the first hexane partition was concentrated, adjusted to pH 12 and partitioned with dichloromethane. The aqueous fraction was hydrolysed with β-glucuronidase and sulfatase and the hydrolysate passed through a solid-phase extraction column from which methanolic and aqueous fractions were eluted. The methanolic fraction was hydrolysed with HCl. The unextractable fractions containing the DBA label were tested for association with carbohydrates (phenylhydrazone derivatization) or proteins (pepsin/pronase digestion), or characterized by size-exclusion chromatography (to detect highly polar residues in protein fractions).

Similar separative steps were used for other samples, although extraction solvents varied and blending was required. Fats were dissolved in hexane before extraction with acetonitrile (the analysis included saponification with ethanolic KOH and esterification). Liver samples were initially blended with methanol buffered at pH9 and kidney and muscle samples with methanol buffered at pH 6.

The analytical procedures included combustion and liquid scintillation counting, normal and reversed-phase TLC, HPLC, GC-MS (quadrupole MS), LC-MS and chemical derivatization (dansyl, *p*-chlorobenzoyl, diazomethane, *p*-bromophenacyl and trimethylsilyl (TMS) derivatives. Components of the residues were identified in samples with the highest residues, namely milk at 7 days for the phenyl label and 5 days for the DBA label, lumber muscle and omental fat for the DBA label, and liver and kidney for both labels. The residues in phenyl-labelled muscle and fat were too low for identification. The total cumulative percentages of the dose recovered are shown in Table 5 and the total radioactivity in milk and tissues in Table 6.

Table 5. Percentages of administered ^{14}C recovered from urine, faeces, milk, tissues and cage rinses of goats dosed with [^{14}C]carbosulfan (Curry and Weintraub, 1996).

SAMPLE	% OF DOSE RECOVERED			
	PHENYL LABEL		DBA LABEL	
	GOAT 1	GOAT 2	GOAT 1	GOAT 2
URINE	80.77	84.37	70.02	66.19
Faeces	6.50	7.41	4.02	2.54
Cage rinse	1.40	1.30	0.35	0.31
Milk	0.16	0.17	1.97	2.66
Liver	0.02	0.02	0.37	0.31
Kidney	0.01	<0.01	0.03	0.04
Rear leg muscle	<0.01	<0.01	0.08	0.07
Lumbar muscle	<0.01	<0.01	0.05	0.04
Omental fat	<0.01	<0.01	0.18	0.13
Peripheral fat	<0.01	<0.01	0.06	0.05
Total	88.86	93.27	77.13	72.34
Mean total	91.07		74.74	

Table 6. Total radioactive residues (TRR) in milk and tissues of goats dosed with phenyl- and DBA-labelled carbosulfan (Curry and Weintraub, 1996).

^{14}C expressed as carbosulfan, mg/kg						
Label	Milk (pm sample)	Liver	Kidney	Muscle	Peripheral fat	Omental fat
Control	ND	ND	ND	ND	ND	ND
Phenyl	0.04-0.09	0.06	0.18	<0.01	0.01	0.009
DBA	0.3 - 0.94	1.13	0.75	0.18	0.74	1.2

The distribution and identity of residues from the phenyl label are shown in Table 7 and the DBA-label in Table 8.

Table 7. Identity and distribution of carbosulfan metabolites from feeding of phenyl-labelled carbosulfan to goats for 7 days (Curry and Weintraub, 1996).

Metabolite	% of TRR		
Total residue, mg/kg, as carbosulfan	Milk[1]	Liver	Kidney
3-hydroxycarbofuran	34.20	9.50	21.50
3-oh-7-phenol	21.10	15.60	13.30
3-keto-7-phenol	29.90	3.00	8.30
7-phenol	9.20	4.60	8.90
Minor components(<6)	1.20	4.40[2]	7.60[3]
Characterized organosolubles	2.30	17.30	17.40
Protein-associated metabolites	-	22.60	2.50
Polar aqueous metabolites	0.70	10.40	18.40
Unextractable residue	1.40	12.70	2.10
Total Residue (% of the TRR)	100.00	100.00	100.00
Total residue (mg/kg as carbosulfan)	0.09	0.06	0.154

[1]Day 7, pm. Metabolites identified in hydrolysed organo-extractable fraction containing 96.8% of the milk radioactivity. 3-keto-carbofuran (1.2% of the TRR) was also identified.

[2]Comprising 5-hydroxy-carbofuran (1.1%), 3-keto-carbofuran (1.5%), *N*-hydroxy-carbofuran (0.5%), carbofuran (0.2%), carbosulfan (0.1%) and a combination of 3-keto-carbosulfan sulfone, 3-hydroxy-carbosulfan and carbosulfan sulfone (1%, unresolved by the HPLC system).
[3]Comprising 5-hydroxycarbofuran (1.7%), 3-keto-carbofuran (3.7%), *N*-hydroxy-carbofuran (1%), carbofuran (0.8%), carbosulfan (0.1%) and a combination of 3-keto-carbosulfan sulfone, 3-hydroxy-carbosulfan and carbosulfan sulfone (0.3%, not resolved by the HPLC system).

Table 8. Identity/characterization and % of the TRR of carbosulfan metabolites from feeding of DBA-labelled carbosulfan to goats for 7 days (Curry and Weintraub, 1996)

METABOLITE	PERCENT OF TRR				
Total residue, mg/kg, as carbosulfan	MILK[1]	FAT	LIVER	KIDNEY	MUSCLE
Aminobutanols[1]	29.7	0.8	8.1	11.9	ND
Dibutylamine + related cpds [2]	6.7	0.6	13.4	10.5	9.6
Natural constituents[3]	30.2	87.3	29.1	13.8	32.0
Unconjugated amines[4]	11.8	ND	6.3	24.3	5.9
Conjugated or bound amines	10.5	ND	18.0	12.3	14.7
Lipophilic metabolites	0.6	0.5	1.3	4.5	1.2
Polar aqueous metabolites	7.6	0.2	16.6	18.5	26.5
[Un]extractable residues	2.9	10.5	7.2	4.2	10.0
Total residue (% of the TRR)	100.0	100.0	100.0	100.0	100.0
Total residue (mg/kg as carbosulfan)	0.680	1.286	0.986	0.823	0.193

[1]Day 5, pm. Metabolites identified in 4-aminobutanol; 1-amino-2-butanol.
[2]e.g. dibutylamine; hydroxydibutylamine; butylamine. In liver carboxy-DBA (10% of the TRR).
[3]e.g. in milk, fatty acids (13.4% of the TRR), amino acids (5.5%), carbohydrates (10.3%) and triglycerides (1.1%). In omental fat, fatty acids (82%) and triglycerides (5.3%). In lumbar muscle 20.6% of the TRR was associated with conjugated, unconjugated or bound amines and 32%of the TRR with amino acids.
[4]e.g. in milk 7 unknowns, each ≤0.024 mg/kg carbosulfan equivalent in omental fat 0.6% of the TRR, in liver 6.3% , in kidney (6 unknowns, each ≤0.06 mg/kg) 24.3% of the TRR (5 unknowns).

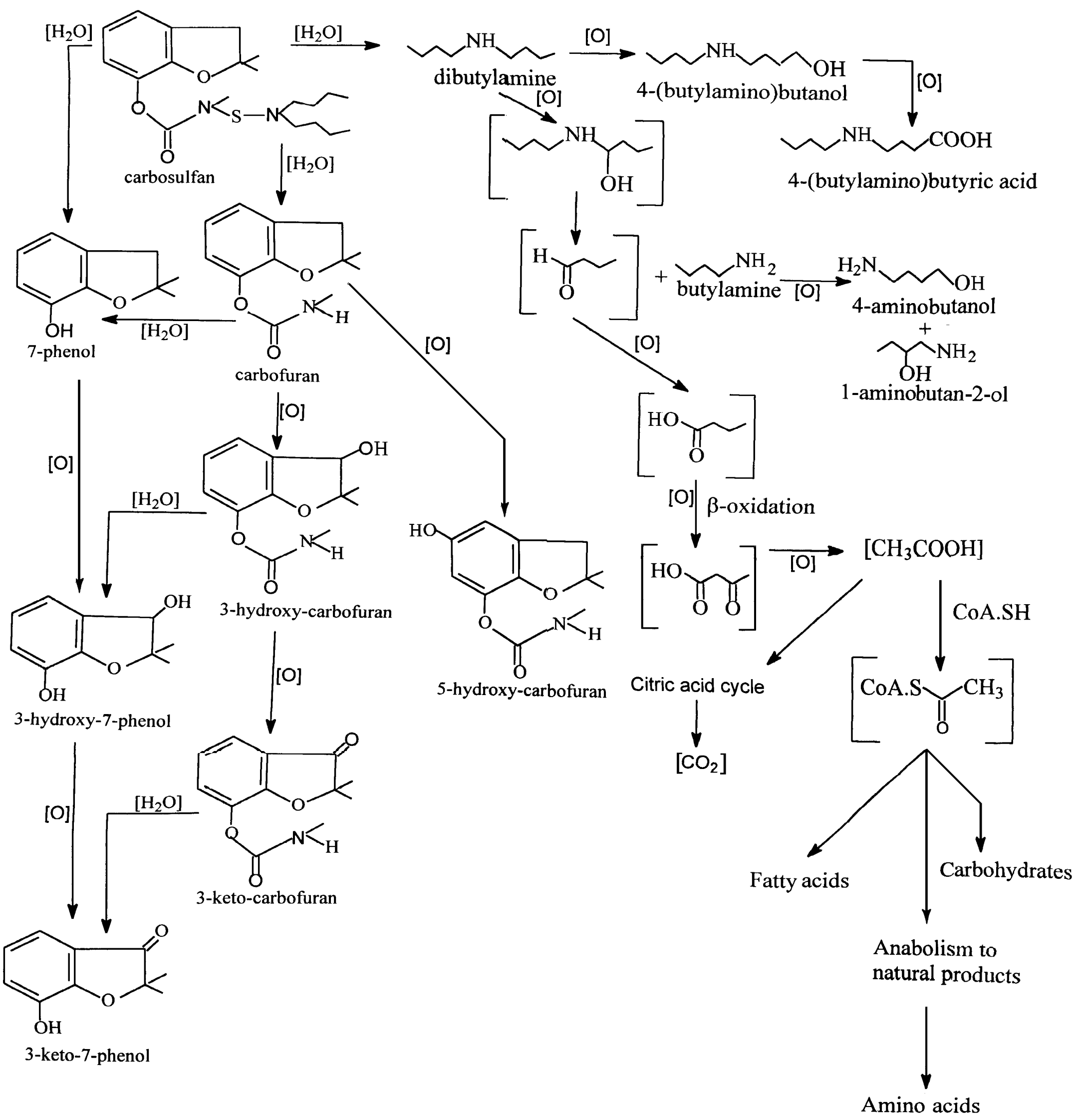
The proposed metabolic pathways in goats are presented in Figure 2.

Plant metabolism

A plant metabolism study was conducted according to US EPA GLP on three separate 21-year-old California commercial Navel orange trees in the field. One was treated with phenyl-labelled carbosulfan (99.3% purity, isotopic dilution 13.9 mCi/mmole), one with DBA-labelled carbosulfan (97.4% purity, isotopic dilution 15.08 mCi/mmole) and the third with a formulation blank as a control. Both labelled compounds were applied as 250 EC formulations at a nominal concentration of 0.5 g ai /l (*c*.450 ml/tree or an estimated 123 l/ha), with the trees protected from rain by plastic sheeting after the application. The trees were not irrigated during the study. Mature oranges were individually sprayed twice with a spray bottle with minimum run off (*c*.1 ml/orange), and the foliage was sprayed with a nitrogen-pressurized spray wand.

Ten to 20 mature fruit were collected for analysis on days 0, 7, 15 and 30, and leaves on days 0 and 30. Precautions were taken during shipment and storage to preserve the residues. Individual fruits from each sampling were processed and the fractions combined. Surface residues were removed by mild agitation in methanol/methylene chloride (1/1 v/v). The rinsed fruits were peeled and the peels homogenized under dry ice, as were leaves. The peeled oranges were puréed and centrifuged, and the juice was decanted.

Figure 2. Proposed metabolic pathways of carbosulfan in goats (Curry and Weintraub, 1996).



The total ^{14}C was determined in the processed fractions by combustion counting of $^{14}CO_2$ and in the rinses directly by LSC. Peels containing the phenyl label were extracted with KH_2PO_4/NaOH buffer and methanol. Non-polar fractions were separated by methylene chloride partition, and polar and conjugated fractions on a C18-solid phase extraction column (SPE) with methanol as eluant after hydrolysis with HCl and adjustment to pH 1. Post-extraction solids were refluxed with HCl and separated by SPE chromatography, yielding released extractable, released unextractable and bound residues.

Samples containing the DBA label were extracted and separated in a similar manner, except that the polar fractions were not adjusted to pH 1 before SPE chromatography. The remaining aqueous solution was adjusted to pH 14 before further SPE chromatography. Total ^{14}C was measured in the post-extraction solids, but they were not further characterized as they accounted for <5% of the TRR.

The separated fractions containing the phenyl label were analysed primarily by gradient reverse-phase HPLC with multi-wavelength UV detection at 230, 225.4, and 280 nm, and those with the DBA label primarily by reverse-phase TLC. Both groups were analysed by normal-phase TLC as a secondary mode. Isolated residues of carbosulfan, carbofuran, dicarbofuran sulfide and carbosulfan sulfone from phenyl-labelled treatments were collected for HPLC-MS (CI) analysis, although the identities of the last two were confirmed only by normal-phase TLC. DBA-labelled compounds were derivatized with dansyl chloride before electron-impact GC-MS. Carbosulfan was unstable under the TLC isolation procedure. Peel rinses were analysed within 2 months, non-polar fractions of peel extracts within 6 months and polar, including conjugated, fractions within 12 months. Analyses by methods used in the field trials gave comparable results for carbosulfan and DBA as determined in the metabolism study. Analyses of fractions from stored 15-day peel samples late in the study gave similar results to those of analyses early in the study. This was adduced by the manufacturers as evidence that residues in the peel were stable under the frozen storage conditions for the duration of the study.

Residues of ^{14}C expressed as carbosulfan in the leaves were 16 mg/kg and 12.8 mg/kg after 0 and 30 days respectively from the phenyl label and 9.3 and 4.6 mg/kg from the DBA label. The distribution of ^{14}C in whole oranges and orange components is shown in Table 9, and in analytical fractions in Table 10. The compounds identified from both labels are shown in Table 11.

Table 9. Distribution of ^{14}C in navel oranges and orange components following treatment at nominal rates of 0.5g ai/l with phenyl- or DBA-labelled [^{14}C]carbosulfan (Weintraub, 1996).

Days after treatment	Label	TRR in whole fruit, mg/kg as carbosulfan	% of the TRR in			
			Peel rinse	Peel	Pulp	Juice
0	Phenyl	0.81	95.8	4.1	0.1	0.0
	DBA	0.72	93.9	5.7	0.3	0.2
7	Phenyl	0.85	86.6	13.2	0.1	0.0
	DBA	0.68	86.8	13	0.1	0.0
15	Phenyl	0.81	75.6	23.9	0.2	0.2
	DBA	0.56	75.1	24.5	0.2	0.2
30	Phenyl	0.78	53.7	45.9	0.1	0.3
	DBA	0.59	58.0	41.5	0.2	0.3

Table 10. Distribution of ^{14}C in polar, non-polar and bound fractions of rinsed peels of Navel oranges treated at nominal rates of 0.5g ai /l with phenyl- or DBA-labelled [^{14}C]carbosulfan (Weintraub, 1996).

DAY	Label	Whole Fruit	Rinsed peel	^{14}C fractions from rinsed peel					
				Extractable					
				Non-polar		Polar/conjug.		Bound	
				mg/kg[1]	% of TRR[2]	mg/kg[1]	`% of TRR[2]	mg/kg[1]	% of TRR[2]
0	Phenyl	0.809	0.033	0.029	3.6	0.002	0.2	0.003	0.3
0	DBA	0.702	0.040	0.026	3.7	0.013	1.9	0.001	1.1
15	Phenyl	0.808	0.193	0.153	19.0	0.024	3.0	0.016	2.0
15	DBA	0.563	0.138	0.054	9.6	0.078	13.9	0.006	1.1
30	Phenyl	0.775	0.356	0.274	35.4	0.052	6.7	0.029	3.7
30	DBA	0.588	0.244	0.085	14.5	0.147	25.2	0.012	2.0

[1]carbosulfan equivalent
[2]in whole fruit

Table 11. Compounds identified in 30-day rinses and extracts from of peel oranges of trees 30 days after treatment at nominal rates of 0.5g ai /l with phenyl- or DBA-labelled [^{14}C]carbosulfan (Weintraub, 1996).

Label	Compound	% of the TRR	mg/kg as carbosulfan
Phenyl	Carbosulfan	40.1	0.311
	Carbofuran	33.9	0.263
	Carbosulfan sulfone	3.1	0.025
	3-hydroxy carbofuran	2.0	0.016
	3-keto-carbofuran	2.0	0.016
	N-hydroxymethyl-carbofuran	1.2	0.01
	Dicarbofuran sulfide	1.0	0.007
	7-Phenol	0.4	0.003
	Total identified	83.7	0.63
DBA	Carbosulfan	31.2	0.183
	Dibutylamine	58.2	0.342
	Total Identified	89.4	0.525

The proposed metabolic pathways in oranges are shown in Figure 3.

Environmental fate in soil

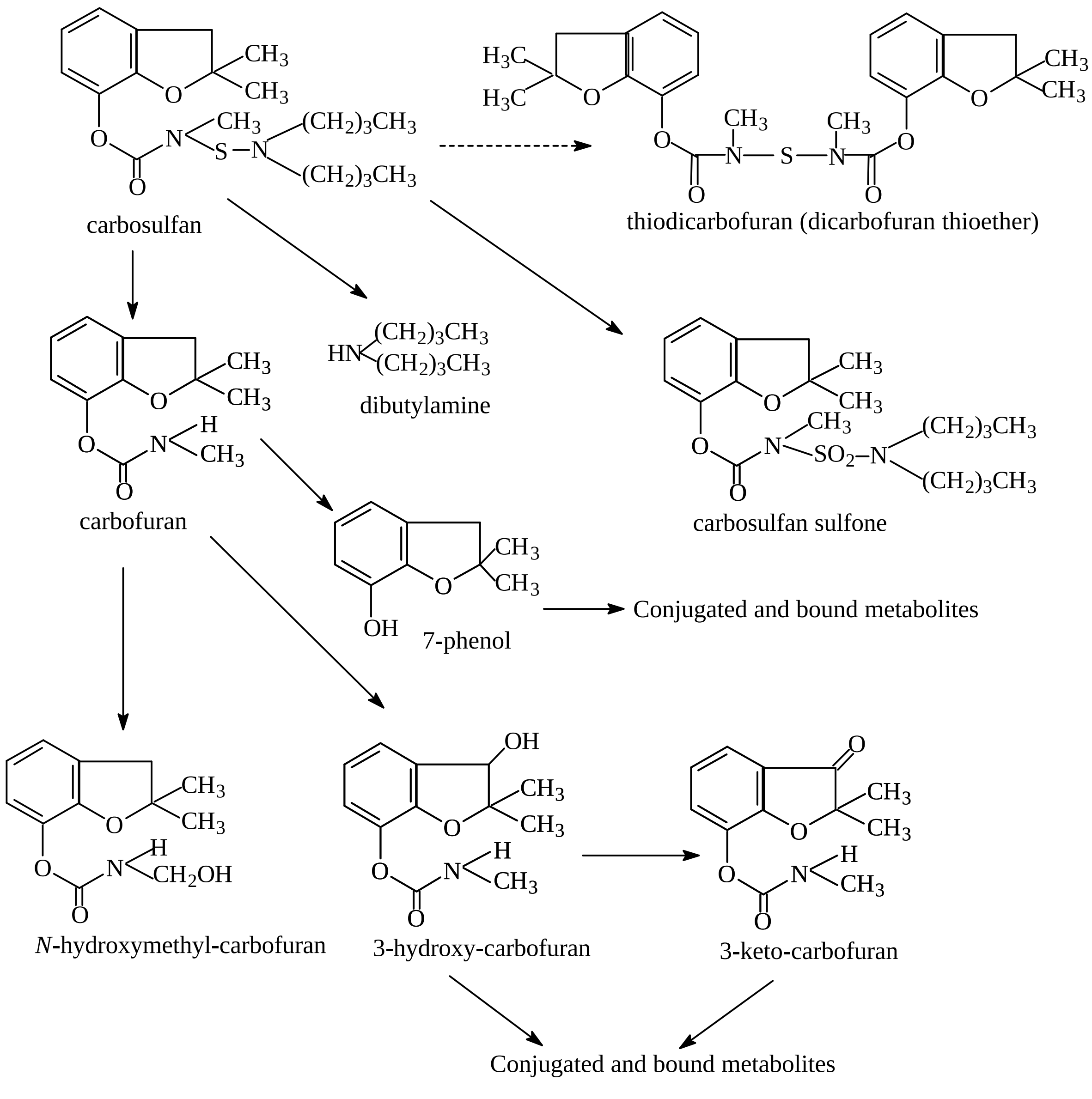
Photodecomposition. The photodecomposition of phenyl- and DBA-labelled carbosulfan was investigated on air-dried (apparently non-sterile) USA New York Dunkirk silt loam soil coated on watch glasses (25 µg ai/g soil) as a thin layer (250µm) (Capps, 1981). The soil characteristics were pH 9, 3.9% organic matter, 51.6% silt, 25.2% sand and 23.2% clay. Irradiation was at 2500 $\mu W/cm^2$ with a sun lamp and samples were removed after 10, 20 and 30 minutes and 1, 5, and 8 days of exposure for analysis by TLC, HPLC, LSC, GLC or in some cases GC-MS. The main compounds identified are shown in Table 12.

Table 12. Compounds identified after irradiating dry soil treated with phenyl- or DBA-labelled carbosulfan for up to 8 days with a sun lamp (Capps, 1981).

Compound	Phenyl label[1], % of TRR				DBA label[1] of TRR			
	10 min.		8 days		10 min.		8 days	
	Irradiated	Control	Irradiated	Control	Irradiated	Control	Irradiated	Control
carbosulfan	1.2	13.4	-	-	11.4	20.1	2.1	0.8
carbofuran	86.4	77	54.5	77.2				
3-hydroxycarbofuran	-	-	3.5	0.7				
carbosulfan sulfone	4.5	3.6	2.6	3.8				
7-phenol	2.6	2.0	1.0	0.5				
3-keto-7-phenol	0.5	-	1.0	-				
N-hydroxymethyl-3-hydroxycarbofuran	-	-	0.8	-				
3-keto-carbofuran	-	-	0.7	0.3				
3-keto-carbosulfan sulfone	-	-	0.6	0.4				
dibutylamine					38.6	43.4	1.8	7.7
N-formyl-dibutylamine					6.4	2.5	4.2	3.1
N-acetyl-dibutylamine					1.1	0.9	1.1	2.0

[1] % of TRR

Figure 3. Proposed metabolic pathways of carbosulfan in oranges (Weintraub, 1996).



The extractability of ^{14}C from the phenyl label in irradiated samples ranged from 96.1% TRR after 10 minutes to 73.3% after 8 days. Decomposition was very rapid. After 10 minutes only 1.2% of the residue was intact carbosulfan, with its sulfone at 4.5%. The main component was carbofuran at 86.4%. After 5 days no carbosulfan was detected, although the sulfone was still 2.7%. After 8 days the identified residues were carbofuran, 3-hydroxycarbofuran, carbosulfan sulfone, the 7-phenol, 3-keto-7-phenol, *N*-hydroxymethyl-carbofuran, 3-keto-carbofuran and 3-keto-carbosulfan sulfone.

A similar rapid degradation was observed with the DBA label, but the extractability was lower (71.2% of the TRR after 10 minutes and 38% after 8 days). After 10 minutes the identified residues were DBA 38.6% of the TRR, carbosulfan 11.4%, *N*-formyldibutylamine 6.4% and *N*-acetyldibutylamine 1.1%. After 8 days these residues were 1.8%, 2.1%, 4.2% and 1.1% of the TRR respectively.

In the same study the author irradiated soil at 70% field moisture, instead of dry soil, treated with DBA-labelled carbosulfan in a similar manner for 48 hours. Intact carbosulfan and DBA accounted respectively for 85.9% and 6.5% of the TRR in irradiated soil and 83% and 10.2% in control soil after 3 hours. After 48 hours the corresponding figures were 76.2 and 86% carbosulfan and 6.7 and 3% DBA.

Environmental fate in water/sediment systems

Water. The photolysis of phenyl- and DBA-labelled carbosulfan was investigated in distilled water, buffered at pH 7 and unbuffed, at concentrations of 5 mg/l with 1% acetonitrile as co-solvent in pyrex flasks (Capps, 1981). The solutions were irradiated at 1500 $\mu W/cm^2$ with a sunlamp and samples were withdrawn for analysis initially and after 1, 4, and 8 days.

About 90% or more of the ^{14}C from both labels was extractable from both buffered and unbuffered water throughout the study, and on 0 day carbosulfan accounted for over 98% of the TRR in all the samples. The major compounds identified in the phenyl- and DBA-label experiments are shown in Tables 13 and 14 respectively.

Table 13. Major compounds identified after irradiating phenyl-labelled carbosulfan in pH 7 buffered and distilled water for 8 days with a sun lamp (Capps, 1981).

Compound	pH 7 buffered water[1]				Distilled water[1]			
	1 day		8 days		1 day		8 days	
	Irradiated	Control	Irradiated	Control	Irradiated	Control	Irradiated	Control
Carbosulfan	72.4	87.6	1.8	46.3	79.7	91.7	22.4	73.4
Carbofuran	12.6	6.1	59.7	37.6	8.6	4.4	38.8	15.8
carbosulfan sulfone	1.2	0.3	4.5	0.4	0.7	0.4	3.1	0.5
7-phenol	1.4	0.3	5.6	2.0	0.9	-	4.3	0.5
3-keto-7-phenol	2.8	0.2	4.5	0.4	2.7	-	1.9	0.3

[1] % of TRR

In buffered water after 1 day of exposure carbosulfan was still the predominant compound, followed by carbofuran and the 3-keto-7-phenol. After 8 days the main compound (60% of the ^{14}C was carbofuran, with the 7-phenol, carbosulfan sulfone, the 3-keto-7-phenol and carbosulfan all below 6%. In distilled water however carbosulfan still accounted for 22.4% of the TRR after 8 days.

Table 14. Major identified compounds after irradiating phenyl-labelled carbosulfan in pH 7 buffered and distilled water for 8 days with a sun lamp (Capps, 1981).

Compound	pH 7 Buffered Water				Distilled Water			
	1 day		8 days		1 day		8 days	
	Irradiated	Control	Irradiated	Control	Irradiated	Control	Irradiated	Control
carbosulfan	76.5	89.2	2.1	57	88.2	96.2	47.3	81.7
dibutylamine	16.6	0.7	8.5	33.8	7.4	-	36.6	12.7
N-formyldibutylamine	1.1	0.3	3.4	2.5	-	3.0	-	-
N-acetyldibutylamine	-	-	-	-	1.5	-	3.4	1.3

In buffered water most of the labelled material in the irradiated sample was not identified after 8 days, but in distilled water carbosulfan and dibutylamine accounted for 47.3 and 36.6% of the TRR respectively.

The degradation of carbosulfan was rapid in buffered water with a half-life of about 1.4 days, but slower in distilled water with a half-life of about 4-8 days.

METHODS OF RESIDUE ANALYSIS

Analytical methods

Two analytical methods were available from the manufacturer for the determination of carbosulfan and metabolites in oranges (one independently validated), one for the determination of residues in processed fractions of oranges and one for the determination of residues in bovine meat and milk (independently validated). A multiresidue method, and methods provided by the government of The Netherlands were also reported.

Method P-2964M (Barros, 1995) was used for the determination of residues in Mexican and Brazilian trials on oranges. The development and application were consistent with GLP principles. It is capable of measuring residues of carbosulfan, its carbamate metabolites carbofuran, 3-keto-carbofuran and 3-hydroxycarbofuran, the 7-phenol, 3-keto-7-phenol, and 3-hydroxy-7-phenol metabolites, and dibutylamine. Whole oranges are homogenized in a Hobart chopper with liquid nitrogen. To determine carbosulfan the sample is extracted with dichlormethane, and the extract is filtered, concentrated, eluted from an aminopropyl solid-phase extraction (SPE) column and concentrated for analysis by reverse-phase HPLC. The HPLC configuration includes a post-column reactor to hydrolyse carbosulfan to carbofuran and a second basic hydrolysis reactor in which carbofuran is derivatized with *o*-phthalaldehyde + *N*,*N*-dimethyl-2-mercaptoethylamine before detection with a fluorescence detector operated at 330 nm excitation and 465 nm emission. The method for the carbamate metabolites is similar, except the sample is hydrolysed with boiling HCl before clean-up directly on C18 and aminopropyl SPE cartridges before HPLC determination. In this case only the second post-column reactor (basic hydrolysis and derivatization) is used.

The determination of phenolics also begins with hydrolysis in boiling HCl, followed by C18-SPE and liquid/liquid partition clean-up before derivatization with pentafluorobenzyl bromide and ethylation of the 3-hydroxy7-phenol. Dibutylamine is extracted with methanol/water (2:1). The extract is cleaned up by organic solvent/water partitions and the dibutuylamine derivatized with dansyl chloride. The derivatized phenolics and dibutylamine are determined by GC-MS (electron ionization) with single-ion monitoring.

The method was validated by six analytical recovery experiments with each compound, 5 at 0.03 mg/kg (the reported limit of determination) and one at 0.1 mg/kg for the carbamates and phenols, 4 at 0.05 mg/kg (the limit of determination) and 2 at 0.1 mg/kg for dibutylamine. The results at 0.1 mg/kg were not significantly different from those at the lower levels. In all cases the limit of detection was <0.01 mg/kg. The reported average percentage recoveries with their standard deviations were carbosulfan 85 ± 21, carbofuran 92 ± 11, 3-keto-carbofuran 89 ± 15, 3-hydroxycarbofuran 100 ± 9, 7-phenol 78 ± 9, 3-keto-7-phenol 81 ± 6, 3-hydroxy7-phenol 76 ± 3, and dibutylamine 89 ± 22. The high variability of the recovery of carbosulfan was attributed to the acidity of oranges, since carbosulfan is unstable at a pH of 4 or lower.

The method P-2964M was independently validated for carbosulfan at 0.03 and 0.1 mg/kg in oranges in two trials (Wood, 1996). The first was not successful at the 0.03 mg/kg fortification level (38.3 and 53.7% recovery) and only marginally so at 0.1 mg/kg (67.6 and 73.4% mg/kg), but the second was satisfactory with 72 and 98.3% recovery at 0.03 mg/kg and 93.8 and 97.6% at 0.1 mg/kg).

Method FCC 0193 (Gill, 1995a) was used for the determination of carbosulfan residues in supervised trials on oranges in Spain. It was developed in accord with GLP principles and measures residues of carbosulfan, carbofuran and 3-hydroxycarbofuran. It actually consists of two methods, one for the first two and the other for 3-hydroxycarbofuran. In analyses for carbosulfan and carbofuran the chopped or homogenized sample is blended with hexane/propan-2-ol (2:1) and the extract is cleaned up by partition between hexane and water, followed by Florisil column chromatography with ethyl acetate/hexane elution. Determination is by GLC with a nitrogen/phosphorus detector. The 3-hydroxycarbofuran is hydrolysed and extracted by HCl reflux, cleaned up by partition with dichloromethane, and derivatized by ethoxylation in acidified ethanol. After liquid/liquid partition and Florisil column clean-up the derivative is determined by GLC with NP detection.

Recoveries of each of the three compounds were determined at 0.01, 0.05 and 0.1 mg/kg (6 replicates at each level). The average percentage recoveries and their SDs were as shown below.

	0.01 mg/kg	0.05 mg/kg	0.1 mg/kg	Controls
carbosulfan	78.5 ±18.5	75.3±3.8	77.0±5.4	<0.01
carbofuran	81.0±13.6	78.7±5.3	85.5±9.7	<0.01
3-hydroxy-carbofuran	86.5±12.2	110.±5.3	109.7±7.1	<0.01

The report showed that carbosulfan was not converted to carbofuran during the procedure. The limit of determination was reported as 0.01 mg/kg for all three compounds (signal to noise ratio 3:1). The limit of "detection" was 50 pg analyte and a "reporting limit" of 0.05 mg/kg was adopted.

Older analytical procedures used for the determination of carbosulfan, carbofuran and 3-hydroxycarbofuran in processed products of citrus (Leppert, 1981) were somewhat similar in principle to those described above (including GLC with NP detection), but there were differences in the extraction and clean-up.

For the determination of carbosulfan and carbofuran macerated frozen samples of juice, finisher pulp or whole fruit are extracted with 2:1 hexane: 2-propanol and cleaned up on an attaclay-aluminum oxide column, concentrated and cleaned up further on a Florisil column. Molasses and dried pulp are extracted with methanol buffered at pH 8 and again cleaned up on attaclay-aluminum oxide and Florisil columns. Oil is partitioned between hexane and acetonitrile (carbosulfan going into hexane and carbofuran to acetonitrile), and the two solutions are cleaned up on the same two columns.

For 3-hydroxycarbofuran determinations samples of whole oranges and processed fractions are hydrolysed by refluxing with HCl (ethanolic HCl for oil), partitioned with methylene chloride and cleaned up on an attaclay-aluminum oxide column with a Florisil column also for finisher pulp and oil. The percentage recoveries from oranges and grapefruit, at the 0.05 mg/kg fortification level unless otherwise indicated, were as shown below.

	carbosulfan	carbofuran	3-hydroxycarbofuran
Unwashed grapefruit	96	74-97/0.2 mg/kg	93/0.2 mg/kg
Unwashed oranges	86/0.2 mg/kg		
Washed oranges	78/0-1 mg/kg	90/10-1mg/kg	88/0.1mg/kg
Washed grapefruit	69/10.1mg/kg		
Orange juice	86	86	
Grapefruit juice	92	94	
Orange molasses	82	100	72
Dried orange pulp (peel)	80	--	
Dried grapefruit pulp			78
Orange finisher pulp			74
Grapefruit finisher pulp	88	94	
Grapefruit oil	78	--	66/0.1 mg/kg

Limits of determination ("sensitivities") were reported as 0.05 mg/kg and limits of detection as 0.01 mg/kg in all samples, except oil where the limits of determination and detection were respectively reported as 0.05 and 0.01 mg/kg for carbosulfan, 1.0 and 0.50 mg/kg for carbofuran and 0.1 (0.01) mg/kg for 3-hydroxycarbofuran.

Animal products

Analytical method P-3065M, developed according to GLP, is available for the determination of carbosulfan, its carbamate metabolites carbofuran, 3-keto-carbofuran, 3-hydroxycarbofuran, the 7-phenol, 3-keto-7-phenol and 3-hydroxy7-phenol metabolites, and dibutylamine in bovine milk, meat and meat byproducts (Chen, 1995a). It was used for the cow feeding study reported below. It is generally similar to method P-2964M. Carbosulfan is extracted from tissues and milk by shaking with acetone (instead of dichloromethane) and partitioned sequentially with dichloromethane and ethyl acetate which are combined, concentrated and analysed by HPLC. Fat and cream extracts are partitioned with acetonitrile before analysis (there is no SPE column clean-up). The HPLC configuration is essentially that used for Method P-2964M. Analyses for the carbamate metabolites also follows Method P-2964M, including the HCl reflux and sequential chromatography on C18- and aminopropyl-SPE columns (cream and fat samples are partitioned with acetonitrile before HPLC). The phenols are extracted by acid reflux as in P-2964M followed by clean-up on a C18-SPE column for milk and C18- and SPE columns for tissues, fat and cream. Fat and cream samples are basified, partitioned with hexane, and the aqueous layer re-acidified before application to the SPE columns. Phenolic samples are derivitized with PFBBr and ethylated. Tissue, fat and cream samples are further eluted through an SI SPE cartridge before concentration for analysis. The phenolic derivatives are determined by single-ion GC-MS as before. The determination of dibutylamine also follows P-2964M, but extraction is with acetone.

Analytical recoveries were determined by analysing 14-19 fortified samples for each analyte of milk and 12-15 of tissues and cream (only 8 for DBA), mainly at low levels. Control levels of all compounds except DBA were <5 in milk and <10 in tissues and cream. Apparent DBA residues ranged from <5 to 37 in milk and <10 to 34 in kidney, fat and muscle. The recoveries were as shown below.

	Milk			Tissues and cream		
	Fortification,	Recovery, %		Fortification,	Recovery, %	
	μg/kg	Mean	± SD	μg/kg	Mean	± SD
carbosulfan	25	87	13	25-100	88	14
carbofuran (CF)	25, 50	93	13	50, 200	85	9
93-keto-CF	25, 50	92	11	50, 200	92	11
3-hydroxy-CF	25, 50	84	13	50, 200	79	15
7-phenol	25, 50	88	12	50, 500	79	24
3-keto-7-phenol	25, 50	99	11	50, 500	114	21
3-hydroxy-phenol	25, 50	104	15	50, 500	97	20
DBA	25	95	21	50-1000	78	12

The limits of determination of all compounds were 25 μg/kg in milk and 50 μg/kg in tissues and cream. The limits of detection were 5 and 10 μg/kg respectively.

The method was validated by an independent laboratory (Burton, 1996a) for carbosulfan in milk at 25 and 50 μg/kg (recovery 92%, 7.2 SD) and (Burton, 1996b) for carbofuran (97.7% ±7.1 SD), 3-keto-carbofuran (94.2% ±17.9 SD) and 3-hydroxycarbofuran (99.1% ±4.3 SD) in milk at 25 and 100.

Multi-residue methods

Methods in the US EPA's PAM testing protocols were tested for the detection and recovery of carbosulfan (Mayer, 1995). Although carbosulfan was successfully eluted from Florisil columns under some PAM conditions, the sensitivity of the detection systems (including the most sensitive GLC N/P detection) was not considered sufficient at the 0.05 mg/kg level in fatty or non-fatty foods (≥25 ng required for half-scale deflection compared with 1.5 ng for chlorpyrifos, and 1.5 μg/ml considered to be the limit of quantification, defined as average noise +10 x SD).

The government of The Netherlands official multi-residue method Part 1, Method 1 was provided as a method for carbosulfan (Olthof, 1997). Samples are extracted with acetone/dichlormethane/petroleum ether. There is no clean-up and an EC or ion trap detector is used. No validation data were provided for carbosulfan in specific types of sample.

Stability of pesticide residues in stored analytical samples

Several studies have been carried out on the stability of carbosulfan and/or its metabolites in crops, soils or animal products during frozen storage. The studies which include carbosulfan are described here. Other studies on the stability of carbofuran and its metabolites are discussed in the monograph on carbofuran.

`Markle (1980) studied the stability of carbosulfan in alfalfa, oranges and three types of soil stored for a year at 0° (-18°C). Samples of treated oranges (two applications at 1.7 kg ai/ha) and alfalfa (1.12 kg ai/ha) were macerated and mixed before storage. Air-dried soils were treated at 1 mg ai/kg soil. The soils and dry alfalfa were extracted with methanol buffered at pH 8, and green alfalfa and oranges with hexane/propanol (as in the method of Leppert, 1981). Clean-up was by liquid/liquid partition and Florisil column chromatography and carbosulfan was determined by GLC with NP detection. Samples were analysed on the day of application and at intervals during storage for one year. The residue levels after selected intervals are shown below in Table 15.

Table 15. Residues of carbosulfan and carbofuran in carbosulfan-treated crops and soils at intervals after storage at -18°C (Markle, 1980).

Sample	Carbosulfan, mg/kg[1]			Carbofuran, mg/kg[1]		
	Day 0	Day 179	Day 365	Day 0	Day 179	Day 365
Oranges	1.5	1.6	1.5	0.31	0.4	0.53
Green alfalfa	32	34	30	2.9	3.8	3.8
Alfalfa hay	44	50 [2]	45	10	13	12
	Day 0	Day 144	Day 357	Day 0	Day 144	Day 357
Silt loam pH 4.8	Carbosulfan was almost completely degraded after 3 hours. This was attributed to the soil acidity.					
Silty clay loam pH 6.0	0.96	0.51	0.40	0.41	0.47	0.61
Sandy loam pH 6.8	0.63	0.3	0.24	0.15	0.37	0.33

[1] Average of three replicate samples at each sampling[2] Day 185

In the silty clay loam and sandy loam soils the combined residues of carbosulfan and carbofuran expressed as carbosulfan were fairly constant during the storage period.

In an interim report Pearsall (1996) described studies of the stability of carbosulfan, carbofuran, 3-hydroxycarbofuran, 3-keto-carbofuran, the 7-phenol, 3-keto-7-phenol, and 3-hydroxy-7-phenol and dibutylamine in laboratory-fortified oranges and their processed products stored for up to a year at -18°C. Whole oranges, dried pulp, juice, molasses and oil were fortified at 0.25 mg/kg and samples of most of these were taken for analysis on day 0 however, because degradation was rapid, and after approximately 3, 6, and 12 months. Analyses of juice, molasses and oil for carbosulfan were discontinued after day 0 and orange oil was analysed for the metabolites only on day 0 and after 12 months. The reported results were the averages of analyses of triplicate samples. The limits of detection and determination were reported to be 0.025 and 0.125 mg/kg respectively for all compounds in all samples. The results obtained with pulp, juice, molasses and oil are shown in Table 16.

Table 16. Residues of carbosulfan, its carbamate and phenolic metabolites and dibutylamine in processed orange products, each fortified at 0.25 mg/kg and stored for up to one year at -18°C (Pearsall, 1996).

Compound	Residues, mg/kg[1], after storage			
	0 days	3 months	6 months	12 months
DRIED PULP				
carbosulfan	0.24	0.11	0.18	NA[2]
carbofuran	0.22	0.27	0.21	0.30
3-hydroxycarbofuran	0.24	--	0.19	0.24
3-keto-carbofuran	0.21	0.23	0.26	0.26
dibutylamine	0.20	0.21	0.26	0.25
7-phenol	0.22	0.26	0.22	0.29
3-keto-7-phenol	0.21	0.24	0.23	0.23
3-hydroxy-7-phenol	0.20	0.23	0.34	0.22
JUICE				
Carbosulfan	ND	NA	NA	NA
carbofuran	0.24	0.25	0.24	0.25
3-hydroxycarbofuran	0.23	0.21	0.19	0.25
3-keto-carbofuran	0.25	0.23	0.19	0.27
dibutylamine	0.31	0.33	0.21	0.23
7-phenol	0.23	0.28	0.23	0.28
3-keto-7-phenol	0.24	0.25	0.23	0.26
3-hydroxy -7-phenol	0.24	0.28	0.21	0.20
MOLASSES				
carbosulfan	0.08	NA	NA	NA
carbofuran	0.25	0.31	0.26	0.27
3-hydroxycarbofuran	0.21	0.31	0.25	0.27
3-keto-carbofuran	0.22	0.25	0.20	0.22
dibutylamine	0.20	0.28	0.32	0.24
7-phenol	0.25	0.20	0.23	0.27
3-keto-7-phenol	0.21	0.21	0.25	0.27
3-hydroxy-7-phenol	NA	0.21	0.34	0.20
OIL				
carbosulfan	0.12	NA	NA	NA
carbofuran	0.25	NA	NA	0.24
3-hydroxycarbofuran	0.21	NA	NA	0.29
3-keto-carbofuran	0.23	NA	NA	0.25
dibutylamine	NA	NA	NA	0.13
7-phenol	0.19	NA	NA	0.23[2]
3-keto-7-phenol	0.23	NA	NA	0.22[2]
3-hydroxy-7-phenol	0.22	NA	NA	0.22[2]

NA = Not analysed ND = not detected (<0.025 mg/kg)
[1] Average of 3 replicate samples
[2] Two samples only. Third not included because it was inconsistent with other samples

In a study of the stability of carbosulfan and DBA in bovine milk and tissues (Barrett, 1996), samples of milk, muscle and liver were laboratory-fortified at 0.25 mg/kg with either DBA or carbosulfan and stored for 8 months at -18°C. Samples were analysed initially and after 3, 6, or 8 months storage by methods in FMC report P-3065 described above under methods of analysis. Limits of detection and determination for all the compounds were 5 and 25 µg/kg in milk and 10 and 25 µg/kg in tissues. Results are shown in Table 17.

Table 17. Stability of carbosulfan and dibutylamine added to bovine milk, muscle and liver at 0.25 mg/kg and stored up to 8 months at -18°C (Barrett, 1996).

Compound/sample	Residues (mg/kg) at Storage Intervals (months)							
	0		3		6		8	
	Mg/kg	% change	mg/kg	% change	mg/kg	% change	mg/kg	% change
Carbosulfan Milk	0.21	-16	0.15	-40	0.09	-64	0.04	-84
Muscle	0.25	0.0	0.05	-80	0.02	-92	ND	-100
Liver	0.24	-4	0.07	-72	0.07	-72	0.05	-80
Dibutylamine Milk	0.26	+4	0.31	+24	0.23	-8	NA	NA
Muscle	0.31	+24	0.23	-8	0.38	+52	NA	NA
Liver	0.24	-4	0.16	-36	0.24	-4	NA	NA

ND = not detected (<0.01 mg/kg)
NA = not analysed

Because of the losses of carbosulfan mass balances were determined in an attempt to confirm a hypothesis that most of the losses could be attributed to degradation to carbofuran or phenols. Residues of carbosulfan and carbofuran were determined after 3 and 6 months (Table 18), and those of carbosulfan, carbofuran and the 7-phenol after 8 months (Table 19).

Table 18. Carbosulfan and carbofuran expressed as carbosulfan in bovine milk, muscle and liver which had been fortified with 0.25 mg/kg carbosulfan and stored at -18°C (Barrett, 1996).

Sample	Storage period, Months	Residue, mg/kg			Total residue as % of fortification level
		Carbosulfan	Carbofuran as carbosulfan	Total	
Milk	3	0.15	0.02	0.17	68
Muscle	6	0.02	0.2	0.22	88
Liver	6	0.07	0.04	0.11	0.44

Table 19. Carbosulfan, carbofuran and 7-phenol expressed as carbosulfan in bovine milk, muscle and liver which had been fortified with carbosulfan at 0.25 mg/kg carbosulfan (Barrett, 1996).

Sample	Storage period, months	Residue, mg/kg				Total residue as % of fortification level
		Carbosulfan	Carbofuran as carbosulfan	7-phenol	Total	
Milk	8	0.04	0.11	0.01	0.16	64
Muscle	8	ND	0.18	0.19	0.37	148
Liver	8	0.05	0.03	0.05	0.13	52

Definition of the residue

Previously the JMPR had recommended separate limits for carbosulfan and carbofuran to accommodate residues from uses of carbosulfan. This is consistent with the internationally accepted approach of recommending separate MRLs and definitions of residues when a metabolite of a pesticide is also a pesticide in its own right, as is carbofuran. Residues of the carbamate metabolites have been determined separately for the estimation of maximum residue levels of carbofuran, regardless of whether they result from the use of carbofuran or carbosulfan. The Meeting therefore concluded that residues of carbosulfan should be defined as carbofuran.

USE PATTERN

Reported information on GAP is shown in Table 20.

Table 20. GAP for the use of carbosulfan on citrus fruit.

Country Crop	Application				PHI, Days	Comments
	Form.	g ai/ha or [g ai/tree]/appl.	l/ha	No.		
Mexico Valencia oranges	26.1% LE	250	1000	4	7	Last broadcast applic. *c.*230 days after bloom. Ref. Report P-3182.
Other oranges	26.1%	250	1000	3	7	Last broadcast applic. *c.*230 days after bloom. Ref. Report P-3183
Brazil Oranges	CE 250 g ai /l	[0.93-1.69]	----	2	7	Last broadcast spray *c.*50 days after bloom. Ref. Report P-2964.
Spain Valencia oranges	LE 250 g ai /l	[2.83-3.14]	141-156 l/trial	2	112	Last broadcast foliar application *c.*28-30 days after bloom. Ref. Report P-3100.
Mandarin oranges	LE 250 g ai /l	[3.2-3.6]	160-180 l/trial	2	112	Last broadcast foliar application *c.*28 days after bloom. Ref. Report No. P-3101
Oranges	250EC	937.5	3000	2	123-147	
Mandarins	250EC	937.5	3000	2	110	
Clementines	250EC	937.5	3000	2	115	

RESIDUES RESULTING FROM SUPERVISED TRIALS

Plants

Oranges. Seven supervised trials were conducted in Mexico, 6 in Brazil and 17 in Spain. The analytical methods were P-2964 in Mexico and Brazil and FCC 0193in Spain, described above. Limits of detection and determination of 0.01 and 0.05 mg/kg respectively in citrus fruits and their products are reasonable for carbosulfan and its metabolites except dibutylamine, for which a limit of determination of 0.1 mg/kg would be more realistic because apparent DBA is frequently found at about 0.02 mg/kg in control samples. All the trials were in accordance with reported GAP. Generally spray was to run-off and 6 trees were treated in each trial. Precautions were taken during sampling shipment and storage to preserve the integrity of the samples. The results are shown in Tables 21 and 22. Duplicate samples were taken in most trials and the two results have been recorded. The results for carbosulfan and the carbamate metabolites were corrected for analytical recoveries where they were low.

Table 21. Residues of carbosulfan and metabolites from supervised trials on oranges and mandarins. Whole fruit analysed. Underlined results were used for estimation of maximum residue and STMR levels. Details of the trials follow the Table.

Trial ref.	PHI, days	Sample[1]	Residues, mg/kg[2]										
			Sulfan	Furan	CO-furan	HO-furan	Furan + HO-furan	Total carba-mate	7-phenol	CO-phenol	HO-phenol	Total phenols	DBA
Trial A1	7	NCOR C	0.04 0.01 ND	0.13 0.19 ND	0.01 0.04 ND	0.07 0.11 ND	0.20 0.30	0.25 0.35 ND	0.02 0.02 ND	0.01 0.01 ND	0.04 0.08 ND	0.07 0.11 ND	0.11 0.11 0.02
		COR	0.08 0.02	0.17 0.25	0.01 0.04	0.09 0.14	0.26 0.39	0.35 0.45					
Trial A2	7	NCOR C	0.01 0.01 ND	0.08 0.08 ND	0.01 0.02 ND	0.03 0.02 ND	0.11 0.10	0.13 0.13 ND	0.01 ND ND	0.01 ND ND	0.02 0.02 ND	0.04 0.02 ND	0.14 0.14 0.01
		COR	0.02 0.02	0.11 0.11	0.01 0.02	0.04 0.03	0.15 0.14	0.18 0.18					
Trial A3	7	NCOR C	ND ND ND	0.2 0.12 ND	0.04 0.02 ND	0.11 0.05 ND	0.31 0.17	0.35 0.19 ND	0.02 0.01 ND	0.03 0.01 ND	0.07 0.03 ND	0.12 0.05 ND	0.02 0.02 ND
		COR	ND ND	0.26 0.16	0.04 0.02	0.14 0.06	0.40 0.22	0.44 0.24					
Trial A4	7	NCOR C	0.02 ND ND	0.11 0.15 ND	ND 0.01 ND	0.06 0.10 ND	0.17 0.16	0.19 0.26 ND	0.02 0.03 ND	ND 0.01 ND	0.04 0.06 ND	0.06 0.07 ND	0.10 0.09 ND
		COR	0.04 ND	0.14 0.20	ND 0.01	0.08 0.13	0.22 0.33	0.26 0.34					
Mean analytical recoveries, %			51	76	93	80		--	64	74	57	--	65
Trial B1	7	NCOR C	0.02 0.01 ND	0.07 0.07 ND	ND 0.02 ND	ND 0.02 ND	0.07 0.09	0.09 0.12 ND	ND 0.01 ND	ND ND ND	0.03 0.04 ND	0.03 0.05 ND	0.06 0.06 ND
		COR	0.03 0.02	0.08 0.08	ND 0.03	ND 0.03	0.08 0.11	0.11 0.16					
Trial B2	7	NCOR C	ND ND ND	0.08 0.07 ND	ND ND ND	0.05 0.02 ND	0.13 0.09	0.13 0.09 ND	ND ND ND	ND 0.01 ND	0.02 0.03 ND	0.02 0.04 ND	0.07 0.07 0.01
		COR	ND ND	0.10 0.08	ND ND	0.07 0.03	0.17 0.11	0.17 0.11					
Trial B3	7	NCOR C	ND ND ND	0.09 0.09 ND	0.02 0.02 ND	0.06 0.04 ND	0.15 0.13	0.17 0.15 ND	ND ND ND	ND ND ND	0.03 0.03 ND	0.03 0.03 ND	0.06 0.04 0.01
		COR	ND ND	0.11 0.11	0.03 0.03	0.08 0.06	0.19 0.17	0.22 0.20					
Mean analytical recoveries, %			64	83	72	72			55	69	53		70

Trial ref.	PHI, days	Sample[1]	Residues, mg/kg[2]										
			Sulfan	Furan	CO-furan	HO-furan	Furan + HO-furan	Total carba-mate	7-phenol	CO-phenol	HO-phenol	Total phenols	DBA
Trial C1	7	NCOR	ND 0.02	0.04 0.02	ND ND	0.01 ND	0.05 0.02	0.05 0.04	ND ND	ND ND	ND ND	ND ND	0.07 0.06
Trial C2	7	NCOR	ND ND	0.05 0.06	ND ND	0.03 0.02	0.08 0.08	0.08 0.08	ND ND	ND ND	0.02 0.01	0.02 0.01	0.09 0.07
Trial C3	7	NCOR	ND ND	0.03 0.06	ND 0.02	0.02 0.03	0.05 0.09	0.05 0.11	ND ND	ND ND	ND 0.02	ND 0.02	0.05 0.04
Trial C4	7	NCOR	0.02 0.02	0.02 0.01	ND ND	0.01 0.01	0.03 0.02	0.05 0.04	ND ND	ND ND	0.01 ND	0.01 ND	0.07 0.03
Trial C5	7	NCOR	0.03 0.03	0.03 0.04	ND ND	0.02 0.02	0.05 0.06	0.08 0.09	ND ND	ND ND	0.01 0.02	0.01 0.02	0.13 0.15
Trial C6	7	NCOR	0.01 0.01	0.05 0.06	ND ND	0.02 0.03	0.07 0.09	0.08 0.10	ND ND	ND ND	ND 0.01	ND 0.01	0.05 0.04
Mean analytical recoveries, %			85	92	89	100			78	81	76	--	89
Trial D1	28	NCOR C	ND ND	0.61 0.40	ND ND	0.35 0.13	0.96 0.53	0.96 0.53	0.05 0.07	0.02 0.02	0.13 0.19	0.20 0.28	0.17 0.29 0.03
		COR	ND ND	0.82 0.54	ND ND	0.39 0.14	1.2 0.68	1.2 0.68					
	112	NCOR C	ND ND	0.07 0.27	ND 0.04	0.02 0.13	0.09 0.40	0.09 0.44	0.02 0.05	ND 0.03	0.05 0.17	0.07 0.25	0.12 0.14 0.02
		COR	ND ND	0.10 0.36	ND 0.05	0.02 0.14	0.12 0.50	0.12 0.55					
Trial D2	28	NCOR C	ND ND	0.11 0.14	ND ND	0.17 0.27	0.38 0.41	0.38 0.41	0.06 0.05	0.05 0.04	0.34 0.32	0.45 0.41	0.20 0.20 0.07
		COR	ND ND	0.15 0.19	ND ND	0.19 0.30	0.34 0.49	0.34 0.49					
	113	NCOR	ND ND	ND 0.02	0.01 ND	0.05 0.10	0.05 0.12	0.06 0.12	0.01 0.01	0.01 ND	0.06 0.06	0.08 0.07	0.09 0.11
		COR	ND ND	ND 0.03	0.01 ND	0.06 0.11	0.06 0.14	0.06 0.14					
Mean analytical recoveries, %			82	74	81	90			106	103	80		66
Trial E1	28	NCOR C	ND ND	0.23 0.16	ND ND	0.30 0.20	0.26 0.18	0.26 0.18	0.03 0.03	0.23 0.24	0.37 0.41	0.63 0.67	0.09 0.07 0.03
		COR		0.26 0.18	ND ND	0.34 0.23	0.34 0.23	0.60 0.41					
	112	NCOR	ND ND	ND ND	0.01 ND	0.09 0.06	0.09 0.06	0.10 0.06	ND ND	ND ND	0.10 0.08	0.10 0.08	0.06 0.05
		COR	ND ND	ND ND	0.01 ND	0.10 0.07	0.10 0.07	0.11 0.07					

Trial ref.	PHI, days	Sample[1]	Residues, mg/kg[2]										
			Sulfan	Furan	CO-furan	HO-furan	Furan + HO-furan	Total carba-mate	7-phenol	CO-phenol	HO-phenol	Total phenols	DBA
Trial E2	28	NCOR	ND ND	0.18 0.12	ND ND	0.13 0.14	0.31 0.26	0.31 0.26	0.01 ND	ND ND	0.10 0.10	0.20 0.20	0.24 0.29
		C											0.10
		COR	ND ND	0.21 0.14	ND ND	0.15 0.16	0.36 0.30	0.36 0.16					
	112	NCOR	ND ND	ND ND	ND ND	0.02 0.03	0.02 0.03	0.02 0.03	ND ND	ND ND	0.02 0.04	0.02 0.04	0.04 0.04
		COR	ND ND	ND ND	ND ND	0.02 0.03	0.02 0.03	0.03 0.03					
Mean analytical recoveries, %			88	88	92	88			87	87	91		67

[1]C = control . COR = corrected for recovery. NCOR = not corrected for recovery

[2]Sulfan = carbosulfan; Furan = carbofuran; CO-furan = 3-keto-carbofuran; HO-furan = 3-hyrdroxy-carbofuran; CO-phenol = 3-keto-7-phenol; HO-phenol = 3-hydroxy-7-phenol; DBA = dibutylamine. All results below the limits of determination indicated for each reference (0.03 or 0.05 mg/kg) are estimated values. ND = < limit of detection of 0.01 mg/kg. In each trial duplicate field samples were analysed and both results are shown. Some individual field samples were analysed in duplicate. In these cases the means are recorded.

A trials

Mexico, 1995 (Barros, 1996a) Valencia oranges.25LE formulation, 4 x 250 g ai/ha.

LOD 0.05 mg/kg. Recoveries determined at 0.05-0.5 mg/kg, DBA 0.05-0.2 mg/kg. No appreciable difference between 0.05 and 0.5. 20-30 trees/plot, 6 trees per plot sprayed to run-off by backpack sprayer

Trial 1: Nuevo Leon, 979-1025 l spray/ha.
Trial 2: Tamaulipas, 940-1000 l spray/ha.
Trial 3: Veracruz, 966-1035 l spray/ha.
Trial 4: Sonora, 990-1030 l spray/ha.

B trials

Mexico 1995 (Ramsey and Barros, 1996). Other oranges. 25LE formulation, 3 x 250 g ai/ha.

LOD 0.05 mg/kg. Recoveries determined at 0.05-0.5 mg/kg, DBA 0.05-0.1 mg/kg. No appreciable difference between 0.05 and 0.5 except HO-furan 61% at 0.05 mg/kg

Trial 1: Nuevo Leon, 976-1025 l spray/ha.
Trial 2: Tamaulipas, 955-1000 l spray/ha.
Trial 3: Veracruz, 977-1020 l spray/ha.

C trials

Brazil 1993 (Shevchuk, 1996). Oranges, Pera Valencia and Pera Coroa. All trials Campinas region, 2 x 250CE formulation. LOD 0.03 mg/kg except DBA 0.05 mg/kg. Recoveries determined at 0.03-0.1 mg/kg, DBA 0.05-0.1 mg/kg.

Trial 1 1.7 and 1.1 g ai/tree; 410 and 250 l spray/trial.
Trial 2 0.93 and 1.0 g ai/tree; 268 and 240 l spray/trial.
Trial 3 1.0 and 1.1 g ai/tree; 300 and 360 l spray/trial.
Trial 4 1.4 and 1.4 g ai/tree; 330 and 360 l spray/trial.
Trial 5 1.6 and 1.5 g ai/tree; 368 and 360 l spray/trial.
Trial 6 1.4 and 1.5 g ai/tree; 332 and 360 l spray/trial.

D trials

Spain 1994 (Barros, 1996b). Valencia oranges. 25LE formulation, 2 x 3.1 g ai/tree.

Method P-2719 for carbamates, P-0748 for DBA; 6 trees from 30-tree plot treated, 1st treatment post-bloom. LOD 0.03 mg/kg except DBA 0.05 mg/kg. Recoveries determined at 0.03-0.5 mg/kg except carbosulfan 0.03 mg/kg. Controls all ND except DBA where shown

Trial 1 Lepe, 153 and 156 l spray/trial.
Trial 2 Santiponce , 141 and 142 l spray/trial.

E trials

Spain, 1994 (Barros, 1996c). Mandarin oranges. 25LE formulation.

LOD 0.03 mg/kg. Controls ND except DBA where shown. Two applications (1st after full bloom and 2nd 28 days later) to 6 centre trees of 20-tree plots with broadcast spray to run-off. Recoveries determined at 0.03-0.5 mg/kg, except DBA 0.05-0.5 mg/kg and carbosulfan only 0.03 mg/kg. Recoveries not substantially affected by fortification levels.
Trial 1 Lepe. 3.3 and 3.6 g ai/tree; 160 and 180 l spray/trial.
Trial 2 La Algaba. 3.2 and 3.2 g ai/tree; 162 and 180 l spray/trial.

Table 22. Residues of carbosulfan, carbofuran and 3-hydroxycarbofuran in oranges and mandarins resulting from supervised trials in Spain. All 2 applications of 250 EC formulation at 937.5 kg ai/ha, 3000 l/ha. Underlined results were used for estimation of maximum residue and STMR levels.

Location, Variety	PHI, Days	Sample[1]	Residue, mg/kg[2]					Refs. & comments
			Sulfan	Furan	HO-furan	Furan + HO-furan	Total carba-mates	
Benifay	0	Fruit						
		C	0.03	0.06	0.24*			Gill, 1995b
		NCOR	2.6	0.94	0.45			1 trial in 1993
Clementines			1.8	0.78	0.46			
		COR	3.3	1.0	0.45	1.5	4.8	First applicn. At 5% final fruit size.
			2.3	0.83	0.46	1.3	2.1	Report states 1875 g ai/ha (presumably 2 x 937.5) .Summary reports 937.6
	30	C	ND	ND	0.05*			Controls decreasing with time suggests contamination of control plot. Plot diagrams support this.
		NCOR	ND	0.08	0.29			
			ND	0.17	0.46			
		COR	ND	0.09	0.29	0.38	0.38	Results not corrected for 111% recovery at any PHI
			ND	0.18	0.46	0.64	0.64	
	60	C	ND	ND	ND*			
		NCOR	ND	ND	0.12			
			ND	ND	0.11			
		COR	ND	ND	0.12	0.12	0.12	
			ND	ND	0.11	0.11	0.11	
	115**	C	ND	ND	ND*			** Normal harvest
		NCOR	ND	ND	0.05			
			ND	ND	0.06			
		COR	ND	ND	0.05	0.05	0.05	
			ND	ND	0.06	0.06	0.06	
Mean recovery, %, at 0.05 mg/kg					111			
Trial 1	0	Fruit (peel + pulp						Gill, 1995c
								2 trials in 1993
Satsuma mandarins		C	0.01	ND	0.02			
		NCOR	1.0	0.81	0.92			Report states 1875 g ai/ha (presumably 2 x 937.5) .Summary of report states.2 x 1875. Summary in mfgr.'s evaluation states total of 937.5.
			0.84	0.56	0.76			
Carlet		COR	1.3	0.96	0.92	1.9	3.2	
			1.1	0.67	0.76	1.4	2.5	
	45	C	ND	ND	ND			
		NCOR	ND	0.05	0.60			
			ND	0.04	0.19			
		COR	ND	0.06	0.60	0.66	0.66	
			ND	0.05	0.19	0.24	0.66	
	110**	C	ND	ND	ND			** Normal harvest
		NCOR	ND	ND	0.11			
			ND	ND	0.12			
		COR	ND	ND	0.11	0.11	0.11	
			ND	ND	0.12	0.12	0.12	

Location, Variety	PHI, Days	Sample[1]	Residue, mg/kg[2] Sulfan	Furan	HO-furan	Furan + HO-furan	Total carba-mates	Refs. & comments
Trial 2 Sueca	0	C NCOR	ND 1.9 1.0	ND 0.80 0.67	ND 0.76 0.88			
		COR	2.4 1.3	0.95 0.80	0.76 0.80	1.7 1.6	3.1 2.9	
	45	C NCOR	ND ND ND	ND 0.04 0.04	0.01 0.50 0.40			
		COR	ND ND	0.05 0.05	0.50 0.40	0.55 0.45	0.55 0.40	
	110	C NCOR	ND ND ND	ND ND ND	ND 0.11 0.13			
		COR	ND ND	ND ND	0.11 0.13	0.11 0.13	0.11 0.13	
Mean recovery, %, at 0.05 mg/kg			79	84	103			
Trial 1 Carlet Navel oranges	147	Fruit (Peel + pulp)* C Treated	ND ND ND	ND 0.03 0.02	ND 0.04 0.07	ND 0.07 0.09	ND 0.07 0.09	Gill, 1995d 3 trials in 1993 Results corrected for recovery only for sulfan (86% at 0.05 mg/kg). Furan and HO-furan were ∃ 95% *Peel/pulp ratio about 40/60. Peel/pulp residues are means of duplicate samples
Trial 2 Sueca Newhall oranges	0	Fruit C Treated	ND 0.49 0.81	0.02 0.45 0.53	ND 0.94 0.75	ND 1.7 1.3	0.02 2.2 2.1	
	45	C Treated	ND ND ND	ND 0.02 0.13	ND 0.21 0.17	ND 0.23 0.30	ND 0.25 0.43	
		peel*	ND	0.27	0.36	0.65	0.65	
		pulp*	ND	ND	0.01	0.01	0.01	
	105	C Treated	ND ND ND	ND 0.06 0.04	ND 0.05 0.04	ND 0.11 0.08	ND 0.11 0.12	
	123**	C Treated	ND ND ND	ND 0.04 0.04	0. 03 0.08 0.08	0.03 0.12 0.12	0.03 0.12 0.12	** Normal harvest
Trial 3 _ Benifay Navel oranges	0	Peel + pulp C	ND 0.32 0.44	ND 0.37 0.45	ND 0.23 0.64	ND 0.60 1.1	ND 0.92 1.5	
	45	C Treated	ND ND ND	ND 0.13 0.17	ND 0.08 0.24	ND 0.21 0.41	ND 0.21 0.41	

Location, Variety	PHI, Days	Sample[1]	Residue, mg/kg[2]					Refs. & comments
			Sulfan	Furan	HO-furan	Furan + HO-furan	Total carba-mates	
		Peel	ND	0.37	0.36	0.73	0.73	
		Pulp	ND	ND	0.01	0.01	0.01	
	105	C Treated	ND ND ND	ND 0.04 0.04	ND 0.03 0.04	ND 0.07 0.08	ND 0.07 0.08	
	140	C Treated	ND ND ND	ND 0.02 0.03	ND 0.05 0.08	ND 0.07 0.11	ND 0.07 0.11	
Trial 1 Benifay Clementines	0	Whole fruit C Treated	0.01 0.50	ND 0.69	0.02 0.52	0.02 1.2	0.03 1.7	Gill, 1996a 2 trials in 1994 Results not corrected as all were ∃96% at 0.05 mg/kg. .
	30	C Treated	ND ND	ND 0.12	ND 0.35	ND 0.47	ND 0.59	
	60	C Treated	ND ND	ND 0.01	0.01 0.10	0.01 0.11	0.01 0.11	
	104*	C Treated	ND ND	ND ND	ND 0.06	0.11 0.06	0.11 0.06	* Normal harvest
Trial 2 Catadau Clementines	0	Whole fruit* C Treated	<0..02 0.65	ND 0.46	<0.02 0.33	<0.04 0.79	<0.06 1.4	* Estimated from measured peel/pulp ratios at each sampling
		Peel	0.9	0.63	0.44	1.1	2.0	
		Pulp	ND	ND	0.03	0.03	0.03	
	30	C Treated	ND ND	ND <0.02	ND 0.21	ND <0.23	ND <0.23	
		Peel	ND	0.03	0.55	0.58	0.58	
		Pulp	ND	ND	0.02	0.02	0.02	
	60	C Treated	ND ND	ND ND	0.02 <0.06	0.02 <0.06	0.02 <0.06	
		Peel	ND	ND	0.22	0.22	0.22	
		Pulp	ND	ND	ND	ND	ND	
	104**	C Treated	ND ND	ND ND	ND <0.04	ND <0.04	ND <0.04	** Normal harvest
		Peel	ND	ND	0.14	0.14	0.14	
		Pulp	ND	ND	ND	ND	ND	

Location, Variety	PHI, Days	Sample[1]	Residue, mg/kg[2] Sulfan	Furan	HO-furan	Furan + HO-furan	Total carba-mates	Refs. & comments
Trial 1	0	Whole fruit*						Gill, 1996b 2 trials in 1994
Carlet		C	0.02	0.02	0.04	0.06	0.08	
		Treated	0.31	0.51	0.39	0.90	1.2	* Estimated from measured peel/pulp ratios at each sampling
Satsumas	45	C	ND	ND	0.01	0.01	0.01	
		Treated	ND	ND	0.18	0.18	0.18	Results not corrected for recoveries of ∃94%.
	84	C	ND	ND	0.03	0.03	0.03	
		Treated	ND	ND	0.04	0.04	0.04	
Trial 2	0	Whole fruit *						
Sueca		C	<0.02	ND	<0.02	<0.02	<0.04	
Satsumas		Treated	0.43	0.28	0.41	0.69	1.1	
		Peel	0.70	0.45	0.64	1.1	1.8	
		Pulp	ND	0.02	0.05	0.07	0.07	
	45	C	ND	ND	ND	ND	ND	
		Treated	<0.02	<0.03	0.30	<0.33	<0.35	
		Peel	0.04	0.07	0.73	0.8	0.84	
		Pulp	ND	ND	0.03	0.03	0.03	
	92	C	ND	ND	0.02	0.02	0.02	
		Treated	ND	ND	0.13	0.13	0.13	
		Peel	ND	ND	0.40	0.40	0.40	
		Pulp	ND	ND	0.02	0.02	0.02	
Trial 1	0	Whole fruit						Gill, 1996c 3 trials in 1994
Benifay		C	ND	0.14	0.09			
		NCOR	0.56	0.83	0.30			
Naveline oranges		COR	0.62	1.0	0.41	1.4	2.0	
	45	C	ND	0.09	0.03			
		NCOR	ND	0.21	0.17			
		COR	ND	0.26	0.23	0.49	0.49	
	105	C	ND	0.03	0.01			
		NCOR	ND	0.07	0.11			
		COR	ND	0.09	0.15		0.24	
	140	C	ND	0.02	0.01			
		NCOR	ND	0.03	0.05			
		COR	ND	0.04	0.07		0.11	
Mean recovery, %, at 0.05 mg/kg			90	80	74			
Trial 2	0	Whole fruit *						Estimated from peel/pulp. ratio at harvest of 27/73
Carlet		C	<0.02	ND	ND			
Naveline		NCOR	0.17	0.18	0.08			
oranges		COR	0.19	0.23	0.11	0.34	0.53	

Location, Variety	PHI, Days	Sample[1]	Residue, mg/kg[2] Sulfan	Furan	HO-furan	Furan + HO-furan	Total carba-mates	Refs. & comments
		peel C NCOR	0.02 0.25	0.01 0.26	<0.01 0.12	 0.38	 0.63	
		Pulp C NCOR	ND ND	ND ND	ND 0.01	ND 0.01	ND 0.01	
	45	C NCOR COR	ND ND ND	ND <0.12 <0.15	ND <0.08 0.11	 <0.26	 <0.26	
		peel C NCOR	ND ND	ND 0.35	ND 0.24	ND 0.59	ND 0.59	
		pulp C NCOR	ND ND	ND ND	ND ND	ND ND	ND ND	
	105	C NCOR COR	ND ND ND	ND <0.05 <0.06	ND <0.04 <0.05	 <0.11	 <0.11	
		peel C NCOR	0.02 ND	ND 0.14	ND 0.11	ND 0.25	0.02 0.25	
		pulp C NCOR	ND ND	ND ND	ND ND	ND ND	ND ND	
	140	C NCOR COR	ND ND ND	ND <0.05 <0.06	ND <0.03 <0.04	 <0.10	 <0.10	
		peel C NCOR	ND ND	ND 0.14	ND 0.09	ND 0.23	ND 0.23	
		pulp C NCOR	ND ND	ND ND	ND ND	ND ND	ND ND	
Trial 3 Sueca Newhall oranges	0	Whole fruit * C NCOR COR	 <0.05 0.40 0.44	 ND 0.25 0.31	 ND 0.14 0.19			* Estimated from peel/pulp. ratio at harvest of 28/72
		peel C NCOR	0.07 0.56	0.01 0.35	0.01 0.19	0.02 0.54	0.09 1.1	
		pulp C NCOR	ND ND	ND ND	ND ND	ND ND	ND ND	
	45	C NCOR COR	ND ND ND	ND <0.11 <0.14	ND <0.12 <0.16			
		peel C NCOR	ND ND	ND 0.29	ND 0.32	ND 0.61	ND 0.61	
		pulp C NCOR	ND ND	ND ND	ND ND	ND ND	ND ND	

Location, Variety	PHI, Days	Sample[1]	Residue, mg/kg[2]					Refs. & comments
			Sulfan	Furan	HO-furan	Furan + HO-furan	Total carba-mates	
	105	C	ND	ND	ND	ND	ND	
		NCOR	ND	<0.03	<0.04	<0.07	<0.07	
		COR	ND	<0.04	0.05	<0.09	<0.09	
		peel C	ND	ND	ND	ND	ND	
		NCOR	ND	0.09	0.12	0.21	0.21	
		pulp C	ND	ND	ND	ND	ND	
		NCOR	ND	ND	ND	ND	ND	
	140	C	ND	ND	ND	ND	ND	
		NCOR	ND	0.02	<0.03	<0.05	<0.05	
		COR	ND	0.03	<0.04	<0.07	<0.07	
		peel C	ND	ND	ND	ND	ND	
		NCOR	ND	0.07	0.08	0.15	0.15	
		pulp C	ND	ND	ND	ND	ND	
		NCOR	ND	ND	ND	ND	ND	
Mean recovery, %, at 0.05 mg/kg		90	80	74				

[1]Duplicate field samples where two values are given. C = control, ND = <limit of detection of 0.01 mg/kg; LOD = 0.05 mg/kg. Values between 0.01 and 0.05 mg/kg are estimates. NCOR = not corrected for recoveries, COR = corrected for recoveries. Because only one PHI per trial represents GAP, underlined values were used to estimate maximum residue and STMR levels.

Animals

In a feeding study (Chen, 1995b) three groups of 3 Holstein dairy cows were given carbosulfan daily after the morning milking by bolus at rates equivalent to about 1, 3, or 10 ppm in the diet on a dry weight basis for 28 days. Another group of three served as controls and a fifth group of 4 cows was given the equivalent of 50 ppm in the diet for the same period. These levels were chosen to represent 1, 3, 10 and 50 times the expected dietary intakes from feed items.

Milk was collected in the evening and before dosing the next morning at regular intervals and composited, with sampling from 0-33 days. Cream and skimmed milk were separated from some samples to study the propensity for residues to accumulate in the fat. All animals from the 1 and 3 ppm dosing groups and two from the 10 and 50 ppm groups were slaughtered within 24 hours of the last dose for sampling of kidneys, liver, muscle and fat. The remaining animal in the 10 ppm group and one of the two remaining in the 50 ppm group were slaughtered after a 3-day recovery period. The last cow from the 50 ppm group was slaughtered after a 6-day recovery period. The samples were stored frozen and analysis of milk and tissues completed within 4 and 6 months respectively. The analytical method was Chen (1995a). Because significant residues were found only at the higher feeding levels, the results are shown only for the 10 and 50 ppm groups (Tables 23 and 24).

Table 23. Residues ug/kg (ppb)[1] carbosulfan and metabolites in the milk and tissues of three cows dosed with carbosulfan for 28 days at the equivalent of 10 ppm carbosulfan in the diet (Chen, 1995b).

Sample	Residues, µg/kg							
	carbo-sulfan	Carbo-furan	3-keto-carbo-furan	3-0H-carbo-furan	7-phenol	3-keto-7-phenol	3-hydroxy-7-phenol	DBA, range and mean
Milk, day0	ND	ND	ND	ND	ND	ND	ND	(16)-28(21)

Sample	Residues, µg/kg							
	carbo-sulfan	Carbo-furan	3-keto-carbo-furan	3-0H-carbo-furan	7-phenol	3-keto-7-phenol	3-hydroxy-7-phenol	DBA, range and mean
1	ND	ND	ND	ND	ND	ND	ND	32-45/37
2	ND	ND	ND	ND	ND	ND	ND	ND-(23)/(16)
4	ND	ND	ND	**ND-(7)**/(4)	ND	ND	ND	26-3[2]30
7	ND	ND	ND	ND	ND	ND	ND	ND-(10)/(7)
14	ND	ND	ND	ND	ND	ND	ND	(5)-(12)/(9)
21	ND	ND	ND	ND	ND	ND	ND	NA
27	ND	ND	ND	ND	ND	ND	ND	32-54/42
30 (one cow)	ND	ND	ND	ND	ND	ND	ND	(7)
Skimmed milk, day 21	ND	ND	ND	ND	ND	ND	ND	NA
Cream, day 21	ND	ND	ND	ND	ND	ND	ND	NA
Kidney	ND	ND	ND	ND	48,57/53	ND	ND,(12)/(9)	52-106/79
Liver	ND	ND	ND	ND	ND	ND	ND	(39)-(45)/(42)
Muscle	ND	ND	ND	ND	ND	ND	ND	(14)-70/(42)
Fat	ND	ND	ND	ND	ND	ND	ND	(14)-(16)/(15)

ND = Not detectable = <5 µg/kg in milk and <10 µg/kg in tissues for all compounds
NA = not analysed
[1]Where there were finite residues in one or more of the three cows, the range and average is given. In these cases ND was added as 1/2 ND for calculation of the average, presented as range/average. Derivative values adjusted for molecular weight for parent molecule value. A 0.581 m.w. correction factor is required to convert from carbosulfan to carbofuran. The limits of determination for all compounds were 25 µg/kg in milk and 50 µg/kg in cream and tissues.. Data from Chen, 1995 Tables 2, 3 and 4.
[2]Values in parentheses are estimates where results are between the limits of detection and determination

Table 24. Residues of carbosulfan and metabolites in milk and tissues from feeding four cows carbosulfan for 28 days at 50 µg/kg carbosulfan in the diet (Chen, 1995).

Sample	Residues, :g/kg (range/mean)[1]								
	Carbo-sulfan	Carbo-furan	3-keto-carbo-furan	3-0H-carbo-furan	Total carbam-ates[2]	7-phenol	3-keto-7-phenol	3-hydroxy-7-phenol	Total Phenols[2]	DBA range/ average
Milk test day 0	ND/ND	ND	ND	ND	20	ND	ND	ND	(15)	ND-(9)/ND
1	ND/ND	ND	ND	**(12)-(22)**/(16)	31	ND-(6)/(4)	(15)-38/25	(10)-(15)/(13)	(42)	(24)-36/(29)
2	ND/ND	ND	ND	**ND-30**/(17)	32	ND-(5)/(3)	(15)-4[2] 27	(13)-26/(19)	(49)	34-79/57
4	ND/ND	**ND-(6)**/(3)	ND	**(12)-(21)**/(17)	30	ND-(5)/(3)	(16)-26/(19)	(8)-(16)/(11)	(33)	ND-(20)/(11)
7	ND-(5)/ND	**ND-(8)**/(4)	ND	**(15)-(24)**/(19)	33	ND	(16)-25/(19)	(9)-(13)/(11)	(35)	(24)-**119**/77
14	ND-(12)/(7)	ND	**ND-(11)**/(5)	**(12)-(20)**/(15)	32	ND-(8)/(4)	(11)-4[2] (23)	(8)-(13)/(11)	(38)	60-105/73
21	ND-(11)/(6)	ND	ND	**(7)-(17)**/(14)	30	ND-(7)/(4)	(12)-27/(18)	(7)-(13)/(11)	(33)	NA/NA

Sample	Residues, :g/kg (range/mean)[1]									
	Carbo-sulfan	Carbo-furan	3-keto-carbo-furan	3-0H-carbo-furan	Total carbam-ates[2]	7-phenol	3-keto-7-phenol	3-hydroxy-7-phenol	Total Phenols[2]	DBA range/ average
27	ND-(5)/ ND	ND	ND	**(10)-(13)**/ (11)	26	ND-(7)/ (4)	(14)-3[2] (20)	(7)-(11)/ (9)	(33)	38-7[1] 60
30 (2 cows)	ND/ND	ND	ND	ND	20	ND	ND	ND	(15)	(9)-25/ (17)
33 (1 cow)	NA/ND	ND	ND	ND	20	ND	ND	ND	(15)	(15)/NA
Skimmed milk, day 21	ND/ND	ND	ND	**ND-(20)**/ (10)	25	ND-(8)/ (6)	(12)-(39)/ (23)	(7)-(14)/ (12)	(41)	NA
Cream, day 21	(11)-(45)/ (28)	**ND-(16)/ (8)**	ND	ND	56	ND-(20)/ (11)	(10),(17)/ (14)	ND	(35)	NA
Kidney day 28 day 31[4] day34[5]	ND/ND	ND	ND	**90 133**/ 112 ND ND	142	**315 400**/ 358 ND (12)	58,74/ 66 ND ND[7]	**152 173**/ 163 ND ND	587	**290 890**/ 590 (26)/ (22)
Liver	ND/ND	ND	**ND, (23)** (14) NDND	**47 60**/ 54 19)/ ND	88	ND	ND	(29),(34) /(32) (14)/ND	52	149 294/ 222 (36)/ (24)
Muscle	ND/ND	ND	ND	**20 (30)**/ (25) ND ND	55	ND	ND	ND,(12)/ (9) ND ND	29	(38) 58/ (48) (23)/ (19)
Fat	(11), 76/ (44) (33)/ (10)	ND	ND	ND	74	ND,(14)/ 10 ND ND	ND	ND,(11)/ (8)	28	(25) (47)/ (36) (15)/ND

[1]ND = Not detectable = <5 µg/kg (all compounds) in milk and <10 µg/kg in other samples. Where there were finite residues in one or more of the three cows, the range and average is given. In these cases ND was added as 1/2 ND for calculation of the average, presented as range/average. Derivative values adjusted for molecular weight for parent molecule value. A 0.581 m.w. correction factor is required to convert from carbosulfan to carbofuran. The limits of determination for all compounds were 25 µg/kg in milk and 50 µg/kg in cream and tissues. Data from Chen, 1995, Tables 2, 3 and 4.
[2]Simple sum of mean values where ND = 5 for milk and skimmed milk, 10 for other samples without m.w. conversion.
[3]Parentheses indicate residue estimates between the limits of detection and determination.
[4]3-day recovery period
[5]6-day recovery period

FATE OF RESIDUES IN STORAGE AND PROCESSING

Storage

No information

Processing

Citrus fruit. Two processing studies were conducted. In one, Florida grapefruit were treated 4 times by a hydraulic sprayer with a 2.5 EC formulation at 1.1-4.2 kg ai/ha (total 10.5 kg ai/ha), and in the other Valencia oranges were treated 5 times at the same rates (total 11.5 kg ai/ha, 14025 l/ha). In both trials the fruit were harvested on the day of the last application and processed (Leppert, 1991).

Processing was in an FMC Corporation citrus processor according to the method of Kesterson and Braddock (1979). A summary of the processing method was provided. It includes a pre-wash rinse, a soap and water brush wash, water removal between foam rollers, juicing with an FMC modified Model 35 finisher and collection of unfinished juice in cold-wall cooling tanks from which finisher pulp is removed to leave "single" strength juice. The oil emulsion fraction from the rollers is screened and centrifuged for the collection of citrus oil. The peel, rag and seeds are chopped, soaked in 0.3% dehydrated lime slurry and passed through a reaction screw to remove press liquor which is boiled under vacuum and concentrated to produce citrus molasses. The solids are dried to about 8% moisture to yield dried pulp and meal.

The analytical method is described above (Leppert, 1981). Carbosulfan, carbofuran and 3-hydroxycarbofuran were determined. The results are shown in Table 25.

Table 25. Residues and processing factors for carbosulfan, carbofuran and 3-ydroxycarbofuran in whole fruit and processed fractions of oranges and grapefruit.

Sample	Residues, mg/kg (range/mean)[1]									
	Valencia oranges					Marsh grapefruit				
	Carbo-sulfan	Carbo-furan	3-hydroxy-carbo-furan	Total carba-mate	Carbo-furan + 3-hydroxy-carbo-furan	Carbo-sulfan	Carbo-furan	3-hydroxycarbo-furan	Total carba-mate	Carbo-furan + 3-hydroxy-carbo-furan
Unwashed whole fruit	0.17	0.27	0.30	0.73	0.57	0.12	0.14	0.32	0.57	0.46
Washed whole fruit	0.08	0.34	0.40	0.82	0.74	(0.04)	(0.11)	0.30	0.45	0.41
Processing factor	0.47	1.31	1.3	1.1	1.3	0.33	0.79	0.94	0.79	0.89
Juice	ND	(0.01)[1]	ND	(0.01)	(0.01)	ND	(0.01)	ND	(0.01)	(0.01)
Processing factor	NF[2]	0.04	NF	0.01	0.02	NF	0.07	NF	0.02	0.02
Molasses	(0.02)	0.42	0.38	0.81	0.80	ND	0.24	0.94	1.18	1.18
Processing factor	0.12	1.6	1.3	1.1	1.4	NF	1.7	2.9	2.1	2.6
Dried pulp	0.14	0.31	1.67	2.11	1.98	0.10	0.15	1.28	1.53	1.43
Processing factor	0.82	1.2	5.6	2.9	3.5	0.83	1.1	4.0	2.7	3.1
Finisher pulp	ND	(0.01)	(0.02)	(0.03)	(0.03)	ND	ND	ND	ND	ND
Processing factor	NF	0.04	0.07	0.04	0.05	NF	NF	NF	NF	NF
Oil	1.22	3.94	(0.04)	5.20	3.98	2.42	1.58	(0.04)	4.04	1.62
Processing factor	7.2	14.6	0.13	7.1	7.0	20.2	11.3	0.13	7.1	3.5

NF = no processing factor could be calculated: ND = not detected
[1]Mean of duplicate results (whether duplicate samples or analyses is not indicated)
[2]Values in parentheses are estimates at levels below the limit of determination

Residues in the edible portion of food commodities

Nearly all of the residue in citrus fruit is in or on the peel (≤0.3% of the TRR was found in the pulp or juice). There is little likelihood of carbosulfan residues occurring in the edible portions of citrus fruits.

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

No information.

NATIONAL MAXIMUM RESIDUE LIMITS

The following national MRLs in The Netherlands were reported to the Meeting.

Commodity	MRL, mg/kg	
	carbosulfan	carbofuran + 3-hydroxycarbofuran
Carrots	0.1	0.3
Parsnips	0.1	0.3
Tea	0.1*	0.2*
Other foods	0.05*	--

* At or about the limit of determination

APPRAISAL

Carbosulfan, 2,3-dihydro-2,2-dimethylbenzofuran-7-yl (dibutylaminothio)methylcarbamate is a broad-spectrum carbamate pesticide used on a variety of crops, but mainly on citrus fruits, and this use is the focus of the present evaluation. Carbosulfan was first evaluated by the 1984 JMPR, which recommended a temporary ADI and a temporary MRL of 2 mg/kg for citrus fruits. The temporary ADI was converted to an ADI of 0-0.01 mg/kg bw by the 1986 JMPR.

Because information required by the 1984 and 1991 Meetings had not been provided, the 1993 JMPR recommended withdrawal of the proposed TMRLs for carbofuran and carbosulfan in citrus fruits. The 1993 Meeting was informed that additional studies were under way. Carbosulfan was subsequently scheduled for periodic review by the FAO Panel in 1997. New studies on citrus fruit have been reviewed by the Meeting, together with supporting data.

Carbofuran is a major metabolite of carbosulfan as well as being itself a pesticide. The present periodic review of carbosulfan includes estimates of maximum residue levels, an STMR and STMR-Ps for carbosulfan *per se* resulting from its use on citrus fruit. The concurrent review of carbofuran includes estimates to accommodate residues of carbofuran and 3-hydroxycarbofuran resulting from the use of carbosulfan on citrus fruit.

Metabolism studies on rats and goats were available. The distribution, excretion and fate of carbosulfan was investigated in rats by oral gavage administration of dibutylamine- or phenyl-labelled [^{14}C]carbosulfan at low (4 mg/kg) or high (30 mg/kg) dosing levels. About 66-88% was eliminated in the urine, 5-22% in the faeces and 10 to 17% as CO_2 from the dibutylamine (DBA) label but none from the phenyl label. Up to about 2% remained in the carcase. Eighty to 90% was excreted within 24 to 48 hours of dosing at the lower dose and within 72 hours or so at the higher dose. The main excreted compounds identified from the phenyl label in decreasing order were the 7-phenol, 3-keto-7-phenol, 3-hydroxycarbofuran, 3-hydroxy-7-phenol and carbosulfan, with minor residues of 5-hydroxycarbofuran, 3-ketocarbofuran, carbofuran, 3-ketocarbosulfan sulfone, 3-hydroxycarbosulfan and 3-ketocarbosulfan. From the DBA label DBA, hydroxy-DBA, CO_2 and carbosulfan were found in decreasing order. No major sex differences were observed. Higher residues of ^{14}C were found in tissues from the DBA label than from the phenyl. This was attributed to incorporation of the DBA moiety into natural stored fat by oxidation, *N*-dealkylation or deamination and further oxidation to fatty acids.

The metabolites are consistent with metabolic routes which include a series of hydrolyses, oxidations and conjugations. A main indicated route includes hydrolysis to the 7-phenol, oxidation to 3-hydroxy-7-phenol and further to 3-keto-7-phenol, and conjugation as sulfates or glucuronides. Another route involves oxidation to 3-hydroxycarbosulfan which may be hydrolysed to 3-hydroxycarbofuran or oxidized further to 3-ketocarbosulfan, which in turn may be oxidized again to 3-ketocarbosulfan sulfone or hydrolysed to 3-ketocarbofuran. The 3-hydroxycarbofuran or 3-ketocarbofuran may be hydrolysed to their phenols before conjugation. Hydrolysis also results in the release of DBA which may be oxidized at different carbons to hydroxydibutylamines. The authors also postulate the *N*-dealkylation/deamination and oxidation to fatty acids which may be incorporated in natural fats as described above, or result in the release of CO_2 by the citric acid cycle as indicated by the detection of radiolabelled CO_2.

Lactating goats were dosed with either phenyl- or DBA-labelled carbosulfan for 7 days at levels corresponding to approximately 25 ppm in the diet. Samples of urine, faeces, milk and tissues were analysed. As in rats, most of the ^{14}C was eliminated in the urine, approximately 82% of the phenyl label and 68% of the DBA label. About another 7% and 3% respectively were eliminated in the faeces. Approximately 0.2% of the dose (0.04-0.09 mg/kg carbosulfan equivalent) was found in the milk, 0.02% (0.06 mg/kg) in liver and 0.01% (0.2 mg/kg) in kidney from the phenyl label, but less than 0.01% (≤0.01 mg/kg) in muscle or fat. Because of these low levels, the identification of ^{14}C residues in muscle and fat from the phenyl label was not attempted.

The residues were higher from the DBA label: 2.3% (0.3-0.94 mg/kg) in the milk, 0.34% (1.13 mg/kg) in the liver, 0.04% (0.75 mg/kg) in the kidneys, 0.08% (0.18 mg/kg) in the muscle, and 0.15% (1.2 mg/kg) in the omental fat. The higher levels were attributed to incorporation into natural body constituents such as carbohydrates and proteins. The detection of radioactivity in fatty acids, amino acids, triglycerides and amines was consistent with that hypothesis.

A series of extractions, partitions, pH adjustments and acid or enzymatic hydrolyses were used to separate metabolites for comparison with authentic standards by HPLC, TLC, GC-MS, HPLC-MS and size-exclusion chromatography. From the phenyl label 98.6% of the TRR was extractable from milk, 37.3% from liver and 62% from kidney.

The major metabolites identified in milk, liver and kidney from the phenyl label were 3-hydroxycarbofuran and the 3-keto-7-phenol, 3-hydroxy-7-phenol and 7-phenol, accounting for approximately 94.4% of the TRR in milk (reaching a plateau after about 2 days), 32.7% in liver (37.1% for all identified residues) and 52% in kidney (59.6% for all identified residues). 3-hydroxycarbofuran (34.2% of the TRR) and the 3-keto-7-phenol (29.9% of the TRR) predominated in the milk, 3-hydroxycarbofuran in kidney (21.5% of the TRR) and the 3-hydroxy-7-phenol in liver (15.6% of the TRR). Minor identified residues included 5-hydroxycarbofuran, 3-ketocarbofuran, *N*-hydroxymethyl-carbofuran, carbofuran, carbosulfan, 3-ketocarbosulfan sulfone, 3-hydroxycarbosulfan and carbosulfan sulfone. None of these exceeded 4% of the TRR in milk, liver or kidney. Carbosulfan and carbofuran were detected only at very low levels in the milk and tissues (0.001 mg/kg).

Although only 37.1% of the phenyl label radioactivity was extractable from the liver with the initial solvent extraction, enzymatic and HCl hydrolysis allowed further characterization. Unidentified radioactivity was characterized as very polar (10.4% of the TRR), protein-associated (22.6% of the TRR) or unextractable (12.7% of the TRR).

In the kidney very polar unidentified metabolites accounted for 18.4% of the TRR, with another 17.4% characterized, but not identified.

Residue levels were much higher from the DBA label and this was attributed largely to incorporation into natural products as in rats. Residues in the fat and muscle from the phenyl label were too low for identification or characterization, but were high enough with the DBA-labelled samples (1.3 and 0.2 mg/kg carbosulfan equivalent in fat and muscle respectively). Approximately 80.1% of the DBA TRR (0.6 mg/kg carbosulfan equivalent) in milk and 90% in fat was organo-extractable, but only 45% in liver, slightly more than with the phenyl label. Approximately 70% of the TRR was extractable from kidney and 52% from lumbar muscle.

Residues in milk and tissues from the DBA label consisted mainly of aminobutanols, dibutylamine-related compounds, material incorporated into natural constituents (fatty acids, amino acids, carbohydrates, triglycerides etc.), amines (conjugated , unconjugated and bound) and polar water-soluble metabolites. In milk aminobutanols accounted for approximately 30% of the TRR and another 30% was found in natural constituents. 87% of the TRR in fat, 32% in muscle and 30% in liver was found in natural constituents. In liver another 21% was in the form of aminobutanols or dibutylamine and related compounds. In kidney 24% of the TRR was characterized as unconjugated amines, approximately 19% as polar water-soluble metabolites, and 14% as natural constituents.

The main metabolic routes in goats are similar to those in rats, starting with hydrolysis either directly to the 7-phenol or to carbofuran and dibutylamine. The 7-phenol is oxidized progressively to the 3-hydroxy-7-phenol and 3-keto-7-phenol and carbofuran to 3-hydroxy- or 5-hydroxycarbofuran. The 3-hydroxycarbofuran may be oxidized to 3-ketocarbofuran and each of these hydrolysed to the corresponding phenol. Dibutylamine may be oxidized to 4-(butylamino)butanol and further to the corresponding butanoic acid, or undergo a series of reactions to form butylamines and butanols. The degradation of DBA may also lead to incorporation into fatty acids, amino acids and carbohydrates and presumably through the citric acid cycle to CO_2, but CO_2 was not trapped.

The minor residues derived from the phenyl-labelled compound also indicate a subsidiary metabolic route in which the carbamate structure is retained with either direct oxidation to the sulfone or by 3-hydroxycarbosulfan and 3-ketocarbosulfan to 3-ketocarbosulfan sulfone.

Plant metabolism. Metabolism studies with both phenyl- and DBA-labelled carbosulfan were conducted in the field on navel oranges with spray application at a nominal rate of 0.5 g ai/l. Orange samples were taken at 0, 7, 15 and 30 days and leaves at 0 and 30 days. Oranges were rinsed and samples of peel rinse, peel, pulp and juice were analysed by HPLC, TLC, MS and LSC. The TRR in whole oranges amounted to 0.81 and 0.7 mg/kg carbosulfan equivalent from the phenyl and DBA labels on day 0 to 0.78 and 0.59 mg/kg on day 30.

Nearly all of the residue in the whole fruit was in or on the peel (99.9% of the phenyl ^{14}C, 99.6 of the DBA) on day 0 and these proportions remained essentially unchanged even after 30 days. Almost all of the residue was on the peel surface on day 0 (95.8% of the phenyl TRR, 93.9% of the DBA), but by 30 days more of the residue had penetrated into the peel (45.9% of the ^{14}C from the phenyl label and 41.5% from the DBA). No more than 0.3% of the TRR (<0.01 mg/kg carbosulfan equivalent) from both labels was in the pulp or juice over the 30-day period. More than 90% of the TRR in rinsed peel from both labels was extractable throughout the 30 days with the proportion of polar and conjugated material increasing with time, especially that from the DBA label where it reached 57% by day 30.

The peel rinses and extracts were examined to identify the residues. Residues from the phenyl label after 30 days as a proportion of the TRR were carbosulfan 40.1%, carbofuran 33.9%, carbosulfan sulfone 3.1%, 3-hydroxycarbofuran and 3-keto carbofuran 2% each and *N*-hydroxymethyl-carbofuran, dicarbofuran sulfide and the 7-phenol less than 2% each, making a total of 83.7% of the TRR. From the DBA label carbosulfan and DBA accounted for 31.2 and 58.2% of the TRR respectively. This is consistent with the primary metabolic cleavage of the two N-S bonds to form carbofuran and DBA. Some oxidation to carbosulfan sulfone occurs before cleavage of these bonds and a minor route resulted in the formation of dicarbofuran sulfide. The rest of the metabolism is effectively that of carbofuran. This includes direct oxidation to the 7-phenol or retention of the intact carbamate with oxidation at the *N*-methyl to form *N*-hydroxymethylcarbofuran or on the ring to form 3-hydroxycarbofuran which may be further oxidized to 3-ketocarbofuran.

The only metabolite found in oranges which was not also identified in goats was dicarbofuran sulfide at very low levels. The other notable difference between plants and animals is that only very low levels of intact carbamates were detected in animals (apart from 3-hydroxycarbofuran at exaggerated feeding levels), whereas they were the main residues in plants after 30 days.

Environmental fate. Although limited information was available on photolysis in soil and water, other environmental studies noted as being necessary in the 1995 JMPR report (Section 2.5.2) were not provided. The degradation of carbosulfan in dry soil and soil at 70% water capacity exposed to a sun lamp was investigated with phenyl- and DBA-labelled [^{14}C]carbosulfan. The spectral characteristics of the sun lamp were not reported, nor was the temperature.

The half-life of carbosulfan with both labels was less than 10 minutes in the dry soil. After 8 days the main residue from the phenyl label was carbofuran (54.5% of the TRR), with 3.5% of 3-hydroxycarbofuran, 2.6% of carbosulfan sulfone, and lesser amounts of phenols or oxidized carbamate metabolites of carbosulfan or carbofuran. The predominant residues from the DBA label after 10 minutes were dibutylamine (38.6% of the TRR), carbosulfan (11.4%), *N*-formyldibutylamine (6.4%) and *N*-acetyldibutylamine (1.1%). After 8 days the same compounds were detected, but at very low levels (the rest was unidentified). Degradation was substantially slower with the wet soil treated with DBA-labelled carbosulfan. After 48 hours carbosulfan was still 76.2% of the TRR.

The authors concluded that as the results from irradiated and control soils with both labels were so similar exposure to light had very little effect, suggesting that degradation resulted from soil contact rather than the effect of light. The Meeting could not draw such a firm conclusion from the data, although it is likely that the soil was the main contributor to the degradation.

Photolytic and hydrolytic degradation were also investigated in water buffered at pH 7 and distilled water with both DBA- and phenyl-labelled [^{14}C]carbosulfan (5 mg/l) and irradiation for up to 8 days with a sun lamp. Apart from specifying that the radiation was above 300 nm, the spectral characteristics of the sun lamp were not reported nor was the kept. The half-life was about 1.4 days in buffered water and 4-8 days in distilled water. Degradation was much more rapid in irradiated samples than in controls, although the identified products were the same. Degradation was mainly to carbofuran and dibutylamine. Other lesser products from the phenyl label were carbosulfan sulfone, the 7-phenol and the 3-keto-7-phenol. From the DBA label the main product was dibutylamine, with lesser amounts of *N*-formyldibutylamine and *N*-acetyldibutylamine.

No other studies on environmental fate were submitted to the Meeting.

Methods of analysis. A number of analytical procedures are available for the determination of carbosulfan, its carbamate and phenolic metabolites and dibutylamine in citrus and animal products. Recent methods used in some of the field trials with citrus fruit and animals are based on the extraction of carbosulfan with dichloromethane (from citrus) or acetone (from animals products) and clean-up on solid-phase extraction (SPE) cartridges before analysis. Carbamate and phenolic metabolites are hydrolysed with HCl before SPE column extraction and dibutylamine is extracted with methanol/water. Some procedures include liquid-liquid partitions.

The HPLC configuration for the determination of carbosulfan includes two post-column reactors, one with H_2SO_4 to hydrolyse carbosulfan to carbofuran and the other with *o*-phthalaldehyde + *N*,*N*-dimethyl-2-mercaptoethylamine to form a chromophore for fluorescence detection. The configuration is the same for carbamate metabolites, except that only the second reactor is used. Phenolic fractions are derivatized with pentafluorobenzyl bromide (PFBBr), and 3-hydroxy-7-

phenols also by ethylation, before analysis. Dibutylyamine fractions are derivatized with dansyl chloride for analysis. Both the phenolic and DBA derivatives are analysed by GC-MS with single ion monitoring.

In citrus a limit of determination of 0.05 mg/kg for all analytes would appear to be supported for this group of methods by adequate recoveries and sample chromatograms, but citrus controls consistently had apparent DBA levels up to 0.02 mg/kg. For this reason 0.1 mg/kg may be a more realistic limit of determination for DBA. For animal products limits of determination of 0.025 mg/kg in milk and 0.05 mg/kg in tissues also appear to be supported for all compounds on the basis of adequate recoveries and sample chromatograms, but DBA was again reported near the limit of determination in some milk samples (0.005-0.037 mg/kg). The methods were independently validated.

The methods used in some other citrus trials involved hexane/propanol extraction of carbosulfan and carbofuran, and HCl reflux extraction of 3-hydroxycarbofuran after ethoxylation, followed by liquid/liquid partition, Florisil column clean-up and GLC with NP detection. The reported limit of determination was 0.01 mg/kg and the methods were validated at that level. However sample chromatograms and corroborating information from multi-residue methods indicated that 0.05 mg/kg would appear to be a more practical limit of determination; it was also recommended as the reporting level.

Older methods used in citrus processing studies were similar to that just described, including GLC with NP detection, but with different clean-up columns. A limit of determination of 0.05 mg/kg is again reasonable, except perhaps for citrus oil where 0.1 mg/kg might be more realistic. Published multi-residue methods were not adequate for carbosulfan, mainly owing to low detector sensitivity.

Stability of residues in stored analytical samples. In a 1980 storage stability study no significant losses of carbosulfan were observed when orange and alfalfa samples fortified with carbosulfan were stored for one year at -18°C. Carbofuran was the predominant metabolite. However, in a pH 4.8 silt loam soil in this same study carbosulfan was almost completely degraded after only three hours at -18°C. This was attributed to the acidity, although carbosulfan was stable in the orange samples, also likely to be acidic. It was more stable in a pH 6 silty clay loam and a pH 6.8 sandy loam at -18°C, with half-lives of about 220 and 144 days respectively.

An interim report described studies of the stability of carbosulfan, its carbamate and phenolic metabolites and DBA in laboratory-fortified oranges and processed orange products stored for up to a year at -18°C. Samples of whole oranges, dried pulp, juice, molasses and oil were fortified at 0.25 mg/kg and most samples were taken for analysis on day 0 and after approximately 3, 6, and 12 months. On day 0 no residues were detected in the juice, and residues in molasses and oil were only 0.08 and 0.12 mg/kg respectively. In these cases carbofuran was the main product of carbosulfan degradation as demonstrated by mass balance investigations. Later samples of juice, molasses and oil were therefore analysed for carbosulfan. Orange oil was analysed for carbosulfan metabolites only on day 0 and after 12 months. There were no appreciable losses of the other carbamate or phenolic residues in any of the samples. It was reported that results of analyses for DBA after 18 and 24 months will be available at an unspecified future time.

The stability and mass balance of residues in bovine milk and tissues fortified at 0.25 mg/kg with carbosulfan and DBA were investigated after storage at -18EC for intervals up to 8 months. Carbosulfan was shown to be degraded rapidly in milk, muscle and liver with losses of 16 and 4% from milk and liver respectively in the first month and of 84% from milk, 100% from muscle and 80% from liver after 8 months. Losses of DBA from milk and liver were 8 and 4% respectively after 6 months, but DBA residues in muscle showed an apparent increase of 52% after 6 months. Overall the analyses for DBA were erratic over the test period. Mass balance studies showed that carbosulfan + carbofuran accounted for 68% of the fortification level in milk after 3 months and 88 and 44% in muscle and liver respectively after 6 months. After 8 months carbofuran and the 7-phenol together accounted for over half of the fortification level in milk and liver and 148% in muscle.

These results confirm the instability of carbosulfan *per se* in animal products even under frozen storage conditions, and add confidence to the prediction that there is little likelihood of finding it in animal products as a result of using carbosulfan on citrus. The results also confirm that degradation is likely to be mainly to carbofuran and its metabolites. The trials did not include analyses for 3-ketocarbofuran, 3-hydroxycarbofuran, or other minor carbamate metabolites. On the basis of the results of the cow feeding study it is likely that 3-hydroxycarbofuran especially may constitute a significant proportion of the residue unaccounted for in these studies.

<u>Citrus residue trials</u>. 30 supervised trials were conducted in 1993-4 in Brazil, Mexico and Spain using the analytical methods described above. The six 1993 Brazilian trials on Valencia and Pera Coroa oranges with a CE formulation (*c*.1-1.7 g ai/tree) were typical. Six trees/trial were treated, and 4 oranges/tree or 24 oranges/trial sample were taken. Duplicate samples were analysed separately in each trial and both results are tabulated. All trials were according to GAP (a maximum of 2 foliar applications at 0.9-1.7 g ai/tree, the first after full bloom and the 2nd approximately 50 days later). Sprays were to run-off and the 7-day GAP PHI was observed. Sampling, transport and storage were adequate to provide confidence in sample integrity. Analytical results were not corrected for recoveries. At the 7-day GAP PHI the maximum residues were carbosulfan <0.01-0.03 mg/kg, carbofuran 0.02-0.06 mg/kg, and 3-hydroxycarbofuran <0.01-0.03 mg/kg. The 3-ketocarbofuran metabolite occurred in only one trial, at 0.02 mg/kg. Total phenols were up to 0.02 mg/kg and dibutylamine up to 0.15 mg/kg.

A 7-day GAP PHI was also observed in the 7 Mexican trials on Valencia and other oranges with an LE formulation (250 g ai/ha). Residues from application according to GAP, corrected for recoveries, were <0.01-0.08 mg/kg carbosulfan, 0.08-0.26 mg/kg carbofuran, <0.01-0.14 mg/kg 3-hydroxycarbofuran, <0.01-0.04 mg/kg 3-ketocarbofuran, and <0.01-0.14 mg/kg 3-hydroxycarbofuran. Residues of total phenols (uncorrected) were 0.01-0.12 mg/kg and of DBA up to 0.14 mg/kg.

In four 1994 Spanish trials according to GAP with an LE formulation (*c*.3 g ai/tree) both mature (112-day PHI) and immature (28-day PHI) Valencia oranges were analysed. No residues (<0.01 mg/kg) of carbosulfan were detected at either PHI. In the mature oranges carbofuran residues (corrected for recovery) were <0.01-0.36 mg/kg, 3-ketocarbofuran <0.01-0.05 mg/kg, and 3-hydroxycarbofuran 0.02-0.14 mg/kg. The residues of total phenols (uncorrected) were up to 0.25 mg/kg and of DBA up to 0.14 mg/kg. Not unexpectedly, residues (except of 3-ketocarbofuran) were substantially higher in the immature oranges with a corrected maximum of 0.82 mg/kg carbofuran, <0.01 mg/kg 3-ketocarbofuran, and 0.39 mg/kg 3-hydroxycarbofuran. Maximum (uncorrected) residues of total phenols were 0.63 mg/kg and of DBA 0.29 mg/kg.

Additional Spanish trials according to GAP were conducted in 1993 and 1994 with high-volume applications of an EC formulation (937.5 g ai/ha, 3000 l/ha). Sampling was not only at the harvest GAP PHI (84-147 days), but also at days 0, 30, 45, 60...147. Analyses were only for carbosulfan, carbofuran, and 3-hydroxycarbofuran. Because these compounds are those that the Meeting recommended for inclusion in the definitions of the residues arising from the use of carbosulfan and carbofuran (see below), the results can be used to estimate maximum residue levels and STMRs. The JMPR had previously recommended that all field trials should also include analyses for 3-ketocarbofuran. The Meeting upheld that recommendation with respect to future submissions of data on commodities other than citrus but concluded that, because a number of trials had demonstrated that the compound occurs at relatively low levels in citrus, the data on 3-ketocarbofuran were adequate for citrus fruit.

No residues of carbosulfan were detected at these extended PHIs (84-147 days), but residues of carbosulfan at day 0 (corrected for recoveries) were as high as 3.3 mg/kg. It was seldom detectable after 30 days and even then only in the peel. Carbofuran and especially 3-hydroxycarbofuran were present in some cases after 30 days, and even at harvest after the lengthy PHIs. Generally 3-hydroxycarbofuran was the higher of the two at this stage.

In the six 1993 trials residues at harvest (110-147 days) were <0.01-0.04 mg/kg carbofuran (mostly <0.01) and 0.05-0.13 mg/kg 3-hydroxycarbofuran. Peel and pulp samples were also analysed at 45 days in two of the trials: carbosulfan was not detected in the peel or pulp (<0.01 mg/kg) and carbofuran residues were about 0.27 or 0.37 mg/kg in the peel and <0.01 mg/kg in the pulp, giving 0.02-0.17 mg/kg in whole oranges. 3-hydroxycarbofuran was at a similar level in the peel and 0.01 mg/kg in the pulp.

In the seven 1994 studies (PHIs 84-140 days) residues in whole oranges were calculated from those in the peel and pulp and the measured peel/pulp weight ratios (24/76, 27/73, 28/72 or 30/70 at harvest, depending on the type of orange). The calculated residues were <0.01 mg/kg carbosulfan, <0.01-0.06 mg/kg carbofuran and 0.04-0.13 mg/kg 3-hydroxycarbofuran. At day 0 the maximum residues in the peel and pulp (uncorrected for recovery) were 0.9 and <0.01 mg/kg carbosulfan, 0.63 and <0.01 mg/kg carbofuran and 0.64 and 0.05 mg/kg 3-hydroxycarbofuran.

The Meeting concluded that MRLs for citrus fruits should be established both for carbosulfan defined as carbosulfan and for carbofuran defined as the sum of carbofuran and 3-hydroxycarbofuran, expressed as carbofuran. The Meeting examined the distribution of data from trials complying with GAP according to these definitions in order to estimate MRLs and STMRs, and observed (not surprisingly) the absence of detectable carbosulfan residues in the Spanish trials with PHIs of 84-147 days compared with the measurable but low levels after 7 days in the Mexican and Brazilian trials. There is much less variation in the sum of carbofuran and 3-hydroxycarbofuran residues however, even with the wide divergence of national PHIs.

Duplicate samples were analysed in most of the trials and the results recorded separately. In order to avoid averaging problems in cases where one of two duplicate results was below the level of detection and to avoid the possibility of over-estimating the median residue, the Meeting decided to treat all the results separately for the estimation of MRLs and STMRs, and included estimated levels for residues between the limits of detection and determination.

The Meeting had two options for the estimation of STMRs for carbosulfan and carbofuran in oranges. One was to use the residues found in the pulp in four 1994 Spanish trials in which carbosulfan was undetected (<0.01 mg/kg) in all four trials and carbofuran + 3-hydroxycarbofuran was undetected in three and at a level of 0.02 mg/kg in the fourth. Since residues were detectable and estimated to be 0.01 mg/kg in each of two 45-day pulp samples in Spanish trials, the residue of 0.02 mg/kg found in the one trial according to GAP is not likely to be aberrant.

The second option was to estimate STMRs for residues of carbosulfan and carbofuran + 3-hydroxycarbofuran in whole oranges from the much larger database of 30 trials. Because of the greater uncertainty associated with the database of only four trials, the Meeting took the second option.

The low or undetectable residues found in the limited number of orange pulp samples and the results of the orange metabolism study described above which showed ≤0.2% and ≤0.3% of the TRR in the pulp and juice respectively give added assurance that residues of carbosulfan and carbofuran + 3-hydroxycarbofuran in the edible portions of oranges, if present, are likely to be very low.

The residues of carbosulfan from all the treatments according to GAP (counting duplicate samples separately) were 0.08, 0.04, 0.03 (3), 0.02 (7), 0.01 (2), and <<u>0.01</u> mg/kg (39) in a total of 53 samples. If the Spanish trials are excluded, the residues of carbosulfan were 0.08, 0.04, 0.03 (3), 0.02 (7), <u>0.01</u> (2), and <0.01 mg/kg (12): 26 samples. From these results, the Meeting estimated a maximum residue level of 0.1 mg/kg and an STMR of 0.01 mg/kg for carbosulfan in oranges. The STMR is at the limit of detection.

The residues of the simple sum of carbofuran + 3-hydroxycarbofuran were 0.5, 0.4 0.39, 0.33, 0.26, 0.22 (2), 0.19, 0.17 (2), 0.15, 0.14 (2), 0.13 (2), 0.12 (4), 0.11 (6), 0.10, <0.10, 0.09 (3), 0.08 (3), 0.07 (4), <0.07, 0.06 (4), 0.05 (4), 0.04, <0.04, 0.03 (3), and 0.02 (2) mg/kg (53 samples).

On the basis of this distribution, recognizing that the residues would be only very slightly lower if an adjustment were made for the molecular weight of 3-hydroxycarbofuran (about 7% higher than carbofuran), the Meeting estimated a maximum residue level of 0.5 mg/kg and an STMR of 0.1 mg/kg for the sum of carbofuran and 3-hydroxycarbofuran, expressed as carbofuran, in oranges. The Meeting concluded that the STMR for the total carbamate residues would be essentially the same as for carbofuran + 3-hydroxycarbofuran because of the low proportion of carbosulfan in the total carbamate residue.

The Meeting also received information on GAP (without labels) from Thailand for carbosulfan uses on rice, asparagus and watermelons together with what appeared to be an incomplete report of field trials. Although fairly detailed information was provided on the conduct of the trials, no analytical results were included. The information on GAP for these crops was recorded in the evaluation in case the results of the trials become available in the future and provided the GAP is confirmed by approved labels. However, the two trials apparently completed on each of these crops would not be sufficient to estimate maximum residue levels.

Summary information on GAP for German uses on rape, maize and hops was also received but no labels or residue data were provided. Official information on GAP for several commodities was also received from the UK, but again without data on residues. The information on GAP should be re-submitted, together with relevant labels, with any future reports of residue trials.

Feeding studies. Holstein dairy cows were dosed at levels equivalent to 0, 1, 3, 10 and 50 ppm carbosulfan in the diet for 28 days. Milk, kidney, liver, muscle and fat were analysed for carbosulfan, carbofuran, 3-hydroxycarbofuran, 3-ketocarbofuran, the 7-phenol, 3-keto-7-phenol and 3-hydroxy-7-phenol metabolites and dibutylamine. Selected cows were held for an additional 3 or 6 days for recovery studies. The residues in the milk and tissues were generally in the decreasing order 3-hydroxycarbofuran, 3-ketocarbofuran, carbofuran and carbosulfan, although in some samples of milk carbosulfan residues were of the same order as those of carbofuran.

No carbamate residues, except one of 7 µg/kg 3-hydroxycarbofuran in the milk of one of three cows at the 10 ppm feeding level after four days, were detected in any samples at the 1, 3 or 10 ppm feeding levels. Phenols were detected at the 10 ppm level, but only in kidney (max. 57 µg/kg 7-phenol and 12 µg/kg 3-hydroxy-7-phenol). Dibutylamine was found at the 10 ppm feeding level up to 54 µg/kg in milk and in all the tissues (highest in kidney at 106 µg/kg). Carbamates were found in the milk and tissues from the 50 ppm feeding level. In summary, the maximum and mean residues at the 50 ppm feeding level were as shown below.

	Maximum/mean residue, µg/kg						
Compound	Milk	Skimmed milk	Cream	Kidney	Liver	Muscle	Fat
carbosulfan	12/7	ND	45/28	ND	ND	ND	76/44
carbofuran	8/4	ND	16/8	ND	ND	ND	ND
3-hydroxycarbofuran	30/19	20/10	ND	133/112	60/54	30/25	ND
3-ketocarbofuran	11/5	ND	ND	ND	23/14	ND	ND
7-phenol	8/4	8/6	20/11	400/358	ND	ND	14/10
3-keto-7-phenol	42/27	39/23	17/14	74/66	ND	ND	ND
3-hydroxy-7-phenol	26/19	14/12	ND	173/163	34/32	12/9	11/8
dibutylamine	119/77	---	---	890/590	294/222	58/48	47/36

where ND = 5 µg/kg in milk and 10 µg/kg in the other substrates, with limits of determination of 25 µg/kg and 50 µg/kg respectively.

At the 50 ppm feeding level carbosulfan was found in milk up to 12 µg/kg, cream up to 45 µg/kg, and fat up to 76 µg/kg, but not in kidney, liver or muscle. At this feeding level carbofuran was found only in milk (up to 8 µg/kg in one cow) and in cream up to 16 µg/kg in a different cow after a 3-day withdrawal period. 3-ketocarbofuran was detected only at the 50 ppm feeding level and then only in the milk and liver at maximum levels (in the same cow) of 11 and 23 µg/kg respectively. No residues were detected in either milk or liver after 3- or 6-day withdrawal periods.

Most of the carbamate residue at the 50 ppm level consisted of 3-hydroxycarbofuran, except in cream and fat. In milk the mean and maximum residues of 3-hydroxycarbofuran were 19 µg/kg after 7 days and 30 µg/kg after 2 days respectively, gradually decreased to 11 µg/kg after 27 days, and were undetectable after a 3-day withdrawal period. There was some reduction of carbosulfan and dibutylamine in fat during the 3- and 6-day recovery periods and no 7-phenol was detected in fat during these periods. The total carbamate residues in milk were fairly constant at approximately 30 µg/kg after the first day of sampling through the dosing period. The 3-hydroxycarbofuran metabolite was up to 133 µg/kg in kidney, ≤60 µg/kg in liver and ≤30 µg/kg in muscle. It was not detected in fat.

Both carbosulfan and carbofuran were found in cream (at mean levels of 28 and 8 µgkg respectively) but not in skimmed milk (<5 µg/kg). 3-ketocarbofuran was not found in either, and 3-hydroxycarbofuran in skimmed milk (mean level 10 µg/kg) but not in cream, not unexpectedly in view of the polarity afforded by the hydroxyl group.

In the milk, the residues of total phenols were fairly constant over the test period after the first day, with the 3-keto-7-phenol generally predominating. The highest average residues were 3-keto-7-phenol 27 µg/kg, 3-hydroxy-7-phenol 19 µg/kg and 7-phenol 4 µg/kg. These were all undetectable (<5 µg/kg) after 3 or 6 days withdrawal. The highest phenolic residues in the tissues were in the kidney with mean levels of the 7-phenol of 358 µg/kg, the 3-hydroxy-7-phenol of 163 µg/kg and the 3-keto-7-phenol of 66 µg/kg. In the liver and muscle only the 3-hydroxy-7-phenol was detected, at mean levels of 32 and 9 µg/kg respectively, and in fat only the 7-phenol (10 µg/kg) and 3-hydroxy-7-phenol (8 µg/kg).

Apparent dibutylamine was reported in most controls at maximum levels of about 50 µg/kg in both milk and tissue samples, and the residues in treated groups did not correlate well with the dose rates. Its apparent natural occurrence made reliable estimates of the DBA derived from carbosulfan difficult in all samples from animals at the 1 to 10 ppm feeding levels, and in muscle and fat at the 50 ppm level. The mean residues of 590 µg/kg dibutylamine in the kidneys and 222 µg/kg in the livers of the 50 ppm group clearly arose mainly from the treatment however.

Since the highest carbamate residues likely to result from the use of carbosulfan in an animal feed item would be about 2 mg/kg from dry citrus pulp with an STMR of 0.29 mg/kg and this is likely to constitute no more than 20-25% of a cattle diet, and since there were no significant residues at the 10 ppm feeding level and relatively low levels even at 50 ppm, the Meeting concluded that no MRL was required for carbosulfan or its metabolites in milk or tissues to accommodate the use of carbosulfan on citrus. Any residues that might occur would be covered by the maximum residue levels estimated for animal products to accommodate the use of carbofuran (see Section 4.5).

Processing

The Meeting examined reports of two processing studies, one on grapefruit and one on oranges, although data on supervised trials were available only for oranges.

Washing the fruit reduced residues of carbosulfan in grapefruit and oranges by about 67% and 53% respectively. Both carbofuran and total carbamates were reduced by about 21% in grapefruit, but there was no reduction in oranges. The loss of carbosulfan from oranges appears to be offset by increases in carbofuran and 3-hydroxycarbofuran. This situation is analogous to the finding of low or undetectable residues of carbosulfan in harvest samples of oranges although total carbamate residues remain relatively constant over long periods. Because the oranges were processed on the day of the last application instead of after the normal pre-harvest interval, the Meeting decided to consider the total carbamate levels as a measure of the residue in evaluating the processing study. To omit carbosulfan, which would have been largely converted to carbofuran and 3-hydroxycarbofuran at harvest, would underestimate the residues.

No residues (<0.01 mg/kg) of carbosulfan were found in orange juice, so no processing factor could be calculated. Because the residue of 0.17 mg/kg carbosulfan in the whole unwashed oranges is similar to or slightly above the maximum residue found in field trials according to GAP, there would be no real expectation of finding carbosulfan in orange juice. An STMR-P of 0 for carbosulfan *per se* in orange juice would be reasonable. The residue of 0.73 mg/kg total carbamates is also slightly higher than the residues found from GAP applications in field trials. There was no concentration of any carbosulfan metabolite in the juice, although carbofuran was detected at low levels. A processing factor of about 0.01 for total carbamates applied to an STMR of 0.1 mg/kg for the sum of carbofuran and 3-hydroxycarbofuran would give an STMR-P of 0.001 mg/kg for carbofuran + 3-hydroxycarbofuran in orange juice. Although no MRL for either carbosulfan or the sum of carbofuran and 3-hydroxycarbofuran would appear to be needed since the residues would be expected to be below the limit of detection of 0.01 mg/kg, an MRL of 0.05 mg/kg, at the limit of determination, would be reasonable if one is needed.

The processing factor for carbosulfan on processing unwashed orange fruit to molasses was approximately 0.12. A worst-case STMR-P for carbosulfan in orange molasses would be the STMR for oranges, 0.01 mg/kg, x 0.12 = 0.0012 mg/kg. The processing factor for total carbamate residues was 1.1, and this multiplied by the STMR of 0.1 mg/kg for the sum of carbofuran and 3-hydroxycarbofuran in unwashed whole fruit gives an STMR-P of 0.11 mg/kg. If an MRL for carbosulfan in molasses is needed, a value of 0.05 mg/kg, at the limit of determination, would be appropriate (the maximum expected residue being 0.012 mg/kg). Because there is no significant concentration, the residues of carbofuran + 3-hydroxycarbofuran would not be expected to exceed the maximum residue level of 0.5 mg/kg estimated for whole fruit.

The processing factor for carbosulfan from unwashed oranges to dry pulp was 0.82, so the STMR-P = the STMR for oranges, 0.01 mg/kg, x 0.82 = 0.0082 mg/kg. The STMR-P for the sum of carbofuran and 3-hydroxycarbofuran = STMR 0.1 x processing factor 2.9 = 0.29 mg/kg. On the basis of the 0.82 processing factor and the recommended MRL of 0.1 mg/kg for carbosulfan in whole oranges, 0.1 mg/kg would also be sufficient as an MRL for carbosulfan in dry citrus pulp. The processing factor of 2.9 and the recommended MRL for carbofuran + 3-hydroxy-carbofuran in oranges of 0.5 mg/kg, indicate that 2 mg/kg should be the MRL for the sum of carbofuran and 3-hydroxycarbofuran in dry citrus pulp.

<u>Orange oil</u>. The processing factor for carbosulfan was 7.2 and that for the sum of carbofuran and 3-hydroxycarbofuran was 7.1. Since the STMR levels in oranges were 0.01 and 0.1 mg/kg respectively the corresponding STMR-Ps for orange oil would be 0.072 and 0.71 mg/kg. The same processing factors applied to the recommended MRLs of 0.1 mg/kg for carbosulfan and 0.5 mg/kg for carbofuran in oranges would lead to recommended MRLs of 1 and 5 mg/kg respectively for the oil.

The results of these estimates are shown below.

Sample	Carbosulfan				Carbofuran + 3-hydroxycarbofuran			
	Processing factor	Max. res. level[1], mg/kg	Orange STMR, mg/kg	Processed fraction STMR-P, mg/kg	Processing factor	Max. res. level[1], mg/kg	Orange STMR, mg/kg	Processed fraction STMR-P, mg/kg
Whole	--	0.1	0.01	--	--	0.5	0.1	--

Sample	Carbosulfan				Carbofuran + 3-hydroxycarbofuran			
	Processing factor	Max. res. level[1], mg/kg	Orange STMR, mg/kg	Processed fraction STMR-P, mg/kg	Processing factor	Max. res. level[1], mg/kg	Orange STMR, mg/kg	Processed fraction STMR-P, mg/kg
oranges								
Juice	NF[2]	<0.01 (0.05*)	0.01	0.0	0.01	0.005 (0.05*)	0.1	0.001
Molasses	0.12	0.012 (0.05*)	0.01	0.0012	1.1	0.55 (0.5)	0.1	0.11
Dry Pulp	0.82	0.08 (0.1)	0.01	0.0082	2.9	1.5 (2.0)	0.1	0.29
Oil	7.2	0.72 (1.0)	0.01	0.072	7.1	3.5 (5.0)	0.1	0.71

[1] The first number is the estimated maximum residue based on the processing factor and the maximum residue level for whole oranges. The numbers in parentheses are the recommended MRLs. If the estimated maximum residue level is less than the 0.05 mg/kg limit of determination, the limit of determination is recommended as the MRL.

[2] No factor could be estimated because no residues were detected in the juice

RECOMMENDATIONS

The Meeting estimated the maximum residues levels and STMR levels shown below. The maximum residue levels are recommended for use as MRLs.

Definition of the residue for compliance with MRLs and estimation of dietary intake: carbosulfan

Commodity		Recommended MRL, mg/kg		STMR, mg/kg
CCN	Name	New	Previous	
DM 001	Citrus molasses			0.0012 P[1]
AB 0001	Citrus pulp, dry	0.1	-	0.0082 P
JF 0004	Orange juice		-	0 P
FC 0004	Oranges, sweet, sour	0.1		0.01

[1]STMR-P

FURTHER WORK OR INFORMATION

<u>Desirable</u>

1. Information on residues of carbosulfan in food in commerce or at consumption

2. The final report on the studies of the stability of carbosulfan and its metabolites in oranges and their processed products during frozen storage (final version of Interim Report P-3154)

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CHLOROTHALONIL

EXPLANATION

Chlorothalonil (2,4,5,6-tetrachloroisophthalonitrile) was first evaluated for residues in 1974 and has been reviewed several times since, most recently as a periodic review in 1993. The 1993 JMPR recommended the withdrawal of a number of MRLs for commodities for which residue data or information on GAP were not available, and required additional residue data from supervised trials on different types of melons, residue data on grapes treated according to GAP in Australia, and animal transfer studies.

At the 27th (1995) Session of the CCPR the manufacturers indicated that they would provide information on GAP and residue data to the 1997 JMPR for some crops. The representative of the EU was invited to submit residue trials data and information on GAP for the use of chlorothalonil on tomatoes to the JMPR, to support extrapolation and to establish an MRL for peppers (ALINORM 95/24A, paras 107-111). The 1996 CCPR was informed that additional data would be provided for peaches, and decided to keep the MRL for peach at Step 7B.

Extensive supporting information, as well as updated information on GAP and residue trials data, was supplied by the manufacturer (Bliss, 1997). The available studies were on farm animal metabolism, farm animal transfer and the stability during frozen storage of commodities of animal origin. Residue trials data were available for citrus fruits, peaches, grapes, blackberries, currants, bananas, broccoli, peppers, sweet corn and beans (fresh and dry). Information on GAP and national MRLs was reported by Australia (Anon., 1996a) and Germany (Anon., 1996b), and on GAP by Norway (Anon., 1997a). The Netherlands provided information on analytical methods and use patterns, and residue data for celeriac, gherkins, mushrooms, wheat and fresh herbs (Anon., 1997b). Information on the fate of chlorothalonil residues during the processing of analytical samples and on GAP were received from the UK (Anon., 1997c).

This monograph reviews the residue data and other information which were not available to the 1993 JMPR. The Meeting reviewed the new information on residues in peaches, grapes, bananas, flowering brassicas, sweet corn and wheat in the context of that previously reviewed.

METABOLISM AND ENVIRONMENTAL FATE

Animal metabolism

Goats. A study to determine the nature of the residues in milk, meat and other tissues from lactating goats fed uniformly ring-labelled [^{14C}]chlorothalonil was carried out by Duane and Doran (1990). Two lactating goats at each dose level were dosed daily for eight days with [^{14C}]chlorothalonil at levels equal to 3 or 30 ppm in the diet. The average total radioactive residue (TRR) found in the milk and tissues (calculated as chlorothalonil equivalents), and the overall recovery of ^{14}C expressed as a percentage of the total dose in each compartment are shown in Table 1. Faeces and urine were the only major contributors to the total recovered dose. The percentage of the total dose recovered is greatly affected by the time of slaughter. The goats were all slaughtered within 8-10 hours after the administration of the last dose. Since no goats were depurated in this study, the unrecovered radioactivity is presumed to be in the intestinal tract.

Table 1. Mean concentrations of total radioactivity calculated as chlorothalonil and as a percentage of the total dose (Duane and Doran, 1990).

Sample	^{14}C			
	3 ppm dose		30 ppm dose	
	mg/kg[1]	% of total dose	mg/kg[1]	% of total dose
Faeces		61		63
Urine		6.6		6.9
Blood	0.04	0.2	0.5	0.2
Muscle	0.004	0.1	0.03	0.08
Liver	0.08	0.18	0.71	0.16
Kidney	0.22	0.09	2.2	0.07
Milk	0.009	0.17	0.096	0.25

[1]As chlorothalonil

The levels of ^{14}C expressed as chlorothalonil were highest in the blood, liver and kidney (apart from the excereta). The levels in the milk and meat were extremely low, with milk residues of 0.005 and 0.015 mg/kg and meat residues of 0.003-0.004 mg/kg from the low dose. The tissues with the highest TRR were the liver and kidney with residues which averaged 0.08 and 0.22 mg/kg respectively in the low-dose goats. In these organs the residues were complex mixtures of components with differing chemical and solubility characteristics. Because only very low levels of any discrete metabolites were present in any of the low-dose samples, the identification and characterization of the residues were conducted with the high-dose milk, liver and kidney.

There were no detectable residues of the parent compound in the milk or tissue samples (limit of detection 0.0004 mg/kg in milk and 0.003 to 0.005 mg/kg in liver and kidney). The 4-hydroxy metabolite of chlorothalonil (4-hydroxy-2,5,6-trichloroisophthalonitrile), designated SDS-3701, was identified in milk, liver and kidney, and was quantified at levels up to 0.007 mg/kg in low-dose milk, up to 0.05 mg/kg in high-dose milk, 0.05 mg/kg in high-dose liver and 0.08 mg/kg in high-dose kidney. The other major components of the residue that could be characterized were chlorothalonil conjugates with glutathione obtained as polar, water-extractable fractions from liver and kidney, and other conjugates that involved covalent binding of the ring to acid-precipitable protein in milk and extractable protein in kidney.

A study to determine the nature of the residues in the milk, meat and other tissues from lactating goats dosed with uniformly ring-labelled [^{14}C]4-hydroxy-2,5,6-trichloroisophthalonitrile (SDS-3701) was carried out by Han San Ku (1990). The distributions of the TRR after dosing at an exaggerated rate (2 ppm, 10 times the likely intake level) and at 0.2 ppm are shown in Table 2. More than 90% of the TRR in each fraction was solvent-extractable, and more than 90% of this was identified as SDS-3701 in each of the tissue fractions. No other metabolite was found in the milk or tissue samples. On the basis of these findings one can conclude that SDS-3701 is the only terminal residue resulting from the consumption of SDS-3701 by lactating goats, and that the level of SDS-3701 in the milk and tissues corresponds to the level of the TRR.

Table 2. Distribution of total radioactivity in goats dosed with [^{14}C-]SDS-3701, calculated as SDS-3701 (Han San Ku, 1990).

Sample	^{14}C, mg/kg as SDS-3701	
	0.2 ppm dose	2 ppm dose
Kidney	0.17-0.26	0.88-1.5
Liver	0.07	0.56-0.76
Heart	0.04-0.05	0.44-0.5
Muscle	0.01-0.02	0.12-0.14 (rear leg) 0.13-0.14 (loin)
Fat	0.01-0.02	0.08-0.09 (omental) 0.07-0.09 (perireneal)
Milk	0.09-0.15	0.22-1.0
Urine		0.04-0.3

Two minor metabolites SDS-47524 (2,5,6-trichloro-3-cyanobenzamide) and SDS-47525 (2,4,5-trichloro-6-hydroxy-3-cyanobenzamide) were tentatively identified in urine samples on the basis of HPLC retention times. The concentration of these metabolites in urine was very low (<0.014 mg/kg).

Poultry. A residue study on laying hens was conducted with [^{14}C]chlorothalonil to determine whether chlorothalonil would produce residues in eggs and tissues (Capps *et al.*, 1983a). Four dose levels (10 birds/dose) were used.: 0, 2, 6 and 20 ppm based upon 120 g/day food consumption. The doses were equivalent to about 0.22, 0.65, and 2.18 mg/kg bw, on the basis of an average body weight of 1.1 kg. The birds were dosed once daily for 21 days. The TRR was calculated as chlorothalonil.

No radioactivity (<0.04 mg/kg) was found in egg whites at any dose level, nor in egg yolks at the low- or mid-dose levels at any sampling interval. The high-dose yolks (20 ppm) showed a maximum total radioactivity of 0.047 mg/kg from day 13 of dosing. Since no ^{14}C was found in egg whites, the residues on a whole-egg basis would be decreased by at least 50%. In the tissues the only detectable residues were in the liver. The low-dose (2 ppm) liver showed no detectable residue (<0.04 mg/kg). The highest TRR of 0.098 mg/kg was in the mid-dose (6 ppm) liver within 6 hours after the final dose. These residues also were lost within three days. The high-dose (20 ppm) liver showed total radioactivity equivalent to 0.05 mg/kg within 6 hours after the final dose, and had disappeared within three days. There is no explanation for the fact that the mid-dose residue was higher than the high-dose. The only possible explanation given by the authors is that the samples were mislabelled or switched at the time of slaughter or analysis.

A similar study was carried out with uniformly ring-labelled SDS-3701 (Capps *et al.*, 1983b). The details were the same except that the doses were 0, 0.1, 0.3 and 1 ppm, approximately equivalent to 0, 0.011, 0.033, and 0.11 mg/kg bw, on the basis of an average body weight of 1.1 kg. The TRR was calculated as SDS-3701.

Again no ^{14}C (<0.04 mg/kg) was detectable in egg whites at any dose level, and in yolks the TRR from the low dose reached a plateau at approximately 0.04 mg/kg on day 21. These residues are very close to the limit of detection and decreased rapidly (undetectable on day 23). The mid-dose yolks showed a maximum TRR of 0.12 mg/kg by day 21 of dosing, which disappeared in seven days. The high-dose yolks contained a maximum TRR of 0.42 mg/kg which reached a plateau by day 16. Approximately 50% of the residue was lost in seven days.

The identity of the residue found in the egg yolks was established as unchanged SDS-3701 (Nelson *et al*., 1984). Analysis of the poultry tissues revealed no detectable radioactivity in fat (<0.04 mg/kg), adductor muscle (<0.03 mg/kg), or pectoral muscle (<0.04 mg/kg). The skin from the high-dose (1 ppm) group showed a maximum TRR of 0.04 mg/kg but that from the low and mid-dose groups contained no detectable residues (<0.03 mg/kg). The liver from the low-, mid- and high-dose groups showed maximum residues of 0.06, of 0.27, and 0.78 mg/kg respectively. The residues disappeared within seven days. Residues in the heart tissues were undetectable (<0.02 mg/kg) in the low-dose group and reached maximum levels of 0.055 and 0.15 mg/kg in the mid- and high-dose groups respectively. These residues were lost within seven days (Capps *et al*., 1983b).

Reaction kinetics in ruminant tissues. A reaction kinetic study with ruminant tissues by Jenhoft (1994) was designed to determine the mechanism of the rapid loss of chlorothalonil in biological systems and to measure how rapidly it reacts with bovine tissue components at physiological temperatures.

The general approach used was to incubate [^{14C}]chlorothalonil with bovine tissue homogenates or blood and measure the rate of its disappearance. Tissues were homogenized in pH 7 phosphate buffer. Blood was mixed with EDTA to prevent clotting and used directly, and plasma was prepared from whole blood by centifigation. After the addition of a solution of [^{14C}]chlorothalonil the reaction mixtures were incubated at 37°C until the reactions were terminated by the addition of perchloric acid. Each mixture was then extracted three times with ethyl acetate and centrifuged to give three fractions for analysis: the combined extracts, an aqueous phase and a pellet of precipitated protein. Unreacted chlorothalonil was recovered in the ethyl acetate extracts and the rate of decrease of extractable radioactivity was used to measure the rate of chlorothalonil metabolism. The residual unextractable radioactivity was recovered as polar metabolites in the aqueous phase and as residue covalently bound to protein in the pellet.

Chlorothalonil reacts extremely rapidly with components of bovine tissue homogenates giving rise to polar metabolites and bound residues. The half-lives of chlorothalonil in liver, kidney, and muscle homogenates were 15 seconds, 30 seconds, and 45 seconds. The half-life in whole blood was 15 seconds and in plasma 1 minute. In livestock, chlorothalonil absorbed from the gastrointestinal tract would be very short-lived and would not remain as a residue in food items such as meat, liver, milk or edible offal.

HPLC retention times and the results of studies with model compounds indicate that the polar metabolites are largely glutathione conjugates. The bound residue is attributed to the reaction of free thiols in proteins with chlorothalonil (whole blood 14-17%, muscle 10-13%, liver 15-18%, kidney 30-35% of the original ^{14}C).

METHODS OF RESIDUE ANALYSIS

Analytical methods

Chlorothalonil is determined in fatty and non-fatty foods in The Netherlands by a multi-residue method (Anon., 1996c). The determination is by gas chromatography with an electron capture or ion trap detector with an LOD of 0.01 mg/kg and recoveries of 89-104 %.

Stability of pesticide residues in stored analytical samples

Chlorothalonil residues were stable during freezer storage for one year in cherries, cucumbers, tomatoes, carrots, potatoes, celery and wheat grain (1993 JMPR). The current Meeting received data on the storage stability of chlorothalonil and SDS-3701 in bovine milk and tissues.

The stability of chlorothalonil in milk and cow tissues stored at -25 to -10°C was determined by King and Prince (1995a). The results (Table 3) show a slow loss from body fat and a rapid loss from the other samples.

Table 3. Rate of loss of chlorothalonil during frozen storage (King and Prince, 1995a).

Sample	Mean loss of chlorothalonil, %, after								
	0 h	8 h	16 h	24 h	48 h	4 d	7 d	14 d	29 d
Liver	27	91	94	100					
Muscle	19	75	90	90	95				
Kidney	21	74	87	87	94				
Milk	0	13	51	59	82	91			
Body fat	23	22	27	26	28	33	29	32	41

King and Prince (1995b) also determined the stability of SDS-3701 in milk and cow tissues stored under frozen conditions for a year. The compound was stable in milk but decreased in muscle by 8%, body fat by 9% and liver by 17%.

Losses of chlorothalonil residues during the laboratory processing of various fruit and vegetable samples (broccoli, celery, lemons, lettuce) were first identified by Hill and Oliver (1994). The disappearance of chlorothalonil occurred quite rapidly during and after sample comminution at room temperature (the mean losses after 1 h were 95% in lettuce, 80% in broccoli, 60% in celery and 45% in lemons), but subsequent losses were minimal during storage in the freezer. The losses during the processing of lettuce were inhibited if the chlorothalonil was added after killing the lettuce cells by heating in a microwave oven. There were no losses from ethyl acetate extracts of lettuce but chlorothalonil was found to disappear rapidly and completely from similar extracts of onions.

In two further studies by Chambers *et al*. (1996) and Hill *et al*. (1996), the fate of chlorothalonil added to onion extracts and pulped fresh lettuce was investigated with labelled and (by LC-MS) unlabelled pesticide. Lettuce and onions were processed in the fresh state and frozen with dry ice. About 30% of the chlorothalonil added to pulped fresh lettuce became bound to components of the lettuce which were insoluble in water and ethyl acetate. The extractable portion of the residue remained largely as intact chlorothalonil but it is not known whether the bound product was capable of liberating the intact pesticide. Chlorothalonil added to ethyl acetate extracts of onions reacted rapidly and completely to form several more polar components, none of which appeared likely to be able to regenerate chlorothalonil. It is not known whether these were produced sequentially or in parallel. Reaction with sulphur compounds in the onion extracts is a likely route of degradation and one product was partially characterized by LC-MS as a trichlorodicyanothiophenol. The product was not sufficiently volatile or stable for gas chromatographic separation and further characterization was hindered by its breakdown during LC-MS ionization to the corresponding phenylthiolate ion.

Processing frozen lettuce samples in dry ice ("cryogenic milling") appeared to reduce losses of labile pesticides, including chlorothalonil.

Definition of the residue

Because the metabolite SDS-3701 is considered to be of toxicological importance, the Meeting recommended its inclusion in the definition of the residue for the estimation of dietary intake in products of animal origin.

Definition of the residue in animal products for compliance with MRLs: chlorothalonil.

Definition of the residue in animal products for estimating dietary intake: sum of chlorothalonil and 4-hydroxy-2,5,6-trichloroisophthalonitrile, expressed as chlorothalonil.

Definition of the residue for compliance with MRLs and for estimation of dietary intake in plants: chlorothalonil.

Chlorothalonil is not fat-soluble (log P_{OW} = 2.87).

USE PATTERN

Chlorothalonil is a non-systemic protectant fungicide. The Meeting received updated information from the manufacturer on GAP for commodities for which new data from supervised trials are available. GAP for other commodities is detailed in the 1993 evaluation. Additional information on GAP was received from Australia (Anon., 1996a), Germany (Anon., 1996b), Norway (Anon., 1997a), The Netherlands (1997b) and the UK (Anon., 1997c). Registered uses in various countries are shown in Table 4. The registered use is outdoors unless otherwise stated. "Foliar spray" and "overall spray" refer to ground and aerial applications respectively.

Table 4. Registered uses of chlorothalonil.

Crop	Country	Form.	Application			PHI, days
			Method	Rate per appl. kg ai/ha (kg ai/hl)	Number	
Almonds	Australia	SC		2.3	Multiple	
Artichokes	Australia	SC		1.3-1.65	Multiple	1
Bananas	Australia	SC		1.1 -2.16	Multiple	1
	Latin America	SC	aerial	0.88- 1.63	Multiple	0
Barley	UK	SC	overall spray	0.5-1	1-2	NS
Beans (dry)	European Union[1]	SC WG	foliar spray	1.5	2	EF
	UK	SC WG	overall spray	1.5	2	EF
	USA	SC	aerial	1.2-1.75	multiple	43
		SC	ground	1.2-1.75	multiple	43

Crop	Country	Form.	Application			PHI, days
			Method	Rate per appl. kg ai/ha (kg ai/hl)	Number	
Beans (fresh)	European Union[1]	SC WG	foliar spray	1.5	2	10
	UK	SC	overall spray	1 1.5	2 2	7 14
		WG	overall spray	1 1.5	2 2	7 14
Blackberries	European Union[1] UK	SC WG	foliar spray	2.5	4	28
		SC WG	overall spray	2.5	4+2[2] 4	3 28
Broad beans (*Vicia faba*)	Australia	SC	foliar spray	0.8-1.65	multiple	7
Broccoli	Australia	SC		1.25-2.5	multiple	3
	European Union[1]	SC WG	foliar spray	1.5	2	7
	UK	SC	overall spray	1.5	2	7
	USA	WG		1.7		7
Brassica vegetables (under breeding)	Norway	EC	foliar spray	(0.15)	2	BF
Brussels sprouts	Australia	SC		1.25-2.5	multiple	3
	The Netherlands	SC	spraying	1.5	2-3	14
	UK	SC WG	overall spray	1.5	2	7
Cabbage	Australia	SC		1.25-2.5	multiple	3
	UK	SC WG	overall spray	1.5	2	7
Calabrese	UK	SC WG	overall spray	1.5	2	7
Carrots	Australia	SC		1.3 (0.12)	multiple	7
	Norway	EC	foliar spray	1.5-2.25 (0.15-0.55)	1-2	14
Cauliflower	Australia	SC		1.25-2.5	multiple	3
	UK	SC WG	overall spray	1.5	2	7
Celeriac	The Netherlands	SC WP	spraying	1.88	3-5	28
	UK	SC	overall spray	1.5	3	28
Celery	Australia	SC		0.86-1.3 (0.115)	multiple	1
	Norway	EC	foliar spray	1.5-2.25 (0.15-0.55)	1-2	14
	UK	SC WG	overall spray	1.5	3	7
Celery leaves	The Netherlands	SC, WP	spraying	1.87	3-5	28

Crop	Country	Form.	Application			PHI, days
			Method	Rate per appl. kg ai/ha (kg ai/hl)	Number	
Chinese cabbage	UK	SC WP	overall spray	1.5	2	7
Citrus fruit	European Union[1] Spain	WG WG	foliar spray foliar spray	1.25 1.25	2 2	28 28
Cucumbers	Norway The Netherlands UK	EC SC WP SC	foliar spray spraying overall high volume spray	1.5-2.5 (0.15-0.55) 0.75-2.25 (0.15) (0.11)	1-2 3-5 2	4 3 (G) 2 (G)
Cucurbits	Australia	SC		1.2-1.8	multiple	1
Currants	European Union[1] UK	SC WG SC WG	foliar spray overall spray	2.5 2.5	4 3+2[3]	28 28
Endive	Australia	SC		1.3-1.65	multiple	1
Gherkins	The Netherlands	SC, WP	spraying	0.75-2.25 (0.15) 0.60-1.2 (0.15)	3-5 2-4	3 (G) 3 (F)
Gooseberry	UK	SC WG	overall spray	2.5	3+2[3]	28
Grapes	Australia	SC	foliar spray	1.3-1.65 (0.12-0.15)	multiple	7[4] 14[5]
Hops	UK	SC WG	overall spray	1.5		10
Leeks	Australia Norway The Netherlands UK	SC EC SC, WP SC	 foliar spray spraying overall spray	1.3-1.65 1.5 (0.3-0.6) 1.5 1	multiple 1-2 4-6 3	1 14 14 14
Melons	The Netherlands	SC, WP	spraying	0.75-2.25 (0.15)	3-5	3 (G)
Mushrooms	The Netherlands UK	WP SC WG	soil treatment	22.5 (0.23) 30 (0.3)[6] 11.25	2 1 2	7 (G) 10 G) 1 (G)
Onion	Norway The Netherlands UK	EC SC, WP SC WG	foliar spray overall spray	1.5 (0.3-0.6) 0.5[7] 1-1.5 0.98	1-2 4-6 6	14 7[7] 14 14
Onions (excluding spring onion)	Australia	SC		1.65	multiple	7

Crop	Country	Form.	Application			PHI, days
			Method	Rate per appl. kg ai/ha (kg ai/hl)	Number	
Parsley	Norway	EC	foliar spray	1.5-2.25 (0.15-0.55)	1-2	14
	The Netherlands	SC, WP	spraying	1.87	3-5	28
Peaches	European Union[1]	SC	foliar spray	1.5	4	FS
	Italy	SC	foliar spray	1.5	4	14
	Spain	SC	foliar spray	1.5	4	FS
Peanuts	Australia	SC		0.8-1.3 (0.07-0.12)	multiple	
Peas	Australia	SC		0.8-1.3 (0.07-0.12)	multiple	7
a) vining b) combining	UK	SC	overall spray	1-1.5	2	a) 14 b) 42
Peppers	Australia	SC		1.3-1.65	multiple	1
	Brazil	SC WP	foliar spray	1.75	multiple	7
	Latin America[8]	SC WP	foliar spray	0.75-1.8	multiple	7
Plums	Australia	SC		2.3	multiple	1
Potatoes	Australia	SC		0.8-1.3	multiple	14[9]
	The Netherlands	SC, WG	spraying	1.5-2.25	15	-
				0.75-1.0[10]	15	-
		SC		1.0[11]	4-8	-
	UK	WP WG	a)overall spray b) aerial	1.0-1.5	5	7
Radishes	Australia	SC		1.3-1.65	multiple	1
Rape	UK	SC	overall spray	1.5	2	NS
Rhubarb	Australia	SC		2.0	multiple	1
Shallots	Australia	SC		1.3-1.65	multiple	1
	The Netherlands	SC; WP	spraying	0.5[7] 1-1.5	4-6	28
Squash, Summer	The Netherlands	SC, WP	spraying	0.75-2.25 (0.15) 0.60-1.2 (0.15)	3-5 2-4	3 (G) 3 (F)
Stone fruits	Australia	SC		2.3	multiple	7
Strawberries	Norway	EC	foliar spray	(0.125)	1-2	14
	UK	SC WG	overall spray	3	4	14
Sweet corn	Australia	SC		1.3-1.65	multiple	1
	USA	SC	foliar spray	0.7-1.6	multiple	14
		SC	aerial	0.7-1.6	multiple	14
Tobacco	Australia	SC		(0.16)		

Crop	Country	Form.	Application			PHI, days
			Method	Rate per appl. kg ai/ha (kg ai/hl)	Number	
Tomatoes	Australia	SC		1.3-1.65 (0.12-0.15)	multiple	1
	The Netherlands	SC WP	spraying	0.75-2.25 (0.15) 0.37-1.12 (0.75)[12] (0.11)	3-5	3 (G)
	UK	SC	overall high volume spray		2	2 (G) 2
Watercress	Australia	SC		1.3-1.65	multiple	1
Wheat	Germany	WG SC		0.5-1.1 0.75-0.125[13]	3 1	42 35
	The Netherlands	SC, WP	spraying	1.0	1	42
	UK	SC	overall spray	0.5 -1.0	1-3	NS

[1]Proposed GAP
[2]4 applications pre-harvest, 2 post-harvest
[3]3 applications pre-harvest, 2 post-harvest
[4]Table grapes
[5]Wine grapes
[6] Mixture of chlorothalonil and prochloraz
[7]Mixture of chlorothalonil and other fungicides (vinclozolin, prochloraz), PHI 7 days
[8] Argentina, Costa Rica, Dominican Republic, Ecuador, Guatemala, El Salvador
[9]Before desiccation or harvest
[10]Mixture of chlorothalonil and maneb
[11]Mixture of chlorothalonil and propamocarb
[12]Mixture of chlorothalonil and vinclozolin
[13]Mixture of chlorothalonil and propiconazole, PHI of 35 days based on the use of propiconazole
BF-Before planting
EF-End of flowering
FS-Last treatment when fruit is nut size
F-Outdoors
G-Indoors
NS-PHI controlled by stage of growth at time of application. PHI in days not stated

RESIDUES RESULTING FROM SUPERVISED TRIALS

Data from supervised residue trials on oranges, mandarins, peaches, grapes, blackberries, black currants, bananas, broccoli, gherkins, peppers, mushrooms, sweet corn, beans, celeriac, wheat and fresh herbs are shown in Tables 5 to 19.

In the Tables each entry in the left hand column represents a different site or year. Where two or more residues are shown for a single combination of trial, type of sample, and PHI they are the residues found in separate field samples. Where reports listed replicate analytical results their means are shown in the Tables. Residues are not corrected for recovery except where indicated.

Underlined residues in the Tables reflect current GAP. Double-underlined residues have been used for the estimation of supervised trials median residue (STMR) levels.

Citrus fruits (Table 5). Residue data were available from a series of trials in Spain according to GAP (2 x 1.25 kg ai/ha, PHI 28 days). Chlorothalonil residues were 0.26-1.9 mg/kg after 28 days.

Table 5. Residues of chlorothalonil in oranges and mandarins in Spain. All WG applications. Whole fruits analysed.

Year	ApplicationNo	kg ai/ha	PHI,days	Residues,mg/kg	Report
Oranges					
1995			071426	2.51.51.50.91	5-ISKCIT95/13
1995	2	1.25	071428	1.40.770.740.81	5-ISKCIT95/13
1995	2	1.25	28	0.26	5-ISKCIT95/13
1996	2	1.25	071429	1.32.51.11.8	5-ISKORA96/07
1996	2	1.25	27	1.9	5-ISKCIT95/13
Mandarins					
Spain,1995	2	1.25	27	0.72	5-ISKCIT95/13

Peaches (Table 6). Chlorothalonil is registered in Italy for 4 x 1.5 kg ai/ha and a PHI of 14 days, but the proposed GAP in the EU requires the last treatment to be not later than nut size of the fruit (PHI about 60 days). Table 6 includes new data from southern Europe previously considered by the 1993 JMPR. The residues are in the pulp without stone.

Table 6. Residues of chlorothalonil in peaches. Pulp analysed.

Country, Year	Form.	Application No	kg ai/ha (kg ai/hl)	PHI, days	Residues, Mg/kg	Report
Italy, 1990	WP	2	0.84 (0.1)	21	0.18	JMPR 1993
Italy, 1990	WP	2 3	1.7 (0.2) 0.82 (0.1)	21 21	0.57 0.14	JMPR 1993
Italy, 1990	SC	4 4	1.0 (0.04) 2.0 (0.09)	21 21	0.98 1.3	JMPR 1993
Italy, 1990	WG	3	1.5 (0.1)	64	<0.01	JMPR 1993
Italy, 1990	WP	3	1.25 (0.09)	64	<0.01	JMPR 1993
Spain, 1990	WP	1 1	2.0 (0.11) +2.6 (0.15)	61	0.16	JMPR 1993
Spain, 1990	WP	1 1	2.0 (0.11) +2.6 (0.15)	83	0.01	JMPR 1993
Spain, 1990	WP	1 1	2.0 (0.11) +2.6 (0.15)	155	0.02	JMPR 1993

Country, Year	Form.	Application No	kg ai/ha (kg ai/hl)	PHI, days	Residues, Mg/kg	Report
Spain, 1990	SC	4 4 4	0.5 0.75 1.25	82 82 82	<0.01 ≤0.01 ≤0.01	JMPR 1993
Spain, 1991	SC	4 4 4	0.5 0.75 1.25	69 69 69	≤0.01 ≤0.01 0.01	JMPR 1993
Italy, 1992	SC	3	0.5	66	<0.01(4)	CTL/PEACH 19/I/92
Italy, 1992	SC	3	0.75	66	<0.01(4)	CTL/PEACH 19/I/92
Italy, 1992	SC	3	1.5	66	≤0.01 (3), 0.04	CTL/PEACH 19/I/92
Italy, 1994	SC	4	1.5	21	0.65	CTL/PEACH 28/I/94
Italy, 1994	WG	4	1.5	21	0.59	CTL/PEACH 28/I/94
Spain, 1994	SC	4	1.5	21	0.87	CTL/PEACH 25/E/94
Spain, 1994	WG	4	1.5	21	1.4	CTL/PEACH 25/E/94
Spain, 1994	SC	4	1.5	20	0.77	CTL/PEACH 26/E/94
Spain, 1994	WG	4	1.5	20	0.54	CTL/PEACH 26/E/94
Spain, 1994	SC	3	1.5	87	0.03	CTL/PEACH 27/E/94
Spain, 1994	WG	3	1.5	87	0.15	CTL/PEACH 27/E/94

Grapes (Table 7). Five new residue trials in Australia were reported. In three of them, fresh fruit and dried sultanas were analysed. Table 7 includes the new data and Australian data previously reviewed by the 1983 JMPR.

Table 7. Residues of chlorothalonil in grapes, Australia.

Region, Year	Form.	Application No	kg ai/ha (kg ai/hl)	Sample	PHI, days	Residues, mg/kg[1]	Report
Hunter Valley, 1973/74	WP	7	(0.11)	fresh fruit	0 10	6.1, 7.1 5.6 (8.8)	JMPR 1983
Hunter Valley, 1973/74	WP	7	(0.22)	fresh fruit	0 10	11 8.7 (13.6)	JMPR 1983
South Australia 1973/74	WP	6	(0.13)	fresh fruit	1 7 18 26	1.4 0.6 1.6 (2.9) 0.6, 0.3	JMPR 1983
South Australia 1973/74	WP	6	(0.26)	fresh fruit	1 7 18 26	2.3 3.1 2.7 (4.9) 0.8	JMPR 1983
NorthvAustralia 1991/92	SC	7 5 3	(0.15) (0.15) (0.15)	fresh fruit	28 77 113	0.6 0.04 <0.01	JMPR 1993
Hunter Valley, 1990/91	SC	7 6 4	(0.15)	fresh fruit	15 30 66	1.4 0.5 0.2	JMPR 1993
Langhorn Creek, 1991	SC	6 5 3	(0.15) (0.15) (0.15)	fresh fruit	19 63 111	2.3 <0.02 <0.02	JMPR 1993
1993	SC(b)	1	2.25 (0.15)	fresh fruit sultanas, dry	0 7 14 21 28 14	10.3 5.2 4.2 1.9 1.3 0.53	ISK-AUST-94-1
1993	SC(a)	1	1.9 (0.125)	fresh fruit	0 7 21	11 4.8 1.5	ISK-AUST-94-1
1993	SC(b)	1 2 3 4	2.3 2.3 2.3 3.4	sultanas, dry	96 84 78 60	<0.03 <0.03 0.05 0.11	ISK-AUST-94-1
1992	SC(a)	4	1.9 (0.125)	fresh fruit sultanas, dry	60 60	0.08 <0.05	ISK-AUST-94-1
1992	SC(a)	4	4.6 (0.3)	fresh fruit sultanas, dry	60 60	0.43 0.30	ISK-AUST-94-1

[1] Figures in parantheses are corrected for recovery (64% Hunter Valley, 55% S. Australia)
(a) treatment with Flute (500 g ai/l chlorothalonil and 8 g ai/l flusilazole)
(b) treatment with Bravo (500 g ai/l chlorothalonil)

Blackberries and currants (Table 8). Chlorothalonil is registered for pre-harvest use on blackberries and currants in the UK (4 and 3 x 2.5 kg ai/ha respectively, 28-day PHI). One underdosed trial on blackberries in Sweden and 6 trials on black currants in the UK (3 x 2.5 kg ai/ha, PHI 28 days) were reported.

Table 8. Residues of chlorothalonil in blackberries and black currants.

Country, Year	Form.	Application No	kg ai/ha	Residues, mg/kg	PHI, days	Report
Blackberries						
Sweden,1983	SC	1	1.25	<0.01 <0.01 <0.01	71421	CTL/RUBFR01/S/83
Blackcurrants						
UK,1995	SC	3	2.5	3.3	28	AK/2782/1B
UK,1995	SC	3	2.5	3.8	26	AK/2782/1B
UK, 1996	SC	3	2.5	1.9	28	CTL/RIBNI03/6B/96
UK,1996	WG	3	2.5	1.5	28	CTL/RIBNI03/6B/96
UK,1996	SC	3	2.5	0.94	27	CTL/RIBNI03/6B/96
UK,1996	WG	3	2.5	0.83	27	CTL/RIBNI03/6B/96

Bananas (Table 9). The results of 6 new trials on bananas have been reported. These included field trials in 1992/93 from Columbia, Costa Rica, Guatemala, Honduras and Panama according to the Latin American GAP of multiple aerial treatments with a maximum of 1.6 kg ai/ha and a 0-day PHI. All samples were also analysed for the metabolite SDS-3701 and the technical impurity hexachlorobenzene (HCB). Residues were below the LODs of 0.01 and about 0.00025 mg/kg respectively.

Table 9 includes new data and data previously considered by the 1993 JMPR.

Table 9. Residues of chlorothalonil in bananas. Whole fruit analysed.

Country, Year	Form	Application No	kg ai/ha	PHI, days	Residues mg/kg	Report
Australia, 1978	SC	10	1.1 (ground treatment)[1]	1 14 28	0.6 0.44 0.03	JMPR 1993,
Australia, 1978	SC	10	2.2 (ground treatment)[1]	1 14 28	2.0 0.1 0.09	JMPR 1993,
Columbia, 1985	SC	11	1.5 (aerial treatment)	3	<0.01 (6)	JMPR 1993
Costa Rica, 1985	WP	10	1.75 (aerial treatment)[1]	6	0.02, 0.03 (2), 0.1, 0.11, 0.12	JMPR 1993
Mexico, 1984/85	WP	13	1.1-1.5 (aerial	2	<0.01 (6)	JMPR 1993

Country, Year	Form	Application No	kg ai/ha	PHI, days	Residues mg/kg	Report
			treatment)			
Panama, 1978	SC	8	1.3 (aerial treatment)	0	<0.01 (4)	JMPR 1993
Colombia, 1993	SC	20	1.7 (aerial treatment)[2]	0	<0.01	5529-92-0515-CR-001
Guatemala, 1993	SC	20	1.7 (aerial treatment)[2]	0	<0.01	5529-92-0515-CR-002
Honduras, 1993	SC	20	1.7 (aerial treatment)[2]	0	<0.01	5529-92-0515-CR-002
Costa Rica, 1993	SC	20	1.7 (aerial treatment)[2]	0	<0.01	5529-92-0515-CR-002
Honduras, 1993	SC	15	1.7 (aerial treatment)[2]	0 0	<0.01	5529-92-0515-CR-002
Panama, 1993	SC	20	1.7 (aerial treatment)[2]	0	<0.01	5529-92-0515-CR-002

[1] Unbagged bananas
[2] Bagged bananas

Broccoli (Table 10). Two new trials according to UK GAP were carried out on a single site in 1996.The use pattern in two US trials reported to the 1993 JMPR was similar.

Table 10. Residues of chlorothalonil in broccoli.

Country,Year	Form	Application No	kg ai/ha	PHI,days	Residues,mg/kg	Report
USA, 1985		4	1.3	6	2.2	JMPR 1993
USA, 1987		8	1.3	7	2.6	JMPR 1993
UK, 1996	SC	2	1.5[1]	7	1.2[2] (0.83, 1.5)	CTL/BRSOK 01/GB/96
UK, 1996	WG	2	1.5[3]	7	2.1[2] (1.8, 2.3)	CTL/BRSOK 01/GB/96

[1]Treatment with Bravo 500
[2]Mean of two field samples, single values in parantheses
[3]Treatment with ISK 375

Gherkins (Table 11). One trial was reported by The Netherlands. Four field samples were analysed for chlorothalonil only.

Table 11. Residues of chlorothalonil in gherkins (indoor), The Netherlands, 1973. WP formulation.

Application No.	kg ai/ha	Sample	PHI, days	Residues, mg/kg	Report
1	2.19	unwashed	0	2.9, 3.8, 4.3, 5.4	KvW174/CvF/PD4.2.(2.1.08)-1973
		washed	0	0.9, 1.0, 1.2 ,1.7	
		unwashed	3	0.64, 0.7, 0.85, 1.1	
		washed	3	0.24, 0.26, 0.33, 0.37	

Peppers (Table 12). Eight trials on bell peppers were undertaken in Australia during 1996 at two sites (Waikerie, South Australia and Bundaberg, Queensland). Only chlorothalonil was determined. In five trials in Latin America on bell peppers in 1996, all the samples were analysed for SDS-3701 as well as chlorothalonil. Residues of chlorothalonil were found in the peppers from all the treated plots except those harvested at the 3-day PHI from Ensenada, Baja California, Mexico. These may have been from the untreated plot. The levels of chlorothalonil from the five trials at the GAP PHI of 7 days ranged from 0.05 mg/kg (Santa Rita, Honduras) to 5.4 mg/kg (Santa Ana, Costa Rica). SDS-3701 was detected in peppers from the plots in Costa Rica and Chile at levels not exceeding 0.04 mg/kg. No SDS-3701 was detected in peppers from the other three locations.

Two further trials were carried out in Brazil in 1986, one according to GAP. Samples were analysed for chlorothalonil only.

Table 12. Residues of chlorothalonil in sweet peppers.

Country, Year	Form.	Application No	kg ai/ha	PHI, days	Residues, mg/kg	Report
S. Australia, 1996	SC	6 6	1.65 3.3	6 6	2.5 7.1	960732/815
S. Australia, 1996	SC	7	1.65	0 1 3 7	3.7 <u>5.3</u> 7.1 2.4	960732/815
S. Australia, 1996	SC	7	3.3	0 1 3 7	15 13 12 8.4	960732/815
Australia, Queensland, 1996	SC	7 7	1.65 3.3	7 7	0.26 0.69	960732/815
Australia, Queensland, 1996	SC	8	1.65	0 1 3 7	0.57 <u>0.43</u> 0.28 0.22	960732/815
Australia, Queensland, 1996	SC	8	3.3	0 1 3 7	2.3 1.3 1.3 0.54	960732/815
Brazil, 1986	SC	3 3	1.75 3.5	7 7	<u>0.04</u> 0.06	318/36
Costa Rica, 1996	SC	7	1.88	3 7	4.5 <u>5.4</u>	6870-96-0152-CR-001
Chile, 1996	SC	10	1.83	3 7	6.9 <u>4.1</u>	6870-96-0152-CR-001
Honduras, 1996	SC	9	1.83-1.88	3 7	0.13 <u>0.05</u>	6870-96-0152-CR-001

Country, Year	Form.	Application No	kg ai/ha	PHI, days	Residues, mg/kg	Report
Mexico, Sinaloa, 1996	SC	12	1.78-1.86	3 7	0.96 1.4	6870-96-0152-CR-001
Mexico, Baja Calif., 1996	SC	12	1.74-1.92	3 7	<0.01[1] 1.6	6870-96-0152-CR-001

[1]No residues were detected in either subsample at the 3-day interval. Peppers from the untreated plot may have inadvertently been sampled

Mushrooms (Table 13). One study was reported by The Netherlands. One soil treatment was carried out immediately after casing and repeated after two weeks. Four field samples were analysed for chlorothalonil only.

Table 13. Residues of chlorothalonil in mushrooms (indoor), The Netherlands, 1983. WP formulation.

Applicatios No	kg ai/ha	Residues, mg/kg[1]	PHI, days	Report
2	21.9	0.57, 0.73, 0.75, 0.78[1]	7	KvW240/CTB/PD7101.300.311

[1] Corrected for recovery (0.4 mg/kg: 67%, n=5, CV=4.8%)

Sweet corn (Table 14). In trials at test sites in Pennsylvania, Oregon and Wisconsin (USA) the residues of chlorothalonil, SDS-3701, and HCB were determined in the ears and forage of sweet corn. Eight broadcast applications were made to each treated plot, 20-30 days after planting, approximately every 7 days. All samples of ears were free of SDS-3701 and HCB residues down to the LOD (0.01 and 0.00025 mg/kg respectively). Chlorothalonil was detected in only one sample, at 0.01 mg/kg. The forage samples contained varying residues at the different sites. Table 14 includes the results of one trial reviewd by the 1993 JMPR.

Table 14. Residues of chlorothalonil, SDS-3701 and HCB in sweet corn, USA. All SC formulations, 14-day PHI.

State, Year	Application No	kg ai/ha	Sample	Residues, mg/kg Chloro-thalonil	SDS-3701	HCB	Report
Illinois, 1985	8	1.6	grain	<0.01			JMPR 1993
Oregon, 1995	8	1.3	forage cobs	28 <0.01	0.05 <0.01	0.0086 <0.00025	6513-955-0270-CR-001
Pennsyl., 1995	8	1.3	forage cobs	58 <0.01	0.07 <0.01	0.016 <0.00025	6513-955-0270-CR-001
Wiscon., 1995	8	1.3	forage cobs	8.2 <0.01	0.07 <0.01	0.0033 <0.00025	6513-955-0270-CR-001

Beans, dry (Table 15). Numerous supervised trials were carried out in the UK from 1986 to 1992. The samples (dry beans without pods) were analysed for chlorothalonil only. Residues from trials at rates near UK GAP (2 x 1.5 kg ai/ha) ranged from <0.01 mg/kg to 0.02 mg/kg.

Supervised trials in the USA were carried out in Minnesota, Illinois, North Dakota, Colorado, Delaware, Nebraska, Michigan and Tennesee. Bean plants were treated with three to five applications of 1.2 to 2.3 kg ai/ha. Residues of chlorothalonil in the dry beans (without pods) from trials according to US GAP (1.8 kg ai/ha, 43-day PHI) ranged from undetected to 0.05 mg/kg. All samples in the US trials were also analysed for SDS-3701 and the impurities hexachlorobenzene (HCB) and pentachlorobenzonitrile (PCBN); the residues were below the LODs of 0.03, 0.004 and 0.01 mg/kg respectively.

Table 15. Residues of chlorothalonil in dry field beans.

Country, Year	Form.	Application		Sample	PHI, days	Residues, mg/kg	Report
		No	kg ai/ha				
UK, 1986	SC	2	1.0	beans straw	51 51	<0.01 0.18	CTL/PHSSS 02/GB/86
UK, 1986	SC[1]	2	0.9	beans straw	51 51	<0.01 0.18	CTL/PHSSS 02/GB/86
UK, 1986	SC	2	1.0	beans straw	71 71	<0.01 0.33	CTL/PHSSS 03/GB/86
UK, 1986	SC[1]	2	0.9	beans straw	71 71	<0.01 0.19	CTL/PHSSS 03/GB/86
UK, 1990	SC	2	1.5	beans	84	<0.01	5CTL/PHSSS 14/GB/90
UK, 1990	SC	2	3.0	beans	84	<0.01	CTL/PHSSS 14/GB/90
UK, 1990	SC[2]	2	1.5	beans	84	<0.01	CTL/PHSSS 14/GB/90
UK, 1990	SC[2]	2	3.0	beans	84	<0.01	CTL/PHSSS 14/GB/90
UK, 1990	SC[1]	2	0.9	beans	84	<0.01	CTL/PHSSS 14/GB/90
UK, 1990	SC[1]	2	1.8	beans	84	<0.01	CTL/PHSSS 14/GB/90
UK, 1990	SC	2	1.5	beans	62	<0.01	CTL/PHSSS 14/GB/90
UK, 1990	SC	2	3.0	beans	62	0.14	CTL/PHSSS 14/GB/90
UK, 1990	SC[2]	2	1.5	beans	62	<0.01	CTL/PHSSS 14/GB/90
UK, 1990	SC[2]	2	3.0	beans	62	0.09	CTL/PHSSS

Country, Year	Form.	Application No	kg ai/ha	Sample	PHI, days	Residues, mg/kg	Report
							14/GB/90
UK, 1990	SC[1]	2	0.9	beans	62	<0.01	CTL/PHSSS 14/GB/90
UK, 1990	SC[1]	2	1.8	beans	62	0.02	CTL/PHSSS 14/GB/90
UK, 1991	SC	2	1.5	beans	49	0.02	CTL/PHSSS 15/GB/91
UK, 1991	SC	2	3.0	beans	49	0.03	CTL/PHSSS 15/GB/91
UK, 1991	SC[2]	2	1.5	beans	49	<0.01	CTL/PHSSS 15/GB/91
UK, 1991	SC[2]	2	3.0	beans	49	0.02	CTL/PHSSS 15/GB/91
UK, 1991	SC[1]	2	0.9	beans	49	0.02	CTL/PHSSS 15/GB/91
UK, 1991	SC[1]	2	1.8	beans	49	0.02	CTL/PHSSS 15/GB/91
UK, 1991	WG	2	1.5	beans	49	0.02	CTL/PHSSS 15/GB/91
UK, 1991	WG	2	3.0	beans	49	0.04	CTL/PHSSS 15/GB/91
UK, 1991	WG	2	1.5	beans	49	0.07	CTL/PHSSS 15/GB/91
UK, 1991	WG	2	3.0	beans	49	0.02	CTL/PHSSS 15/GB/91
UK, 1991	SC	2	1.5	beans	60	0.02	CTL/PHSSS 15/GB/91
UK, 1991	SC	2	3.0	beans	60	0.04	CTL/PHSSS 15/GB/91
UK, 1991	SC[2]	2	1.5	beans	60	0.02	CTL/PHSSS 15/GB/91
UK, 1991	SC[2]	2	3.0	beans	60	0.03	CTL/PHSSS 15/GB/91
UK, 1991	SC[1]	2	0.9	beans	60	<0.01	CTL/PHSSS 15/GB/91
UK, 1991	SC[1]	2	1.8	beans	60	<0.01	CTL/PHSSS 15/GB/91

Country, Year	Form.	Application No	kg ai/ha	Sample	PHI, days	Residues, mg/kg	Report
UK, 1991	WG	2	1.5	beans	60	0.02	CTL/PHSSS 15/GB/91
UK, 1991	WG	2	3.0	beans	60	0.03	CTL/PHSSS 15/GB/91
UK, 1991	WG	2	1.5	beans	60	<0.01	CTL/PHSSS 15/GB/91
UK, 1991	WG	2	3.0	beans	60	0.03	CTL/PHSSS 15/GB/91
UK, 1991	SC	2	1.5	beans	57	<0.01	CTL/PHSSS 15/GB/91
UK, 1991	SC	2	3.0	beans	57	<0.01	CTL/PHSSS 15/GB/91
UK, 1991	SC[2]	2	1.5	beans	57	<0.01	CTL/PHSSS 15/GB/91
UK, 1991	SC[2]	2	3.0	beans	57	<0.01	CTL/PHSSS 15/GB/91
UK, 1991	SC[1]	2	0.9	beans	57	<0.01	CTL/PHSSS 15/GB/91
UK, 1991	SC[1]	2	1.8	beans	57	<0.01	CTL/PHSSS 15/GB/91
UK, 1991	WG	2	1.5	beans	57	<0.01	CTL/PHSSS 15/GB/91
UK, 1991	WG	2	3.0	beans	57	<0.01	CTL/PHSSS 15/GB/91
UK, 1991	WG	2	1.5	beans	57	<0.01	CTL/PHSSS 15/GB/91
UK, 1991	WG	2	3.0	beans	57	<0.01	CTL/PHSSS 15/GB/91
UK, 1992	WG	2	1.5	beans	64	0.10	CTL/PHSSS 16/GB/92
UK, 1992	WG	2	3.0	beans	64	0.05	CTL/PHSSS 16/GB/92
UK, 1992	WG	2	1.5	beans	64	0.08	CTL/PHSSS 16/GB/92
UK, 1992	WG	2	3.0	beans	64	0.18	CTL/PHSSS 16/GB/92
UK, 1992	WG	2	1.5	beans	64	0.10	CTL/PHSSS 16/GB/92

Country, Year	Form.	Application No	kg ai/ha	Sample	PHI, days	Residues, mg/kg	Report
UK, 1992	WG	2	1.5	beans	71	0.06	CTL/PHSSS 16/GB/92
UK, 1992	WG	2	1.5	beans	64	0.10	CTL/PHSSS 16/GB/92
UK, 1992	WG	2	1.5	beans	64	0.04	CTL/PHSSS 16/GB/92
USA, Minneso., 1977	SC	6	1.2	beans	28	<0.04	463-3CR-81-0154-001
USA, Minneso., 1977	SC	6	1.8	beans	28	<0.04	463-3CR-81-0154-001
USA, Minneso., 1978	SC	3	2.3	beans	28	<0.04	463-3CR-81-0154-001
USA, N Dakota, 1978	SC	1	1.8	beans	47	<0.04	463-3CR-81-0154-001
USA, Minneso., 1979	SC	3	1.2	beans	40	<0.04	463-3CR-81-0154-001
USA, Minneso., 1979	SC	3	1.8	beans	40	<0.04	463-3CR-81-0154-001
USA, Minneso., 1979	SC	3	2.3	beans	40	0.05	463-3CR-81-0154-001
USA, Minneso., 1979	SC[3]	3	2.3	beans	40	<0.04	463-3CR-81-0154-001
USA, Illinois, 1980	SC	2	1.2	beans	43	0.04	463-3CR-81-0154-001
USA, Illinois, 1980	SC	2	1.8	beans	43	0.05	463-3CR-81-0154-001
USA, Illinois, 1980	SC	2	2.3	beans	43	0.11	463-3CR-81-0154-001
USA, Colorado, 1982	SC	3	1.2	Pinto beans	14	0.02	612-3CR-82-0181-001

Country, Year	Form.	Application No	kg ai/ha	Sample	PHI, days	Residues, mg/kg	Report
USA, Colorado, 1982	SC	3	1.8	Pinto beans	14	0.03	612-3CR-82-0181-001
USA, Delaware, 1982	SC	5 4	1.8 1.8	Lima beans	0 8	0.06 0.03	612-3CR-82-0181-001
USA, Nebraska, 1982	SC	4	1.8	Pinto beans	13	<0.01	612-3CR-82-0181-001
USA, N Dakota, 1982	SC	3	1.8	Pinto beans	22	0.02	612-3CR-82-0181-001
USA, Michigan, 1982	SC	5 4 3	1.8 1.8 1.8	beans	7 14 29	0.04 0.01 <0.01	612-3CR-82-0181-001
USA, Tenness., 1982	SC	4	1.8	Lima beans	9	0.03	612-3CR-82-0181-001

[1] treatment with Bravocarb (450 g chlorothalonil and 100 g carbendazim)
[2] treatment with Bravo 720 SC (720 g chlorothalonil)
[3] treatment with Bravo 500 (500 g chlorothalonil)

Celeriac (Table 16). A single residue study was carried out in The Netherlands. Four field samples were analysed for chlorothalonil only.

Table 16. Residues of chlorothalonil in celeriac, The Netherlands, 1977. Roots analysed. WP formulation.

Application No	kg ai/ha	PHI, days	Residues, mg/kg	Report
2	1.825	28	1.5, 1.9, 2.5, 2.8	KvW212/CvF/PD4.2.(2.1.11a)-1977

Wheat (Table 17). Two residue trials were reported from The Netherlands. The treatment was carried out at the beginning of blossoming. Four field samples were analysed for chlorothalonil only.

Table 17. Residues of chlorothalonil in wheat, The Netherlands, 1976. WP formulations.

Application No	kg ai/ha	Sample	PHI, days	Residues, mg/kg	Report
1	1.2	ears grain straw	41 41 41	0.21, 0.38, 0.44, 0.46 0.03(2), 0.04, 0.06 0.79, 1.5, 1.8(2)	KvW207/CvF/PD4.2.(1.1.05a)-1976-I

1	1.2	ears grain straw	41 41 41	0.64(2), 0.92, 0.94 0.04, 0.05(2), 0.12 2.8, 2.9, 3.8, 4.1	KvW207/CvF/PD4.2.(1.1. 05a)- 1976-II

Fresh herbs (Table 18). Four outdoor trials (1 on parsley, 2 on celery, 1 on celeriac) and two indoor trials (1 on parsley, 1 on celery) were reported by The Netherlands. Four field samples were analysed, for chlorothalonil only, in each trial.

Table 18. Residues of chlorothalonil in the leaves of fresh herbs.

Country, Year	Form.	Application No	kg ai/ha	Residues, mg/kg	PHI, days	Report
Celery, indoor						
Netherlands 1984	SC	4	1.875	6.7, 10.2, 11.7, 13	28	KvW275/CTB/PD3117.300/500. 311-1984
Celery, outdoor						
Netherlands 1985	SC	4	1.875	1.2, 1.8, 1.9, 2.0 0.06, 0.09(2), 0.13	14 28	KvW274/CTB/PD3116.300/500. 311-1985-I
Netherlands 1985	SC	4	1.875	8.1, 8.2, 10.3, 12.4 1.3, 1.6(2), 2.4	14 28	KvW274/CTB/PD3116.300/500. 311-1985-II
Celeriac, outdoor						
Netherlands 1976	WP	3	1.825	1.1, 1.6, 2.1, 2.3	28	KvW211/CvF/PD4.2(2.1.11a)- 1976
Parsley, indoor						
Netherlands 1984	SC	4	1.875	7.2, 8.8, 10.1, 16.1	28	KvW273/CTB/PD3105.300/500. 311-1984
Parsley, outdoor						
Netherlands 1984	SC	3	1.875	2.4, 2.5, 3.3, 4.8 0.3, 0.4, 1.4, 1.6	13 27	KvW272/CTB/PD3104.300/500. 311-1984

Animal transfer studies-cattle

Twenty dairy cows were randomly divided into groups of 4 and dosed by capsule for 28 days. A dose equivalent to 3 ppm chlorothalonil and 0.2 ppm SDS-3701 in the diet was taken to represent the potential level of residues in livestock feeds derived from chlorothalonil-treated crops, and the doses corresponded to 0, half, one, three and ten times this level. Milk samples were collected and daily composite samples from each cow were analysed for SDS-3701. It had previously been determined that chlorothalonil would not be a residue in meat or milk and that analyses for chlorothalonil would not be required (Wiedmann and Kenyon, 1995).

The residues in the milk reached a plateau after about 9 days. If the SDS-3701 in the dose was the only source of SDS-3701 transferring to the milk, the mean transfer rate was 25.2% (for days 10-28 and across all dose levels). If the SDS-3701 in the milk included both the SDS-3701 in the dose and that produced by the metabolism of chlorothalonil the rate of transfer was 1.7%. Both rates are consistent with previous studies of metabolism in goats (Duane and Doran, 1990; Han San Ku, 1990).

Separating milk into butterfat and skimmed milk did not show concentration of SDS-3701 into either fraction. The maximum residues in the milk and tissues found at day 28 are shown in Table 19.

Table 19. Maximum SDS-3701 residues (mg/kg) in the milk and tissues (Wiedmann and Kenyon, 1995).

Sample	SDS-3701, mg/kg			
	1/2 x dose	1x dose[1]	3x dose	10x dose
Milk	0.04	0.1	0.31	0.65
Muscle	<0.01	0.02	0.09	0.24
Fat, omental	0.03	0.07	0.08	0.85
Liver	0.03	0.04	0.18	0.55
Kidney	0.14	0.28	0.55	1.2

[1] Equivalent to 3 ppm chlorothalonil plus 0.2 ppm SDS-3701 in the diet

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

The Netherlands provided data on residues of chlorothalonil in food in commerce during 1995 (Table 20).

Table 20. Residues in food in commerce in The Netherlands (1995).

Commodity	Samples Analysed	Samples without residues (<0.01mg/kg)	Samples with residues < MRL	Samples with residues > MRL	Mean[1], mg/kg	Dutch MRL, mg/kg
Strawberries	1073	1068		5	<0.01	0.01
Carrots	209	205	4		0.01	0.5
Onions	32	31		1	0.28	0.5
Tomatoes	459	446	13		<0.01	2
Peppers	583	579	4		<0.01	2
Lettuce	900	895	2	3	<0.01	0.01
Iceberg lettuce	129	126		3	<0.01	
Endive	341	338	2	1	<0.01	
Parsley	131	127	4		0.07	5
Legume vegetables (fresh)	50	49		1	0.1	0.01
Celery	76	72	4		0.03	5

Commodity	Samples Analysed	Samples without residues (<0.01mg/kg)	Samples with residues < MRL	Samples with residues > MRL	Mean[1], mg/kg	Dutch MRL, mg/kg
Leek	190	185	5		<0.01	5
Mushrooms, cultivated	109	107	2		<0.01	1

[1]For samples without residues (<LOD), a level of half the LOD was taken for the calculation of the mean

NATIONAL MAXIMUM RESIDUE LIMITS

The national MRLs listed below were reported to the Meeting.

Definition of the residue: chlorothalonil

Country	Commodity	MRL, mg/kg
Australia	Almonds	0.1 (T)
	Apricot	7
	Banana	3
	Brussels sprouts	7
	Carrot	7
	Celery	10
	Cherries	10
	Fruiting vegetables, Cucurbits	5
	Grapes	10
	Nectarine	7
	Onion, Bulb	10
	Peach	30
	Peanut	0.2 (T)
	Plums (including Prunes)	10
	Potato	0.1
	Tomato	10
	Vegetables (except named above)	7 (T)
Brazil	Peppers	0.1
Canada	Celery	15
	Beans (snap)	5
	Broccoli	5
	Brussel sprouts	5
	Cabbage	5
	Cauliflower	5
	Cucumbers	5
	Melons	5
	Onions (green and dry bulb)	5
	Pumpkins	5
	Squash (summer and winter)	5
	Tomatoes	5
	Cranberry	2
	Carrots	1
	Parsnip	1
	Mushrooms	1
	Blueberries	0.6
	Cherries	0.5
	Peaches	0.5
	Nectarines	0.5
	Peanuts	0.3

Country	Commodity	MRL, mg/kg
	Chick peas	<0.1
	Potatoes	<0.1
	Strawberry	<0.1
	Sweet corn	<0.1
European Union	Apples, pears	1 (proposed)
	Apricots, Peaches	0.01
		0.3 (proposed)
	Asparagus	0.01 (proposed)
	Bananas	0.5 (proposed)
	Barley, oats	0.2 (proposed)
	Beans, peas (dry)	0.02
		1 (proposed)
	Blackberry	0.01 (proposed)
	Broccoli, cauliflower	0.01
		2 (proposed)
	Brussels sprouts	1 (proposed)
	Cabbage	3 (proposed)
	Carrots, radish, turnips	0.2 (proposed)
	Cherries	0.5 (proposed)
	Citrus fruits	0.1
		1 (proposed)
	Cranberries	5 (proposed)
	Cucumbers, courgettes, gherkins	1 (proposed)
	Currants	0.02
		2 (proposed)
	Garlic, onions, shallots, spring onions	0.5
	Grapes	
	Table grapes	1
	Wine grapes	2 (proposed)
	Leeks	10
	Legume vegetables	2 (proposed)
	Melons	2 (proposed)
	Mushrooms	7 (proposed)
European Union	Peanuts	0.3 (proposed)
	Plums	0.2 (proposed)
	Potatoes	0.1 (proposed)
	Strawberries	1.5 (proposed)
	Sugar beets	0.2 (proposed)
	Sugar beet leaves	2 (proposed)
	Tomatoes, aubergines	2
	Wheat, rye	0.1 (proposed)
Germany	Table grapes	1
	Cranberries	2
	Garlic	0.5
	Onion	0.55
	Shallots	0.5
	Peas with pods (fresh)	2
	Solanacea	2
	Cucumbers	1
	Brussels sprouts	0.5
	Barley	0.1
	Oats	0.1
	Rye	0.1
	Triticale	0.1
	Wheat	0.1

Country	Commodity	MRL, mg/kg
	Tea	0.1
	Other commodities plant origin	0.01
Italy	Peach	0.3
Netherlands	Table grapes	1
	Cranberries	2
	Carrots	0.5
	Celeriac	0.5
	Bulb vegetables	0.5
	Solanacea (tomatoes, peppers, aubergines)	2
	Cucumbers	1
	Gherkins	5
	Courgettes	1
	Cucurbitaceae with inedible peel (melons, squashes, watermelons)	1
	Brussels sprouts	0.5
	Parsley	5
	Celery leaves	5
	Peas (with pods)	2
	Celery	5
	Leek	5
	Mushrooms (other than wild)	1
	Tea	0.1*
	Hops	0.1*
	Wheat, rye, barley, oats, triticale	0.1
	Other food commodities	0.01*
Spain	Citrus fruits	0.1
	Peach	0.5
UK	Broccoli	1
	Bean (dry)	0.01
USA	Banana (whole fruit)	0.5
	Banana (edible pulp)	0.05
	Bean (dry)	0.1
	Bean (snap)	5
	Celery	15
	Papaya	15
	Broccoli	5
	Brussel sprouts	5
	Cabbage	5
	Cauliflower	5
	Cucumbers	5
	Cranberry	5
	Melons	5
	Onions (green)	5
	Pumpkins	5
	Squash (summer and winter)	5
	Tomatoes	5
	Passion fruit	3
	Mint (hay)	2
	Blueberries	1
	Mushrooms	1
	Parsnip	1
	Carrots	1
	Sweet corn	1
	Banana	0.5

Country	Commodity	MRL, mg/kg
	Banana, edible pulp	0.05
	Onion (dry pulp)	0.5
	Soya bean	0.5
	Cherry	0.5
	Peach	0.5
	Nectarine	0.5
	Apricot	0.5
	Plum	0.2
	Prune	0.2
	Coffee beans	0.2
	Peanuts	0.3
	Filberts	0.1
	Potatoes	0.1
	Cocoa beans	0.05

APPRAISAL

Chlorothalonil is a non-systemic protectant fungicide. It was first evaluated for residues in 1974 and has been reviewed several times since, most recently as a periodic review in 1993. The 1993 JMPR required additional residue data from supervised trials on different types of melons, residue data on grapes treated according to GAP in Australia and animal transfer studies.

At the 27th (1995) Session of the CCPR the manufacturers indicated that they would provide information on GAP and residue data to the 1997 JMPR for some crops. The representative of the EU was invited to submit residue trials data and information on GAP for the use of chlorothalonil on tomatoes to the JMPR, to support extrapolation and to establish an MRL for peppers (ALINORM 95/24A, paras 107-111). The 1996 CCPR was informed that additional data would be provided for peaches, and decided to keep the MRL for peach at Step 7B.

The fate of residues has been studied with [^{14}C]chlorothalonil in lactating goats, laying hens and *in vitro* in bovine tissues.

Lactating goats. In goats dosed at a level equivalent to 3 ppm in the daily diet, the total radioactive residue (the TRR, calculated as chlorothalonil equivalents) in the milk and meat were extremely low with residues of 0.009 mg/kg in the milk and 0.004 mg/kg in the meat. The organs with the highest TRR were the liver and kidney which averaged 0.08 mg/kg and 0.22 mg/kg respectively, the residues being complex mixtures. The 4-hydroxy metabolite, SDS-3701 (4-hydroxy-2,5,6-trichloroisophthalonitrile) was identified in the milk, liver and kidney. The metabolite was quantified in a group at 30 ppm at levels up to 0.05 mg/kg in the milk and liver and 0.08 mg/kg in the kidneys. The other major components of the residue that could be characterized were conjugates of chlorothalonil with glutathione. There were no detectable residues of the parent compound in the milk or tissues.

In similar metabolism and transfer studies with SDS-3701 this compound was the only terminal residue. After doses equivalent to 0.2 ppm it was found in muscle and fat at 0.01 to 0.02 mg/kg, in heart at 0.04 to 0.05 mg/kg, in liver at 0.07 mg/kg, in the milk at 0.09 to 0.15 mg/kg and in kidney at 0.17 to 0.26 mg/kg.

Poultry. Laying hens were dosed once daily at levels equivalent to 2, 6 or 20 ppm of chlorothalonil in the diet for 21 days. The TRR was calculated as chlorothalonil equivalents. No radioactivity (<0.04 mg/kg) was detectable in egg whites at the 2 or 6 ppm levels at any sampling interval. The high-dose yolks showed a maximum total radioactivity of 0.047 mg/kg from day 13 of dosing. Since no activity was detectable in the egg whites, the residues in whole eggs would be #50% of those in the yolks. Analysis of the tissues revealed the only detectable TRR to be present in the liver. The maximum TRR of 0.098 mg/kg was present in the livers of the mid-dose group within 6 hours after the final dose (2 ppm dose <0.04 mg/kg; 20 ppm dose 0.05 mg/kg).

Similar metabolism and transfer studies were conducted with SDS-3701 at dose levels equivalent to 0.1, 0.3 and 1 ppm. The TRR were calculated as SDS-3701 equivalents. No radioactivity (<0.04 mg/kg as SDS-3701) was detectable in egg whites at any dose level. In egg yolks the TRR in the low-dose group reached a plateau at approximately 0.04 mg/kg on day 21. The TRR in the mid- and high-dose yolks reached plateaux of 0.12 mg/kg at day 21 and 0.42 mg/kg at day 16 respectively. The residue in the egg yolks was shown to be unchanged SDS-3701. No activity was detectable in the fat or cardiac tissue of the low-dose group. The cardiac tissue from the mid- and high-dose groups showed maximum activities of 0.055 mg/kg and 0.15 mg/kg. The low-dose livers contained maximum residues of 0.06 mg/kg within 6 hours after the final dose. The highest TRR levels in the mid- and high-dose livers were 0.27 and 0.78 mg/kg respectively.

Studies of *in vitro* reactions of chlorothalonil with ruminant tissue systems as well as freezer storage stability studies with meat tissues and milk demonstrated that chlorothalonil was not stable in these substrates. It reacts extremely rapidly with components of bovine tissue homogenates with a maximum half-life of 1 minute, giving rise to polar metabolites and bound residues.

A multi-residue analytical method is used for the determination of chlorothalonil in fatty and non-fatty foods by gas chromatography with electron-capture or ion trap detection, with an LOD of 0.01 mg/kg and recoveries of 89-104%.

Chlorothalonil residues are lost quite rapidly at room temperature during such sample preparation as the comminution of fruits and vegetables (e.g. 95% loss from lettuce and 80% from broccoli), but subsequent losses were minimal during storage in the freezer. The losses have important implications, as analytical results could seriously underestimate chlorothalonil residues. The Meeting wishes to draw the attention of enforcement and monitoring laboratories to the need for sample preparation to be carried out under frozen conditions and followed by immediate extraction. The manufacturer confirmed that the data on residues in the samples from supervised trials evaluated by the present Meeting were valid because the samples were kept frozen throughout sample preparation.

Definition of the residue for animal products. Because the metabolite SDS-3701 is considered to be of toxicological importance, the Meeting recommended its inclusion in the definition of the residue for the risk assessment of residues in products of animal origin.

Definition of the residue in animal products for compliance with MRLs: chlorothalonil.

Definition of the residue in animal products for risk assessment: sum of chlorothalonil and 4-hydroxy-2,5,6-trichloroisophthalonitrile, expressed as chlorothalonil.

Chlorothalonil is not fat-soluble (log P_{OW} = 2.87).

Supervised residue trials gave the following results.

Citrus fruits. The use of chlorothalonil is registered in Spain (2 x 1.25 kg ai/ha, PHI 28 days). Whole fruits were analysed in six Spanish trials (one on mandarins, five on oranges). After two applications of 1.25 kg ai/ha the residues of chlorothalonil at 26-28 days ranged from 0.26 to 1.9 mg/kg. No information was received on residues in the pulp.

The Meeting concluded that the residue data were insufficient to estimate a maximum residue level for a major crop and confirmed the recommendation of the 1993 JMPR to withdraw the CXL.

Peaches. Chlorothalonil is registered in Italy and Spain (4 x 1.5 kg ai/ha). The Italian PHI is 14 days, and in Spain the last treatment should be not later than nut size of the fruit (PHI about 60 days).

Six residue trials were carried out in Italy and Spain at the GAP application rate (4 x 1.5 kg ai/ha), but the PHI was three weeks. The residues ranged from 0.54 to 1.4 mg/kg.

In six Italian trials with 3 applications of 1.25-1.5 kg ai/ha, the last with the fruit at nut size (PHI 64 or 66 days) the residues were <0.01 (5) and 0.04 mg/kg, and four Spanish trials (3 or 4 x 1.25-1.5 kg ai/ha) showed residues of <0.01 (82 days), 0.01 (69 days), 0.03 (87 days) and 0.15 (87 days) mg/kg. As one of the results at 87 days is higher than the Italian residues at 66 days, all these results should be included in the assessment. All the residues in the ten trials carried out in Italy and Spain (with PHIs of 64, 66, 69, 82 and 87 days) in rank order were <0.01 (6), 0.01, 0.03, 0.04 and 0.15 mg/kg.

The JMPR was informed that the reported residues were in the fruit without stone, not calculated for the whole commodity, and that the pulp represented 95% of the total weight. The Meeting concluded that a reduction in the residue values by 5% was not significant and did not recalculate the results.

The Meeting estimated a supervised trials median residue level of 0.01 mg/kg, and a maximum residue level of 0.2 mg/kg, on the basis Spanish GAP, to replace the draft MRL for peach (1 mg/kg) recommended by the 1993 JMPR.

Grapes. The 1993 JMPR listed as desirable additional residue data on grapes treated according to GAP in Australia (multiple treatments of 1.3-1.65 kg ai/ha, 0.12-0.15 kg ai/hl). The PHIs are 7 days for table grapes and 14 days for wine grapes.

Two trials according to GAP were reported to the 1983 and 1993 Meetings. In the first trial (7 x 0.11 kg ai/hl) residues were 8.6 mg/kg after 10 days. In the second (6 x 0.13 kg ai/hl) they were 0.6 mg/kg after 7 days and 2.9 mg/kg after 18 days.

In the five Australian trials reported to the current Meeting, grapes were treated 1-4 times at rates of 1.9-4.6 kg ai/ha. In two of them, residues of chlorothalonil were 4.8 and 5.2 mg/kg in two samples taken 7 days after a single treatment of 1.9-2.25 kg ai/ha (0.125-0.15 kg ai/hl). In the other trials, samples were taken from 60 to 96 days after the last treatment. Thus the trials were with fewer treatments or longer PHIs than the recommended GAP.

The Meeting agreed that the Australian residue data suggest the need for a higher MRL, but the data were not sufficient to support a recommendation to replace the current CXL (0.5 mg/kg).

Blackberries. Chlorothalonil is registered in the UK (4 x 2.5 kg ai/ha, 28-day PHI). One trial on blackberries in Sweden at the lower rate of 1 x 1.25 kg ai/ha was reported. No residues higher than the LOD of 0.01 mg/kg were found 7-28 days after treatment.

The Meeting noted that insufficient data were submitted and could not estimate a maximum residue level. The recommendation of the 1993 JMPR to withdraw the CXL was confirmed.

Currants. Chlorothalonil is registered in the UK (4 x 2.5 kg ai/ha, 28-day PHI). Six trials on black currants in the UK with 3 x 2.5 kg ai/ha, PHI 28 days, were reported. The chlorothalonil residues in rank order were 0.83, 0.94, 1.5, 1.9, 3.3 and 3.8 mg/kg.

The Meeting agreed to extrapolate from black to white and red currants and estimated a supervised trials median residue level of 1.7 mg/kg and a maximum residue level of 5 mg/kg for black, red and white currants.

Bananas. Registered uses exist with multiple treatments and PHIs of 1 or 0 days in Australia (1.1-2.16 kg ai/ha) and Latin America (aerial application, 0.88-1.63 kg ai/ha).

Two Australian trials on unbagged bananas reported to the 1993 JMPR were according to Australian GAP (10 x 1.1 or 2.2 kg ai/ha, 1-day PHI) and resulted in residues of 0.6 and 2.0 mg/kg.

In three of the four Latin American trials evaluated by the 1993 JMPR the residues were below 0.01 mg/kg; it was not stated whether the bananas were bagged or unbagged. In the fourth trial on unbagged fruit carried out in Costa Rica in 1985 (10 x 1.75 kg ai/ha, aerial application) the maximum residue in 6 field samples was 0.12 mg/kg 6 days after treatment.

Six Latin American supervised trials carried out in 1993 according to GAP (10-15 x 1.7 kg ai/ha, aerial application) were reported to the present Meeting. Samples of bagged bananas taken on the day of treatment showed residues below the LOD (<0.01 mg/kg).

On the basis of the residues in bagged bananas, the Meeting estimated an STMR of 0 and a maximum residue level of 0.01* mg/kg as a practical limit of determination.

Broccoli. Chlorothalonil is registered in the UK (2 x 1.5 kg ai/ha, 7-day PHI) and in the USA (1.7 kg ai/ha, 7-day PHI, number of treatments not specified). The Meeting re-evaluated the two US residue trials according to GAP reported to the 1993 JMPR (4 or 8 x 1.3 kg ai/ha, PHI 7 days) and reviewed two new trials (2 x 1.5 kg ai/ha, PHI 7 days).

The residues from the four trials show a median value of 2.25 mg/kg (rank order 1.5, 2.2, 2.3 and 2.6 mg/kg).

The Meeting estimated a supervised trials median residue level of 2.25 mg/kg and a maximum residue level of 5 mg/kg for broccoli.

Gherkins. The residues in four plot samples from one indoor Dutch trial were 0.64-1.1 mg/kg (median 0.78 mg/kg) three days after one treatment with 2.2 kg ai/ha.

As there were too few treatments to comply with Dutch GAP, which specifies 3-5 applications of 0.75-2.25 kg ai/ha, the Meeting could not estimate a maximum residue level.

Peppers. In response to a referral from the 1995 CCPR, the Meeting agreed that an extrapolation from tomatoes to peppers was inappropriate because of the large difference in the surface-to-weight ratio.

Chlorothalonil is registered in Australia, where multiple treatments of 1.3-1.65 kg ai/ha with a PHI of one day are recommended. In Latin America, multiple treatments of 1.8 kg ai/ha and a PHI of seven days are registered.

A total of 15 residue trials were carried out on bell peppers. Eight trials were conducted in Australia with 6 to 8 applications at 1.65-3.3 kg ai/ha, but samples were taken at the 1-day PHI in only two of them. The residues of chlorothalonil one day after treatment with 1.65 kg ai/ha were 0.43 and 5.3 mg/kg. Residues of 0.04 mg/kg were found in one Brazilian trial (3 x 1.75 kg ai/ha) 7 days after treatment. The residues in five trials carried out in 1996 (7-12 x 1.74-1.92 kg ai/ha) in Mexico, Honduras, Chile and Costa Rica 7 days after treatment were 0.05, 1.4, 1.6, 4.1 and 5.4 mg/kg. These were of the same order as the Australian residues and support the conclusion that a maximum residue level higher than 5 mg/kg is appropriate. All the results in rank order were 0.04, 0.05, 0.43, 1.4, 1.6, 4.1, 5.3 and 5.4 mg/kg (median 1.5 mg/kg).

The Meeting estimated a supervised trials median residue level of 1.5 mg/kg and a maximum residue level of 7 mg/kg for sweet peppers.

Mushrooms. Results of four field trials and one indoor trial reflecting Dutch GAP for cultivated mushrooms were reported by The Netherlands. The maximum residue was 0.78 mg/kg seven days after two treatments with 22 kg ai/ha.

The data were insufficient to estimate a maximum residue level.

Sweet corn (corn-on-the-cob). Registered uses of chlorothalonil exist in Australia (multiple treatments, 1.3-1.65 kg ai/ha, 1-day PHI) and the USA (multiple ground or aerial treatments, 0.7-1.6 kg ai/ha, 14-day PHI).

Four trials were carried out in the USA with 8 x 1.3 kg ai/ha. No residues above the LOD of 0.01 mg/kg were found in the cobs or the grain 14 days after treatment. Forage samples from three of the trials showed residues from 8.2 to 58 mg/kg at day 14. The difference between the residue levels in the cobs and the forage shows that surface residues of chlorothalonil would not be expected to translocate into the grain.

The Meeting estimated a supervised trials median residue level of 0.01 mg/kg and a maximum residue level of 0.01* mg/kg as a practical limit of determination.

Beans (dry). Chlorothalonil is registered in the UK with 2 x 1.5 kg ai/ha, and in the USA with multiple treatments of 1.2-1.75 kg ai/ha. The last treatment should be at end of flowering.

Residues from 24 trials with treatments near UK GAP (2 x 1.5 -1.8 kg ai/ha) at 49-71 days after treatment ranged from <0.01 to 0.1 mg/kg.

Chlorothalonil residues in trials according to US GAP (2-6 x 1.2-1.8 kg ai/ha) were <0.04 (2), 0.04 and 0.05 mg/kg at 40 to 43 days after treatment.

Combining the UK and US data gave residues in rank order of <0.01 (10), 0.02 (7), <0.04 (2), 0.04 (2), 0.05, 0.06, 0.07, 0.08 and 0.1 (3) mg/kg. The Meeting estimated a supervised trials median residue level of 0.02 mg/kg.

The Meeting also estimated a maximum residue level of 0.2 mg/kg for beans (dry), and confirmed the recommendation of the 1993 JMPR to withdraw the CXL for lima bean (dry).

Celeriac. A single trial in The Netherlands approximated Dutch GAP of 3-5 x 1.88 kg ai/ha, PHI 28 days. The maximum residue in four field samples was 2.8 mg/kg 28 days after two treatments with 1.8 kg ai/ha.

The data were insufficient to estimate a maximum residue level.

Wheat. Four field samples were taken in each of two trials in The Netherlands at 1 x 1.2 kg ai/ha with a 41-day PHI which approximated Dutch GAP of one treatment at 1 kg ai/ha and a PHI of 42 days.

The residues in the straw ranged from 0.03 to 4.1 mg/kg. No change of the current CXL of 20 mg/kg is proposed. The highest residue in the grain was 0.12 mg/kg. The Meeting agreed that the data suggested that a higher MRL than the current CXL of 0.1 mg/kg was needed, but the two trials were not sufficient to support a new recommendation.

Fresh herbs. Chlorothalonil is registered for outdoor use in the Netherlands an parsley and celery leaves (3-5 x 1.87 kg ai/ha, 28-day PHI). One trial on parsley, one on celeriac leaves and two on celery leaves (3-4 x 1.8-1.9 kg ai/ha, PHI 27-28 days) were reported. The maximum residues of the four replicates from each trial in rank order were 0.13, 1.6, 2.3 and 2.4 mg/kg.

The Meeting estimated supervised trials median residue levels of 1.95 mg/kg and maximum residue levels of 3 mg/kg for parsley and celery leaves (fresh).

Determination of metabolites and impurities in plants. Samples of selected crops were analysed for the metabolite 4-hydroxy-2,5,6-trichloroisophthalonitrile (SDS-3701), and the technical impurities hexachlorobenzene (HCB) and pentachlorobenzonitrile (PCBN). In sweet peppers the highest residue of SDS-3701 was 0.04 mg/kg. SDS-3701 and HCB residues in bananas and sweet corn cobs were below the LODs of 0.01 and 0.00025 mg/kg respectively. SDS-3701, HCB and PCBN were not detected in dry beans (<0.03, <0.004 and <0.01 mg/kg respectively).

Animal products. Animal metabolism and transfer studies with [^{14}C]chlorothalonil on lactating goats and laying hens showed very little or no transfer of the pesticide from animal feed to milk, fat, tissues or eggs. Chlorothalonil *per se* absorbed from the gastrointestinal tract would be very short-lived and could not be transmitted as a residue to food items such as meat, liver, milk or edible offal.

Animal transfer studies on cattle were carried out for 28 days at levels of 1.5 ppm chlorothalonil plus 0.1 ppm SDS-3701, 3 ppm chlorothalonil plus 0.2 ppm SDS-3701, 9 ppm chlorothalonil plus 0.6 ppm SDS-3701 and 30 ppm chlorothalonil plus 2 ppm SDS-3701, to represent potential dietary levels of residues in livestock feeds. The median residue levels of chlorothalonil in such feed items as sugar beet and cereal straw found in supervised trials reported to the 1993 JMPR demonstrate that a level of 3 ppm chlorothalonil plus 0.2 ppm SDS-3701 should be realistic for residues in potential feed items and appropriate for estimating the transfer of chlorothalonil to animal products. The residues of the metabolite SDS-3701 were 0.1 mg/kg in the milk (reaching a plateau after day 9), 0.02 mg/kg in muscle, 0.04 mg/kg in liver and 0.28 mg/kg in kidney at the end of the study (day 28).

Since the full details of the studies were not reported, the Meeting could not estimate maximum residue levels for animal products.

Data on residues of chlorothalonil in foods in commerce in 1995 were reported from The Netherlands. Of 4282 samples analysed, 4228 (98.7%) were without residues (<0.01 mg/kg). Residues above the Dutch MRLs were found in 14 samples (0.33 %).

RECOMMENDATIONS

On the basis of the available data on residues resulting from supervised trials the Meeting estimated the maximum residue and STMR levels listed below. The maximum residue levels are recommended for use as MRLs.

Definition of the residue

for compliance with MRLs and for the estimation of dietary intake for plant commodities: chlorothalonil.

for the estimation of dietary intake for animal products: sum of chlorothalonil and 4-hydroxy-2,5,6-trichloroisophthalonitrile (SDS-3701), expressed as chlorothalonil.

Commodity		Recommended MRL, mg/kg		Estimated STMR, mg/kg	PHI on which based, days
CCN	Name	New	Previous		
FI 0327	Banana	0.01*[1]	W[2]	0	0
VD 0071	Beans (dry)	0.2	-	0.02	40-71
VB 0400	Broccoli	5	W[2]	2.25	7
HH 0624	Celery leaves	3	-	1.95	27-28
FB 0021	Currants (Black, Red and White)	5	W[2]	1.7	26- 28
HH 0740	Parsley	3	-	1.95	27-28
FS 0247	Peach	0.2	1	0.01	64-87
VO 0051	Peppers, Sweet	7	W[2]	1.5	7
VO 0447	Sweet corn (corn on the cob)	0.01*	W[2]	0.01	14

[1]Based on trials with bagged bananas
[2]Withdrawal of existing MRL or CXL was recommended by 1993 JMPR

Note changed definition of residue for STMRs for animal products

FURTHER WORK OR INFORMATION

Desirable

1. Additional residue data on table grapes and sweet corn treated according to GAP in Australia.

2. Additional residue data from supervised trials on different types of melons (from 1993).

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CLETHODIM (187)

EXPLANATION

Clethodim was first evaluated by the 1994 JMPR which recommended a number of MRLs. At the 1996 CCPR the governments of Germany and The Netherlands provided detailed comments on the 1994 monographs which they considered to be unclear and over-summarized. Reservations were expressed because there were no quantitative data on the metabolites in plants and none on the nature or quantities of individual metabolites in goats. The limit of determination of 0.05*mg/kg was questioned and it was pointed out that the method of analysis could not distinguish between sethoxydim and clethodim. A number of individual recommendations for MRLs were also questioned.

The manufacturer has provided an item-by-item response to the comments of Germany and The Netherlands, together with some comments on other points raised at the 1996 CCPR (Tomen Agro 1996).

A number of new studies submitted to the present Meeting were not reviewed because the data were not identified in the 1994 evaluation as being either required or desirable and they do not help to answer the questions raised by member governments at the 1996 CCPR. However, all the new data on residue trials have been reviewed to allow the estimation of additional maximum residue levels.

Animal metabolism

Goats. In a metabolism study reviewed by the 1994 Meeting, a lactating goat was dosed with [*propyl*-1-^{14}C]clethodim at 14.2 mg/dose 3 times a day for 3 days, and once on the fourth day. A control goat received the same number of empty gelatine capsules. Milk was collected twice daily and excreta daily. The goat was slaughtered about 4 hours after the last dose and samples of muscle, fat, liver, kidneys, heart and blood were collected (Rose and Suzuki, 1988).

Ninety one per cent of the administered radioactivity was found in the urine and faeces with the milk containing 0.02-0.05mg/kg clethodim equivalents (about 0.14% of the administered ^{14}C). The highest tissue concentrations were found in the liver (0.414 mg/kg clethodim equivalents) and kidneys (0.378 mg/kg). The extraction scheme for the milk, blood and tissues resulted in a distribution of radioactivity into hexane, acetonitrile and methanol or methanol/water. In blood and all the tissues except peritoneal fat (in which the radioactive residues were very low), 62-86% of the radioactivity in the samples was found in the acetonitrile extract. In milk 30-66% of the radioactivity was unextractable, although again most of the extractable radioactivity (10-33% of the radioactivity in the milk) was associated with the acetonitrile fraction.

Metabolites were identified only in those extracts which contained a relatively high percentage of the radioactivity. The hexane fraction which, excepting the heart and milk extracts, contained less than 4% of the substrate radioactivity, was not examined. Tables 1 and 2 show the distribution and quantification of the major metabolites in the milk, blood and tissues.

Table 1. Quantification of metabolites in milk of a lactating goat treated with [*propyl*-1-^{14}C] clethodim for 4 days.

Compound	% of radioactivity and (mg/kg clethodim equivalents)							
	Day 1		Day 2		Day 3		Day 4	
	a.m.[1]	p.m.	a.m.	p.m.	a.m.	p.m.	a.m.	Slaughter
Clethodim	0.0 (0.00)	0.0 (0.00)	0.0 (0.00)	0.0 (0.00)	0.0 (0.00)	0.0 (0.00)	3.3 (0.001)	0.0 (0.00)
Clethodim sulfoxide	0.0 (0.00)	29.4 (0.006)	19.2 (0.005)	20.2 (0.007)	18.0 (0.006)	14.7 (0.005)	17.7 (0.006)	27.0 (0.013)
S-methyl sulfoxide	0.0 (0.00)	0.0 (0.00)	6.9 (0.002)	5.5 (0.002)	0.0 (0.00)	4.3 (0.001)	5.7 (0.002)	11.1 (0.005)
Lactose	0.0 (0.00)	0.0 (0.00)	54.1 (0.014)	42.0 (0.014)	44.4 (0.014)	49.4 (0.017)	43.4 (0.016)	29.8 (0.015)

[1]Levels too low for identification

Table 2. Quantification of metabolites in blood and tissues of a lactating goat treated with [*propyl*-1-^{14}C]clethodim for 4 days

Compound	% of radioactivity and (mg/kg clethodim equivalents)						
	Blood	Liver	Kidneys	Heart	Forequarter muscle	Hindquarter muscle	Subcutaneous fat
Clethodim	28.0 (0.047)	27.6 (0.114)	1.3 (0.005)	0.0 (0.000)	0.0 (0.000)	0.0 (0.000)	2.8 (0.002)
Clethodim sulfoxide	39.9 (0.067)	33.2 (0.137)	36.9 (0.139)	43.2 (0.025)	51.6 (0.017)	40. (0.014)	47.2 (0.037)
Clethodim sulfone	3.8 (0.006)	3.2 (0.013)	1.0 (0.000)	0.0 (0.000)	0.0 (0.000)	0.0 (0.000)	0.0 (0.000)
S-methyl sulfoxide	11.6 (0.019)	6.2 (0.025)	30.8 (0.116)	37.2 (0.021)	28.5 (0.009)	32.4 (0.011)	29.0 (0.023)
Imine sulfoxide	3.0 (0.005)	1.5 (0.006)	4.1 (0.016)	0.0 (0.000)	0.0 (0.000)	0.0 (0.000)	4.7 (0.004)
5-hydroxy sulfone	2.7 (0.004)	0.0 (0.000)	0.0 (0.000)	0.0 (0.000)	0.0 (0.000)	0.0 (0.000)	0.0 (0.000)
Unidentified	3.2 (0.005)	4.0 (0.016)	4.0 (0.016)	0.0 (0.000)	0.0 (0.000)	7.9 (0.003)	8.1 (0.006)

Plant metabolism

The metabolism of [*cyclohexene*-4,6-^{14}C]clethodim and [*chloroallyl*-2-^{14}C]clethodim was studied on carrots, soya beans and cotton plants which were treated twice at 14-day intervals at an application rate of 0.29 kg ai/ha. The crops were harvested at maturity with PHIs of 20 to 70 days (Chen, 1988). Most of the radiocarbon was found in the leaves and edible parts. The metabolites were characterized and quantified by autoradiography and confirmed by LC-MS. The identified metabolites were clethodim sulfoxide, clethodim sulfone, the imine sulfoxide, imine sulfone, 5-hydroxy sulfoxide and 5-hydroxy sulfone. The results obtained with the two labels are shown in Tables 3 and 4. This study also was reviewed by the 1994 Meeting.

Table 3. Distribution and characterization of ^{14}C in edible parts and leaves of carrots, soya beans and cotton treated with [*cyclohexene*-4,6-^{14}C]clethodim.

Compound	^{14}C , % of TRR and (mg/kg clethodim equivalents)					
	Carrots		Soya beans		Cotton	
	Leaves	Roots	Leaves	Beans	Leaves	Seed
Clethodim		0.8 (0.003)				
Clethodim	15.7 (3.50)	28.6 (0.11)	5.9 (1.65)	32.0 (1.24)	4.1 (0.55)	4.3 (0.0029)

Compound	^{14}C , % of TRR and (mg/kg clethodim equivalents)					
	Carrots		Soya beans		Cotton	
	Leaves	Roots	Leaves	Beans	Leaves	Seed
sulfoxide						
Clethodim sulfone	0.6 (0.13)	3.4 (0.014)	0.9 (0.25)	4.6 (0.178)	0.4 (0.054)	2.8 (0.0019)
Imine sulfoxide	22.1 (4.93)	9.9 (0.04)	13.9 (3.88)	7.8 (0.302)	17.8 (2.40)	6.0 (0.0041)
Imine sulfone	5.9 (1.32)	8.6 (0.034)	8.7 (2.43)	8.1 (0.314)	4.1 (0.55)	2.3 (0.0016)
5-hydroxy-sulfoxide	1.6 (0.36)	6.4 (0.026)	<0.1	7.1 (0.275)	1.4 (0.19)	0.6 (0.0004)
5-hydroxy-sulfone	1.9 (0.42	7.6 (0.030)	3.1 (0.86)	10.7 (0.414)	0.4 (0.054)	1.6 (0.0011)

Table 3. Distribution and characterization of ^{14}C in edible parts and leaves of carrots, soya beans and cotton treated with [*chloroallyl*-4,6-^{14}C]clethodim.

Compound	^{14}C , % of TRR and (mg/kg clethodim equivalents)					
	Carrots		Soya beans		Cotton	
	Leaves	Roots	Leaves	Beans	Leaves	Seed
Clethodim		1.1 (0.007)				
Clethodim sulfoxide	10.5 (0.97)	33.9 (0.210)	4.5 (0.79)	31.5 (1.34)	5.3 (0.35)	3.1 (0.007)
Clethodim sulfone	1.8 (0.17)	4.6 (0.029)	0.9 (0.16)	5.1 (0.217)	1.8 (0.12)	0.4 (0.001)
Imine sulfoxide	------	------	------	------	------	------
Imine sulfone	------	------	------	------	------	------
5-hydroxy-sulfoxide	1.0 (0.09)	7.3 (0.045)	1.4 (0.25)	4.0 (0.17)	1.1 (0.07)	0.4 (0.001)
5-hydroxy-sulfone	1.7 (0.16)	10.1 (0.063)	2.2 (0.39)	10.1 (0.429)	0.6 (0.04)	0.6 (0.001)

METHODS OF RESIDUE ANALYSIS

In a modified confirmatory method for crops, animal tissues, milk and eggs, samples are extracted with methanol or methanol/water and cleaned up by an alkaline precipitation. After partitioning with methylene chloride, the residue is methylated with diazomethane, oxidised with *m*-chloroperbenzoic acid, and cleaned up by silica column chromatography. The methylated sulfones are determined by HPLC on a C-18 column with UV detection. The method is described as suitable for the determination of clethodim sulfoxide, clethodim sulfone, 5-hydroxyclethodim sulfoxide and 5-hydroxyclethodim sulfone, and as “being specific for the metabolites of clethodim and suitable for distinguishing clethodim residues from other similar herbicides (i.e. sethoxydim)”. It is a modified version of the method reviewed in the 1994 monograph (Lai and Ho, 1990). Chromatograms were submitted which showed the separation of methylated 5-hydroxyclethodim sulfone and methylated clethodim sulfone from methylated 5-hydroxysethoxydim and methylated sethoxydim sulfone (Lai and Fujie 1993).

A *diazomethane use justification* was submitted for the unmodified version of this confirmatory method. Methylation was stated to be necessary because compounds containing the 3-hydroxy-2-cyclohexene-1-one moiety cannot be chromatographed under the described conditions. Experiments with the alternative methylating agents dimethyl sulfate and methyl iodide were reported not to give high or reproducible yields of the required enol methyl ether, although further experimental details were not submitted (Rose 1990).

Recovery experiments were carried out with the modified confirmatory method on soya beans fortified at 0.05, 1.0 and 5.0 mg/kg with both clethodim sulfoxide and 5-hydroxyclethodim sulfoxide, liver fortified at 0.2 mg/kg with clethodim sulfoxide, and milk fortified at 0.02 and 0.05 mg/kg with clethodim sulfoxide and *S*-methyl-clethodim sulfoxide. Sethoxydim sulfoxide was also included in some of the experiments. Acceptable recoveries were obtained from soya beans (69-105%) and liver (86-92%) but the mean recoveries from milk were all very low (28-47%). The report stated that the "possible reason for the poor recoveries may be the presence of oil/fat in the sample extracts which may reduce the partition effectiveness of the HPLC mobile phase….". The method was described as being capable of differentiating the clethodim residues from those of the structurally similar sethoxydim, but no chromatograms were provided (Rhoades, 1993).

Another study provided further validation data for the modified confirmatory method at fortification levels of 0.02 and 0.05 mg/kg in milk and 0.2 mg/kg in liver. The report stated that several problems had been encountered with the first attempts to analyse milk and liver, but acceptable recoveries were obtained on making minor changes to the method, with recoveries of 58-85% (mean 70%, SD 12%) and 95-110% (mean 104%, SD 8%) of clethodim sulfoxide from milk and liver respectively. Recoveries of *S*-methyl-clethodim sulfone from milk were 64-120% (mean 93%, SD 28%) after correction for the mean area of interference present in the controls. Experiments with soya beans gave recoveries of clethodim sulfoxide of 300 and 320, 102 and 110 and 62 and 72% at fortification levels of 0.05, 1.0 and 5.0 mg/kg. However recoveries of 60 and 68 % of clethodim sulfoxide and 50 and 82% of 5-hydroxy-clethodim sulfoxide were obtained from soya beans fortified at 0.05 mg/kg when minor modifications were made to the method (Crawford and Dillon, 1994).

Several further studies of the modified confirmatory method with other samples were reported. The data are summarized in Table 5. Recoveries were not always determined at the lowest fortification levels.

Table 5. Recovery data for the modified confirmatory method.

Sample	Fortification levels, mg/kg	Mean recoveries					Reference
		Clethodim sulfoxide	5-Hydroxy-clethodim sulfone	Clethodim	Sethoxydim	5-Hydroxy-sethoxydim sulfone	
Sunflower seed	0.5, 2.0	63	109	95		-	Lai, 1995a
Potato	0.2, 0.5, 1.0, 2.0	-	99	88	96	102	Lai, 1995b
Sugar beet roots	0.1, 0.25, 0.5	80-99	85-122	74	97-98	90-110	Lai, 1994a
Sugar beet tops	0.1, 0.25, 0.5	79-110	118-150	82	95-111	86-103	Lai, 1994a
Dry bean seeds	0.2, 1.0, 2.0, 4.0	92-108	79-88	90-99 at 2& 4 mg/kg	88 at 2 mg/kg	63 at 2 mg/kg	Lai, 1994b
Dry bean vines	0.2, 2.5, 5.0, 10.0	110-122 &, 194[1]	84-112 & 26[2]	97 at 5 mg/kg	55-95 at 5mg/kg	74-86 at 5 mg/kg	Lai, 1994b
Dry bean hay	0.2, 3.5, 7.0, 14	68-100	72-121	-	80 at 7 mg/kg	87 at 7 mg/kg	Lai, 1994b

[1]High recovery of 194% at 5 mg/kg described in report as probably being due to low recovery of the quantitative standard. If referred to 10 mg/kg standard recovery is 112%

[2]Low recovery of 26% described in report as probably due to the subtraction of a 0.2-0.l28 mg/kg interfering peak

USE PATTERN

Clethodim is a post-harvest herbicide, registered for a number of vegetable, fruit, oilseed and fodder crops. It was reported that clethodim is currently registered in some 40 countries and on over 40 food commodities. The manufacturer provided information on the current use patterns which is given in Tables 6-9. This information appears to complement the information already reported in the 1994 monograph and is mainly concerned with those commodities for which trials data were submitted to the present Meeting. Product labels were not submitted in support of all the reported GAP. There is no

GAP in The Netherlands (Olthof, 1997). Registered uses shown shaded were described by the manufacturer as “pending”. The manufacturer informed the Meeting that the applications are all overall sprays except on peaches which are treated by directed application around the base of the peach trees.

Table 6. Registered uses of clethodim on fruit (Tomen Agro, 1997). All field applications of EC.

Commodity	Country	Application			PHI, days	Remarks
		Water vol., l/ha	Rate, kg ai/ha	Spray conc., kg ai/hl		
Fruit	Peru	200-400	0.12-0.18	0.045-0.06	15	
Fruit trees	Chile	200-	0.096-0.480	0.048-0.24	-	
	Ecuador	200-600	0.12-0.18	0.03-0.06	-	
	Saudi Arabia	250-300	0.096-0.24	0.038-0.08	60	
Orchard crops	New Zealand	100-400	0.060-0.720	0.06-0.18		Spray to base of tree to avoid contact with fruit
Peach	Spain	300-400	0.096-0.192[1]	0.032-0.048	[2]	Company stated that there is a current registration which will be included on the label at the next printing.

[1]The manufacturer reported that GAP in Spain was 0.036-0.24kg ai/ha (Byrne, 1997)
[2]The manufacturer reported that although the PHI is not specified in Spain, the "PHI is about 14-40 days (Byrne, 1997)
[1]

Table 7. Registered uses of clethodim on vegetables. All EC applications, field or unspecified.

Commodity	Country	Application			PHI, days	Reference Remarks
		Water vol., l/ha	Rate, kg ai/ha	Spray conc., kg ai/hl		
Beans	Belgium	200-400	0.072-0.36	0.036-0.09	60	Tomen Agro, 1997
	Bolivia	150-200	0.072-0.240	0.048-0.12	65	Tomen Agro, 1997 Ground application
		Min 20-	0.072-0.240	0.3-1.2		Tomen Agro, 1997 Aerial application
	Bulgaria	-	0.096-0.192	-	-	Tomen Agro, 1997
	Paraguay	150-200	0.096-0.240	0.06-0.12	-	Tomen Agro, 1997 Ground application
		Min 10	0.096-0.240	0.96-0.24		Tomen Agro, 1997 Aerial application
	Peru	200-400	0.12-0.18	0.06-0.045	15	Tomen Agro, 1997
	Spain	300-400	0.096-0.192	0.032-0.048	30	Tomen Agro, 1997
	Turkey	200-400	0.072-0.19	0.036-0.048	60	Tomen Agro, 1997 Ground application
	Turkey	30-50	0.072-0.19	0.24-0.38		Tomen Agro, 1997 Aerial application
Beans, Mung	Australia	-	0.06-0.09	-	0	Tomen Agro, 1997 Coleman, 1996 Aerial and ground application
Beans, Fava (i.e. Broad beans)	Australia	-	0.036-0.060 or 0.018	-	0	Coleman, 1996 Aerial and ground application 0.018 kg ai/ha used when applied with another specified product
Beet	France	150-350	0.120-0.300	0.08-0.086	100	Tomen Agro, 1997
	Ukraine	300	0.048-0.192	0.016-0.064	-	Tomen Agro, 1997
Beet, Red (Beetroot)	Australia	-	0.035-0.12-	-	7	Coleman, 1996 Aerial and ground application
	Israel	100-300	0.084-0.120	0.084-0.04	-	Tomen Agro, 1997

Commodity	Country	Application			PHI, days	Reference Remarks
		Water vol., l/ha	Rate, kg ai/ha	Spray conc., kg ai/hl		
Beet, Sugar	Germany	200 - 400	0.181	0.045 - 0.09	(F)	Germany, 1996 Spraying Additive: mineral oil 1.38 g ai/l
	Germany	200 - 400	0.242	0.06 - 0.12	(F)	Germany, 1996 Spraying Additive: mineral oil 1.38 g ai/l
Cabbage	Australia (country submission)	50-150	0.036-0.120	-	7	Tomen Agro, 1997 Aerial application: spray volume 20 to 30 l/ha
	Australia (company submission)	-	0.035-0.12-	-	7	Coleman, 1996 Aerial and ground application
	Poland	200-300	0.096-0.240	0.048-0.08	60	Tomen Agro, 1997
Carrot	Brazil		0.108		40	Tomen Agro, 1997 Registration reported to be <u>pending</u> by Ministry of Agriculture.
	Israel	100-300	0.084-0.120	0.084-0.04	-	Tomen Agro, 1997
	Russia	300	0.048-0.240	0.016-0.08	75	Tomen Agro, 1997
Cauliflower	New Zealand	100-400	0.060-0.240	-	35	Tomen Agro, 1997
Celery	Australia	-	0.035-0.12-	-	9 weeks	Coleman, 1996 Aerial and ground application
Chick-peas	Australia	-	0.036-0.060 or 0.018	-	0	Coleman, 1996 Aerial and ground application, 0.018 kg ai/ha used when applied with another specified product
Cucurbits	Paraguay	150-200	0.096-0.240	0.064-0.12	-	Tomen Agro, 1997 Ground application
	Paraguay	Min 10	0.096-0.240	0.96-0.24	-	Tomen Agro, 1997 Aerial application
Cucumber	Poland	200-300	0.096-0.240	0.048-0.08	60	Tomen Agro, 1997
Garlic	Brazil		0.084-0.108		40	Tomen Agro, 1997 Registration reported to be <u>pending</u> by Ministry of Agriculture.
	Saudi Arabia	250-300	0.096-0.24	0.038-0.08	60	Tomen Agro, 1997
	Spain	300-400	0.096-0.192	0.032-0.048	30	Tomen Agro, 1997
	USA (‘Select 2EC’)	187-373.8	0.066-0.280	-	45	Tomen Agro, 1997 Ground application, no more than 0.56 kg ai/ha (0.28 kg ai/ha on Long Island, New York) per season.
		Min. 187	0.066-0.280	-	45	Tomen Agro, 1997 Aerial application
	USA (‘Prism’)	46.7-373.8	0.066-0.280	-	45	Tomen Agro, 1997 Ground application
Legumes	Chile	200 ~	0.096-0.480	0.048-0.24	-	Tomen Agro, 1997
Lentils	Canada	55-225	0.03-0.09	0.054-0.04	60	Tomen Agro, 1997 No more than 0.09 kg ai/ha per season. Do not apply by air.
	New Zealand	100-400	0.060	0.060-0.015	35	Tomen Agro, 1997
	Turkey	200-400	0.072-0.19	0.036-0.048	60	Tomen Agro, 1997 Ground application
		30-50	0.072-0.19	0.24-0.38	60	Tomen Agro, 1997 Aerial application
Lettuce	Australia (company submission)	50-150	0.036-0.120	-	28	Tomen Agro, 1997 Aerial application: spray volume 20 to 30 l/ha

Commodity	Country	Application			PHI, days	Reference Remarks
		Water vol., l/ha	Rate, kg ai/ha	Spray conc., kg ai/hl		
	Australia (country submission)	-	0.035-0.12-	-	28	Coleman, 1996 Aerial and ground application
	Israel	100-300	0.084-0.120	0.084-0.04	-	Tomen Agro, 1997
Onion	Australia (company submission)	50-150	0.036-0.120	0.12-0.08	14	Tomen Agro, 1997 Aerial application: spray volume 20 to 30 l/ha
	Australia (country submission)	-	0.035-0.12-	-	14	Coleman, 1996 Aerial and ground application
	Belize	200-350	0.072-0.120	0.036-0.034	-	Tomen Agro, 1997 Ground application
		30-60	0.072-0.120	0.24-0.20	-	Tomen Agro, 1997 Aerial application
	Brazil		0.084-0.108		40 days.	Tomen Agro, 1997 Registration reported to be pending by Ministry of Agriculture.
	Dominican Republic	208	0.06-0.12	0.03-0.06	7-10	Tomen Agro, 1997
	Guatemala	200-350	0.072-0.120	0.036-0.03	No Restrictions	Tomen Agro, 1997 Ground application
		30-60	0.072-0.120	0.24-0.20	No Restrictions	Tomen Agro, 1997 Aerial application
	Honduras	200-350	0.072-0.120	0.036-0.03	No Restrictions	Tomen Agro, 1997 Ground application
		30-60	0.072-0.120	0.24-0.20		Tomen Agro, 1997 Aerial application
	Israel	100-300	0.084-0.120	-0.084-0.04	-	Tomen Agro, 1997
	New Zealand	100-400	0.060-0.240	-	35	Tomen Agro, 1997
	Russia	300	0.048-0.240	0.016-0.08	65	Tomen Agro, 1997
	Saudi Arabia	250-300	0.096-0.24	0.038-0.08	60	Tomen Agro, 1997
	Turkey	200-400	0.072-0.19	0.036-0.048	40	Tomen Agro, 1997 Ground application
		30-50	0.072-0.19	0.24-0.38		Tomen Agro, 1997 Aerial application
	USA (‘Select 2EC)	46.7-373.8	0.066-0.280	-	45	Tomen Agro, 1997 Ground application, no more than 0.56 kg ai/ha (0.28 kg ai/ha on Long Island, New York) per season.
		Min. 187	0.066-0.280	-		Tomen Agro, 1997 Aerial application
	USA (‘Prism’)	Min 187	0.06-0.28	-	45	Tomen Agro, 1997 Aerial application, no more than 0.56 kg ai/ha per season. (Long Island, New York, no more than 0.28 kg ai/ha per season.
	Uzbekistan	300	0.168-0.240	0.056-0.08	-	Tomen Agro, 1997
Peas	Belgium	200-400	0.072-0.36	0.036-0.09	60	Tomen Agro, 1997
	Czech Republic	-	0.096-0.26	-	-	Tomen Agro, 1997
	Israel	100-300	0.084-0.120	-	-	Tomen Agro, 1997
	New Zealand	100-400	0.060	-	-	Tomen Agro, 1997
	Spain	300-400	0.096-0.192	0.032-0.048	30	Tomen Agro, 1997
Peas, Field	Australia (company submission)	50-150	0.036-0.12	-	Not Req'd	Tomen Agro, 1997 Aerial application: spray volume 20 to 30 l/ha

Commodity	Country	Application			PHI, days	Reference Remarks
		Water vol., l/ha	Rate, kg ai/ha	Spray conc., kg ai/hl		
	Australia (country submission)	-	0.036-0.060 or 0.018	-	0	Coleman, 1996 Aerial and ground application, 0.018 kg ai/ha used when applied with another specified product
	Canada	5-	0.03-0.09	0.054-0.04	75	Tomen Agro, 1997 No more than 0.09 kg ai/ha per season. Do not apply by air.
Peas, Proteaginous	France	150-350	0.120-0.300	0.08-0.086	60	Tomen Agro, 1997
Potato[1]	Australia	-	0.035-0.12	-	4 weeks	Coleman, 1996 Aerial and ground application
	Belgium		0.072-0.18		60	
	Bulgaria		0.096-0.192		not needed	
	Canada		0.09		60	
	Czech Republic		0.096-0.26		not specified	
	Dominian Republic		0.06-0.12		7-10	
	Ecuador		0.06-0.12		not specified	
	Germany[2] (country submission)	200 - 400	0.181	0.045 - 0.09	(F)	Germany, 1996 Spraying on grass weeds (2nd - 6th leaf stage) Additive: mineral oil 1.38 g ai/l
	Germany[2] (country submission)	200 - 400	0.242	0.06 - 0.12	(F)	Germany, 1996 Spraying on couch grass (15-20 cm high) Additive: mineral oil 1.38 g ai/l
	Israel		0.084-0.12		not specified	
	Peru		0.12-0.18		15	
	Poland		0.096-0.24		60	
	Russia		0.048-0.24		40	
	Switzerland		0.12-0.36		56	
	Yugoslavia		0.096-0.192		30	
Tomato	Belize	200-350	0.072-0.120	0.036-0.34		Tomen Agro, 1997 Ground application
		30-60	0.072-0.120	0.24-0.20		Tomen Agro, 1997 Aerial application
	Brazil		0.108		20	Tomen Agro, 1997 Registration reported to be pending by Ministry of Agriculture.
	Bulgaria	-	0.096-0.192	-	-	Tomen Agro, 1997
	Dominican Republic	208	0.06-0.12	-0.03-0.06	7-10	Tomen Agro, 1997
	Israel	100-300	0.084-0.120	-	-	Tomen Agro, 1997
	Italy	300-600	0.142	-	30	Tomen Agro, 1997
	Italy	300-600	0.14	0.047-0.023	30	Tomen Agro, 1997
	Nicaragua	200-350	0.072-0.120	0.036-0.03	No Restriction	Tomen Agro, 1997 Ground application
		30-60	0.072-0.120	0.24-0.20	No Restriction	Tomen Agro, 1997 Aerial application
	Spain	300-400	0.096-0.192	0.032-0.048	30	Tomen Agro, 1997
	USA	46.7-373.8	0.066-0.280	-	20	Tomen Agro, 1997 Ground application, no more than 0.56 kg ai/ha (0.28 kg ai/ha on Long Island, New York) per season.

Commodity	Country	Application			PHI, days	Reference Remarks
		Water vol., l/ha	Rate, kg ai/ha	Spray conc., kg ai/hl		
		28.0-93.5	0.066-0.280	-		Tomen Agro, 1997 Aerial application
Vegetables	Chile	200 ~	0.096-0.480	-0.048-0.24	-	Tomen Agro, 1997
	Ecuador	200-600	0.06-0.12	0.06-0.03	-	Tomen Agro, 1997
	New Zealand	100-400	0.060-0.240	0.06	35	Tomen Agro, 1997
	Paraguay	150-200	0.096-0.240	0.064-0.12	-	Tomen Agro, 1997 Ground application
		Min 10	0.096-0.240	0.96-0.24		Tomen Agro, 1997 Aerial application
	Peru	200-400	0.12-0.18	0.06-0.045	15	Tomen Agro, 1997

[1]The PHI will in practice be long since treatment is made at the early stage of the development of the crop
[2]The manufacturer stated that “a label request has been submitted to the German authorities” and did not supply full information on GAP to the current Meeting

Table 8. Registered uses of clethodim on oilseed, fodder crops and miscellaneous commodities.

Commodity	Country	Form	F/G	Application			No	PHI, days	Reference/ Remarks
				Water vol. l/ha	Rate kg ai/ha	Spray conc. kg ai/hl			
Alfalfa	Argentina	EC	F	150-200	0.096-0.336	0.064-0.164	-	15	Tomen Agro, 1997 Ground application
				Min 20 ~	0.096-0.336	0.40-1.68		15	Tomen Agro, 1997 Aerial application
	Canada	EC	F	55-225	0.03-0.09	0.054-0.04	-	30	Tomen Agro, 1997. No more than 0.09 kg ai/ha per season. Do not apply by air.
	Chile	EC	F	200 ~	0.096-0.480	0.048-0.24	-	-	Tomen Agro, 1997
	Ecuador	EC	F	200-600	0.12-0.18	0.06-0.03	-	-	Tomen Agro, 1997
	Israel	EC	F	100-300	0.084-0.120	0.084-0.04	-	-	Tomen Agro, 1997
	Peru	EC	F	200-400	0.12-0.18	0.06-0.045	-	15	Tomen Agro, 1997
	USA	EC	F	46.7-373.8	0.066-0.280	-	-	15	Tomen Agro, 1997. Ground application, no more than 0.56 kg ai/ha (0.28 kg ai/ha on Long Island, New York) per season.
				28.0-93.5	0.066-0.280	-	-	15	Tomen Agro, 1997. Aerial application
Cotton	Australia	EC	-	-	0.06-0.09	-	1	12 weeks	Coleman, 1996 Aerial and ground application
Clover	Israel	EC	F	100-300	0.084-0.120	-	-	-	Tomen Agro, 1997
	New Zealand	EC	F	100-400	0.120	0.12-0.03	-	63	Tomen Agro, 1997
Flax	Canada	EC	F	55 ~ 225	0.03 ~ 0.09	0.054 ~ 0.04	-	60	Tomen Agro, 1997. No more than 0.09 kg ai/ha per season. Do not apply by air.
	Russia	EC	F	300	0.048 ~ 0.240	0.016 ~ 0.08	1	80	Tomen Agro, 1997

Commodity	Country	Form	F/ G	Application			No	PHI, days	Reference/ Remarks
				Water vol. l/ha	Rate kg ai/ha	Spray conc. kg ai/hl			
	Ukraine	EC	F	300	0.048 ~ 0.192	0.016 ~ 0.064	1	-	Tomen Agro, 1997
Fodder beet	Belgium	EC	F	200-400	0.072- 0.36	0.036- 0.09		90	Tomen Agro, 1997
	Czech Republic	EC	F	-	0.096- 0.26	-		-	Tomen Agro, 1997
	Germany	EC	F	200 - 400	0.181	0.045 - 0.09	1	(F)	Germany, 1996 Spraying Additive: mineral oil 1.38 g ai/l
	Germany	EC	F	200 - 400	0.242	0.06 - 0.12	1	(F)	Germany, 1996 Spraying Additive: mineral oil 1.38 g ai/l
	Italy	EC	F	300-600	0.1417	-		60	Tomen Agro, 1997
	Italy	EC	F	300-600	0.14	-0.047- 0.023		60	Tomen Agro, 1997
	Russia	EC	F	300	0.048- 0.240	0.016- 0.08	1	65	Tomen Agro, 1997
	Switzerland	EC	F	200-400	0.12-0.36	0.06-0.09		-	Tomen Agro, 1997
Lupin	Australia (country submission)	EC	F	50 ~ 150	0.036 ~ 0.120	0.08 ~ 0.12	1	- Not Req'd	Tomen Agro, 1997 Aerial application: spray volume 20 to 30 l/ha
	Australia (country submission)	EC	-	-	0.042- 0.12 or 0.018	-	1	0	Coleman, 1996 Aerial and ground application, 0.018 kg ai/ha used when applied with another specified product
Peanut	Argentina	EC	F	150 ~ 200	0.096 ~ 0.336	0.064 ~ 0.164	-	70	Tomen Agro, 1997 Ground application
				Min 20 ~	0.096 ~ 0.336	0.40 ~ 1.68		70	Tomen Agro, 1997 Aerial application
	Australia (company submission)	EC	F	50 ~ 150	0.060 ~ 0.090	0.12 ~ 0.06	1	49	Tomen Agro, 1997 Aerial application: spray volume 20 to 30 l/ha
	Australia (country submission)	EC	-	-	0.06-0.09	-	1	7 weeks	Coleman, 1996 Aerial and ground application
	Bolivia	EC	F	150 ~ 200	0.072 ~ 0.240	0.048 ~ 0.12	-	65	Tomen Agro, 1997 Ground application
				Min 20 ~	0.072 ~ 0.240	0.36 ~ 1.2		65	Tomen Agro, 1997 Aerial application
	Israel	EC	F	100 ~ 300	0.084 ~ 0.120	0.084 ~ 0.04	-	-	Tomen Agro, 1997
	Taiwan	EC	F	600	0.192	0.032	-	-	Tomen Agro, 1997
	USA	EC	F	46.7 - 373.8	0.066 - 0.280	-	-	40	Tomen Agro, 1997 Ground application
				28.0 ~ 93.5	0.066 ~ 0.280	-	-	40	Tomen Agro, 1997 Aerial application
Rape, oilseed	Australia	EC	-	-	0.036- 0.060 or 0.018	-	1	8 weeks	Coleman, 1996 Aerial and ground application, 0.018 kg ai/ha used when applied with another specified product
Soya beans	Australia	EC	-	-	0.06-0.09	-	1	0	Coleman, 1996 Aerial and ground application

Table 9. Registered uses of clethodim on non-edible commodities.

Form	F/	Application			PHI, days days	Reference /
	G	Water vol., l/ha	Rate, kg ai/ha	Spray conc., kg ai/hl		Remarks
EC 'Select'	F	187-373.8	0.066-0.280	-		Tomen Agro, 1997 Ground application. *Plants which will not bear fruit or nuts for at least one year following application.
		Min 187	0.066 - 0.280	-		Tomen Agro, 1997 Aerial application
EC 'Prism'	F	Min 187	0.06-0.28	-	45	Tomen Agro, 1997 Aerial application

RESIDUES RESULTING FROM SUPERVISED TRIALS

The following additional information was provided for the trials on dry beans, dry peas, potatoes, sugar beet and sunflower which were reviewed in 1994.

Table 10. Further information on supervised residue trials on dry beans reviewed in 1994. Brazil, 1989.

Application			Sample	PHI, days	Residue, mg/kg
kg ai/ha	Water, l/ha	No.			
0.084	300	1	Beans in pods	25 45 65 85	0.37 0.06 <0.05, <0.05
0.108	300	1	Beans in pods	25 45 65 85	0.48 0.07 <0.05 <0.05
0.168	300	1	Beans in pods	25 45 65 85	0.82 0.11 <0.05 <0.05
0.216	300	1	Beans in pods	25 45 65 85	0.93 0.14 <0.05 <0.05

Table 11. Further information on supervised residue trials on dry peas reviewed in 1994.

Country, Ref.	Application			Sample	PHI, days	Residue, mg/kg
	kg ai/ha	Water, l/ha	No.			
Australia, 1987	0.06	133	1	Dry seed, Straw	110	<0.1
	0.12	133	1	Dry seed, Straw	110	<0.1
	0.24	133	1	Dry seed, Straw	110	<0.1
Belgium, 1992	0.09	750	1	Seed	41	<0.025
	0.18	750	1	Seed	41	<0.025
UK, 1988 432-88	0.36	300	1	Seed Husk	53	<0.03, <0.03 <0.03, <0.03
UK, 1988 434-88	0.36	300	1	Seed	85	<0.03, <0.03 <0.03, 0.045
UK, 1988 552-88	0..72	300	1	Seed	85	<0.03, <0.03 0.065, 0.085
UK, 1988 556-88	0.72	300	1	Seed Husk	53	0.05, 0.04, 0.038, 0.045 <0.03, 0.03, <0.03, <0.03
France, 1987	0.18	400	1	Seed	67	0.05
T-2301	0.48	400	1	Seed	67	0.11
	0.96	400	1	Seed	67	0.29
	0.18	400	1	Seed	85	0.06
	0.48	400	1	Seed	85	0.28
	0.96	400	1	Seed	85	0.75
France, 1987	0.18	400	1	Seed	72	0.03
T-2302	0.48	400	1	Seed	72	0.08
	0.96	400	1	Seed	72	0.15
France, 1987	0.18	400	1	Seed	82	0.06
T-2303	0.48	400	1	Seed	82	0.28
	0.96	400	1	Seed	82	0.75
France, 1987	0.18	400	1	Seed	72	<0.03
T-2304	0.48	400	1	Seed	72	0.13
	0.96	400	1	Seed	72	0.17
France, 1987	0.48	400	1	Seed	80	0.04
T-2305	0.96	400	1	Seed	80	0.08
France, 1987	0.18	400	1	Seed	80	<0.03
T-2306	0.48	400	1	Seed	80	0.04
	0.96	400	1	Seed	80	0.14

Table 12. Further information on supervised residue trials on potato tubers reviewed in 1994.

Country	Application rate per treatment			PHI, days	Residue, mg/kg
	kg ai/ha	Water, l/ha	No.		
Ukraine	0.7 to	300	1	40	<0.2
	0.12	300	1	40	<0.2
Canada, 1990, Ontario, Campbellville #1	0.09	225	1	46	0.11, 0.14
	0.09	225		61	0.14, 0.12
	0.18	225		46	0.25, 0.20
Canada, 1990, Ontario, Campbellville #2	0.09	225	1	46	<0.05, <0.05
	0.09	225		61	<0.05, <0.05
	0.18	225		46	0.20, 0.17
Canada, 1990 Ontario, Guelph	0.09	225	1	46	<0.05, <0.05
	0.09	225		61	<0.05, <0.05
	0.18	225		46	<0.05, <0.05
Canada, 1990 Nova Scotia	0.09	225	1	46	<0.05, <0.05

Country	Application rate per treatment			PHI, days	Residue, mg/kg
	kg ai/ha	Water, l/ha	No.		
	0.09	225		61	<0.05, <0.05
	0.18	225		45	<0.05, 0.13
Italy, 1990 265-90	0.24	600	1	60 70 90	<0.03, <0.03 <0.03, 0.07 <0.03, <0.03
Italy, 1991 267-91	0.24	600	1	30 45 60	0.03, <0.03 <0.03, <0.03 <0.03, <0.03
Morocco 1992 683-92	0.14	400	1	91	<0.03
Belgium, 1992 92CLEPDT1	0.09	750	1	112	<0.025
	0.36	750	1	112	<0.025
France, 1987 T-2283	0.18	500	1	47	<0.03, 0.08
France, 1987 T-2284	0.18	500	1	80	<0.03, <0.03

Full study reports were not available for the trials shown shaded

Table 13. Further information on supervised residue trials on sugar beet reviewed in 1994.

Country	Application			Sample	PHI, days	Residue, mg/kg
	kg ai/ha	Water, l/ha	No			
France, 1986 T-2216	0.18	400	1	roots tops	102	<0.03, <0.03
	0.36	400	1	roots tops	102	<0.03, <0.03
France, 1986 T-2217	0.18	400	1	roots tops	112	<0.03, <0.03
	0.36	400	1	roots tops	112	<0.03, <0.03
France, 1986 T-2218	0.36	400	1	roots tops	119	<0.03, <0.03
	0.48	400	1	roots tops	119	<0.03, <0.03
France, 1986 T-2219	0.18	400	1	roots tops	136	<0.03, <0.03
France, 1986 T-2220	0.18	400	1	roots tops	138	<0.03, <0.03
France, 1986 T-2221	0.36	400	1	roots tops	126	<0.03, <0.03
	0.48	400	1	roots tops	126	<0.03, <0.03
France, 1986 T-2293	0.18	400	1	roots tops	113	<0.03, <0.03
	0.36	400	1	roots tops	113	<0.03, <0.03
	0.48	400	1	roots tops	113	<0.03, <0.03
	0.96	400	1	roots tops	113	<0.03, 0.04
France, 1987 T-2294	0.18	400	1	roots tops	122	<0.03, <0.03
	0.36	400	1	roots tops	122	<0.03, <0.03

Country	Application			Sample	PHI, days	Residue, mg/kg
	kg ai/ha	Water, l/ha	No			
	0.48	400	1	roots tops	122	<0.03, <0.03
	0.96	400	1	roots tops	122	<0.03, <0.03
France, 1987 T-2295	0.18	400	1	roots tops	121	<0.03, <0.03
	0.36	400	1	roots tops	121	<0.03, <0.03
	0.48	400	1	roots tops	121	<0.03, <0.03
	0.96	400	1	roots tops	121	<0.03, 0.03
France, 1987 T-2296	0.18	400	1	roots tops	113	<0.03, <0.03
	0.36	400	1	roots tops	113	<0.03, <0.03
	0.48	400	1	roots tops	113	<0.03, <0.03
	0.96	400	1	roots tops	113	<0.03, 0.03
France, 1987 T-2297	0.18	400	1	roots tops	121	<0.03, <0.03
	0.36	400	1	roots tops	121	<0.03, <0.03
	0.48	400	1	roots tops	121	<0.03, <0.03
	0.96	400	1	roots tops	121	<0.03, 0.03
Germany, 1986 400/86	0.14	350	1	root	92 132	<0.05 <0.05
				top	92 132	<0.05 <0.05
Italy, 1991 263-91	0.24	800	1	root	30 45 60	0.08 0.08 0.17
				top	30 45 60	0.23 0.07 0.07
Italy, 1991 263-91	0.24	800	1	root	30 45 59	0.11 0.04 0.06
				top	30 45 59	0.06 0.07 <0.03
Italy, 1991 263-91	0.24	800		root	30 45 60	0.08,<0.03 0.08,<0.03 0.17,<0.03
				top	30 45 60	0.23,0.05 0.07,<0.03 0.07,<0.03
Italy, 1991 264-91	0.24	800		root	30 45 59	0.11,<0.03 0.04,<0.03 0.06,<0.03
				top	30 45 59	0.06,<0.03 0.07,<0.03 <0.03,<0.03
Morocco 688-92	0.14	400	1	root	153	<0.03, <0.03

Table 14. Further information on supervised residue trials on sunflower seed reviewed in 1994.

Country	Application			Sample	PHI, days	Residue, mg/kg
	kg ai/ha	Water, l/ha	No.			
Argentina, 1987 T 7000	0.1	500	1	Seed	108	0.06
	0.24	500	1		108	<0.05
Argentina, 1987 T 7010	0.12	500	1	Seed	102	<0.05
	0.24	500	1		102	0.085
Argentina, 1987 T 7012	0.12	500	1	Seed	106	<0.05
	0.24	500	1		106	0.065
Italy, 1989 292-89	0.24	600	1	Seed	74	<0.03, <0.03
					92	<0.03, <0.03
					110	<0.03, <0.03
Italy, 1991 266-91	0.24	600	1	Seed	60	0.07, 0.13
					75	0.06, 0.13
					90	0.05, 0.10
Italy, 1991 340-91	0.24	600	1	Seed	60	0.04, 0.12
					75	0.06,<0.03
					90	0.05,<0.03
France, 1987 T-2286	0.18	500	1	Seed and hulls	108	<0.03
France, 1987 T-2287	0.48	500	1	Seed	111	<0.03, <0.03
France, 1987 T-2288	0.48	500	1	Seed	123	<0.03, <0.03

(Tomen Agro, 1996, Bryne, 1997)

Additional residue trials were reported to the present Meeting on alfalfa, artichokes, cabbage, field beans, lupins, carrots, cauliflower, clover, celery, flax, garlic, cucumbers, leeks, lentils, head lettuce, onions, peaches, peanuts, peppers, spinach, summer squash and tomatoes.

The results are given in Table 15-41. The trials were in the field unless stated otherwise. Results used for estimating maximum residue and STMR levels are from maximum GAP (minimum PHI, maximum dose rate and maximum number of treatments). GAP reported as applying to broad groups such as "fruit" or "vegetables", has been ignored in the evaluation. GAP for “fruit” was reported for Peru, and for “vegetables” for Chile, Ecuador, New Zealand, Paraguay and Peru.

The residues in the trials were determined by the oxidation/methylation method described in the 1994 monograph (Fujie, 1990) in which the methylated oxidation products DME[1] and DME-OH[2] are determined by GLC with FP detection. The total residues of DME and DME-OH are expressed as clethodim mg/kg clethodim = [(mg/kg DME x 1.22) + (mg/kg DME-OH x 1.16)]. In a limited number of trials the levels of DME and DME-OH were reported separately. Results which have been corrected for recovery are indicated by a note.

Peaches. GAP was reported for Spain where the manufacturer stated that “there is a current registration, which will be included on the label in the next printing”. GAP was also reported for

[1] dimethyl [2-(ethylsulfonyl)propyl]pentanedioate
[2] dimethyl [2-(ethylsulfonyl)propyl]-3-hydroxypentanedioate

"fruit" in Peru, for "fruit trees" in Chile, Ecuador and Saudi Arabia and for "orchard crops" in New Zealand. The maximum application rates are 0.06-0.24 kg ai/hl or 0.18-0.72 kg ai/ha, with PHIs of 15-60 days or not specified. The manufacturer reported that although the PHI is not specified in Spain, it is about 14-40 days.

Table 15. Supervised residue trials on peaches in Spain.

Location	Application				PHI, days	Sample	Residue, mg/kg	Reference, Notes
	Form	No.	kg ai/ha	kg ai/hl				
Molins de Rei	EC	2	0.18	0.06	0 21 60	Fruit	<0.03 <0.03 <0.03 <0.03 <0.03 <0.03	Report No. 0295-89
Tortosa	EC	2	0.18	0.06	0 7	Fruit	<0.03 <0.03 <0.03 <0.03	Report No. 201464 Last treatment at "green fruit".
Ventallo (Viladamat)	EC	2	0.18	0.06	0 7 14	Fruit	<0.03 <0.03 <0.03 <0.03 <0.03 <0.03	Report No. 201472 Last treatment at "unripe fruit".
Larvern	EC	2	0.18	0.06	0 7 14	Fruit	<0.03 <0.03 <0.03 <0.03 <0.03 <0.03	
La Fortesa	EC	2	0.18	0.06	0 7	Fruit	<0.03 <0.03 <0.03 <0.03	
Castelldans	EC	2	0.18	0.06	0 7	Fruit	<0.03 <0.03 <0.03 <0.03	

Garlic. GAP was reported for Saudi Arabia, Spain and the USA. The maximum application rates are 92-0.28 kg ai/ha with PHIs of 30, 45 or 60 days. No maximum number of applications was reported for any country.

The residues in trials considered to comply with US GAP are underlined in the Table 16. Only two trials were considered to comply with US GAP and in these the manufacturer stated that the DME-OH residues were "not considered to be clethodim related, due to matrix interference peak".

Table 16. Supervised residue trials on garlic.

Location, country	Application				PHI, days	Sample	Residue, mg/kg	Reference and Comment
	Form	No.	kg ai/ha	kg ai/hl				
Curitiba/PR, Brazil	EC	1	0.108 0.216		20	Bulb	<0.05 <0.05	Report No. 94026049
			0.108 0.216		40	Bulb	<0.05 <0.05	
			0.108 0.216		60	Bulb	<0.05 <0.05	

Location, country	Application				PHI, days	Sample	Residue, mg/kg	Reference and Comment
	Form	No.	kg ai/ha	kg ai/hl				
Ibia/MG, Brazil	EC	1	0.108 0.216		20	Bulb	<0.05 <0.05	Report No. 94026099
			0.108 0.216		40	Bulb	<0.05 <0.05	
Itapetininga, Sao Paulo, Brazil	EC	2	0.24	0.096	91	Mature dry bulb (tops removed)	<0.02	Report No. T-7119 Bulbs were dried in shade for 7 days.
			0.48	0.19			<0.02	
La Rhoda, Spain	EC	1	0.24	0.08	0 21	Bulb	0.12 <0.03	Report No. 0293-89
Gilroy, CA USA	EC	2	0.28	0.15	45	Mature bulb	0.13,0.14 (DME) 0.24*, 0.20* (DME-OH)	Report No. T-7429 Last treatment at "mature/drying out".
	EC	2	0.28	0.15	60	Mature bulb	<0.1,<0.1 (DME) 0.18*, 0.20* (DME-OH)	*Residue not considered by manufacturer to be Clethodim related due to matrix interference peak.
Firebaugh, California, USA	EC	2	0.28	0.154 ~ 0.156	44	Mature bulb	<0.10 (DME) 0.10* (DME-OH)	Report No. V-1102A Last treatment at "maturing". *Residue not considered by manufacturer to be Clethodim related, due to matrix interference peak.

Onions. GAP for onions was reported for Australia, Belize, Dominican Republic, Guatemala, Honduras, Israel, New Zealand, Russia, Saudi Arabia, Turkey, the USA and Uzbekistan, and pending GAP for Brazil. The maximum application rate was 0.12-0.28 kg ai/ha with PHIs of 7-65 days.

The residues in trials considered to comply with US GAP are underlined in Table 18. Those according to New Zealand GAP or the pending Brazilian GAP are double underlined, and the single residue from a trial according to Australian GAP is shown in bold italics. Although the Brazilian trials and one of the Ukrainian trials were at elevated dose rates all the residues were <0.05 and <0.01 mg/kg respectively.

Table 17. Supervised residue trials on onions.

Location Country	Application				PHI, days	Sample	Residue, mg/kg	Reference and Comment
	Form	No.	kg ai/ha	kg ai/hl				
Nobby, Queensland Australia	EC	2	0.12	0.094	14 21 28	Bulb	***0.05*** 0.02 0.02	Report No. 5/AU/H6/201/92 Results corrected for mean recoveries
	EC	2	0.24	0.19	14 21 28	Bulb	0.14 0.06 0.08	
	EC	-			-	Bulb	<0.02	
Ibia/MG, Brazil	EC	1	0.108 0.216	- -	20	Bulb	<0.05 <0.05	Report No. 96000097
			0.108 0.216	- -	40		<0.05 <0.05	
			0.108 0.216	- -	60		<0.05 <0.05	

Location Country	Application Form	No.	kg ai/ha	kg ai/hl	PHI, days	Sample	Residue, mg/kg	Reference and Comment
Curitiba/PR, Brazil	EC	1	0.108 0.216	- -	20	Bulb	<0.05 <0.05	Report No. 94026623
			0.108 0.216	- -	40		<0.05 <0.05	
			0.108 0.216	- -	60		<0.05 <0.05	
Itapetininga, Sao Paula, Brazil	EC	1	0.24 0.48	0.08 0.16	68	Whole green onion	<0.03 <0.03	Report No. T-7131
			0.24 0.48	0.08 0.16		Mature dry bulb (tops removed)	<0.03 <0.03	Bulbs were dried in shade for 15 days.
Borgo Piave, Italy	EC	1	0.24	0.04	20	Bulb	<0.03 <0.03	Report No. 0291-89
					30		<0.03 <0.03	Last treatment at "bulb development".
					40		<0.03 <0.03	
Hope, Nelson, New Zealand	EC	1	0.24	0.08	42 84	Bulb	<0.03 <0.03	
			0.48	0.16	42 84		<0.03 <0.03	
Astrakhan, Russia	EC	-	0.096	0.06	65	Bulb	<0.01	
			0.12	0.12	65		<0.01	
			0.096	0.06	90		<0.01	
			0.240	0.12	90		<0.01	
Kiev, Ukraine	EC	-	0.3 to 1.2	-	55	Bulb	<0.01	
Kharkov, Ukraine	EC	-	0.024	0.012	6 58 110	Bulb	<0.02 <0.02 <0.02	
			0.060	0.03	6 58 110		<0.02 <0.02 <0.02	
			-	-	-		<0.02	
Kiev, Ukraine	EC	-	0.168	0.08	61 128	Bulb	<0.02 <0.02	
			0.240	0.12	61 128		<0.02 <0.02	
			-	-	-		<0.02	
Shook, Texas, USA	EC	2	0.30	0.15	45	Mature bulb	0.17 0.12	Report No. T-7300 Duplicate analyses, not duplicate field sample
Fresno, CA, USA	EC	2	0.28	0.15	30	Mature bulb	0.15 <0.05	Report No. T-7301
					45		<0.05 <0.05	Growth stage at last treatment: 30 day: 1¼-2" bulb 45 day: 1-1½" bulb 60 day: ¾-1" bulb
					60		<0.05 <0.05	
Hillsboro, Oregon, USA	EC	2	0.28	0.15	45	Mature bulb	<0.1 <0.1	Report No. T-7302 Last treatment at "6-8 leaf".
Greely, Colorado, USA	EC	2	0.28	0.15	44	Mature bulb	<0.1 <0.1	Report No. T-7303 Last treatment at "bulbing".

Location Country	Application				PHI, days	Sample	Residue, mg/kg	Reference and Comment
	Form	No.	kg ai/ha	kg ai/hl				
Phelps, New York, USA	EC	2	0.28	0.15	30	Mature bulb	<0.1 <0.1	Report No. T-7304
					45		<0.1 <0.1	Growth stage at last treatment:
					60		<0.1 <0.1	30 day: 6-8 leaf 45 day: 4-6 leaf 60 day: 2-3 leaf
Allendale, Michigan, USA	EC	2	0.28	0.145-0.144	30	Mature bulb	<0.1 <0.1	Report No. V-1001-A
					45		<0.1 <0.1	Last treatment at "8 leaves, 2 inch diameter bulbs".
					60		<0.1 <0.1	
Corcoran, California, USA	EC	2	0.28	0.145	44	Mature bulb	0.11 0.14	Report No. V-1001-B
			0.56	0.295-0.301			0.12 0.12	Last treatment at "maturing/drying out" stage. Duplicate analyses, not duplicate field samples

Leeks. No specific GAP for leeks was reported. The manufacturer claimed that "since leek is classified in Codex under bulb vegetables, the use on onions or garlic can also apply to leeks."

Table 18. Supervised residue trials on leeks.

Location	Application				PHI, days	Sample	Residue, mg/kg	Reference and Comment
	Form	No.	kg ai/ha	kg ai/hl				
Bieujac (Gironde), France	EC	1	0.12	0.036	28	Whole plants	0.13 0.07	Report No. TE-2316
					56		0.11 0.09	
			0.18	0.096	28		0.34 0.31	
					56		0.12 0.17	

Cabbages. GAP was reported for Australia and Poland, with maximum application rates of 0.12 and 0.24 kg ai/ha and PHIs of 7 and 60 days respectively.

The residues resulting from trials according to Australian and Polish GAP are underlined and double underlined respectively, in Table 19.

Table 19. Supervised residue trials on cabbages.

Location	Application				PHI, days	Sample	Residue, mg/kg	Reference and Comment
	Form	No.	kg ai/ha	kg ai/hl				
Cranbourne, Victoria Australia	EC	1	0.12	0.096	1 7	Head	0.11 0.07	Report No. 223/AU/94/100/SV01
			0.24	0.192	1 7		0.52 0.20	Last treatment at "mature" stage.
			Control				<0.02	

Location	Application				PHI, days	Sample	Residue, mg/kg	Reference and Comment
	Form	No.	kg ai/ha	kg ai/hl				
Bazas, France	EC	1	0.18	0.036	67	Heart	0.04 0.04	Report No. TE-2324 Duplicate analyses, not duplicate field samples
			0.18	0.036			<u>0.13</u> <u>0.16</u>	

<u>Cauliflowers</u>. GAP was reported only for New Zealand, with a maximum application rate of 0.24 kg ai/ha and a PHI of 35 days. A single trial complied with the New Zealand GAP. The residues is underlined in Table 20..

Table 20. Supervised residue trials on cauliflowers.

Location Country	Application				PHI, days	Sample	Residue, mg/kg	Reference and Comment
	Form	No.	kg ai/ha	kg ai/hl				
Hope, Nelson New Zealand	EC	1	0.24	0.08	42 84	Head	<u>0.28</u> <0.03	Report No. 880542 Last treatment at “no florets formed” and “seedlings just planted”.
			0.48	0.16	42 84		0.59 <0.03	
	Control						<0.03	

<u>Cucumbers</u>. GAP was reported for cucumbers in Poland with a maximum application rate of 0.24 kg ai/ha and a PHI of 60 days, and for cucurbits in Paraguay with the same application rate and an unstated PHI.

The only residue considered to result from Polish GAP is underlined in Table 21. Although the PHIs were shorter than GAP the residues were all <0.05 mg/kg.

Table 21. Supervised residue trials on cucumbers.

Location	Application				PHI, days	Sample	Residue, mg/kg	Reference and Comment
	Form	No.	kg ai/ha	kg ai/hl				
Poland	EC	1	0.24	0.15	35	Fruit	<0.05 <0.05 <0.05	Annual Report, 1995
					38		<u><0.05</u> <0.05 <0.05	Last treatment at “4-6 leaves” stage.
Michigan, USA	EC	2	0.28		13	Mature fruit	<0.14 <0.14	PR No. 5219.94
South Carolina, USA	EC	2	0.28		13		<0.14 <0.14	
Florida, USA	EC	2	0.28		14	Mature fruit	<0.14 <0.14	PR No. 5219.94
New York, USA	EC	2	0.28		14		<0.14 <0.14	
Texas, USA	EC	2	0.28		14	Mature fruit	<0.14 <0.14	PR No. 5219.94
Wisconsin, USA	EC	2	0.28		14		<0.14 <0.14	

Summer squash. The only relevant GAP was for cucurbits in Paraguay but none of the three US trials complied with it. The results are shown in Table 22.

Table 22. Supervised residue trials on summer squash in the USA.

Location	Application				PHI,	Sample	Residue,	Reference
	Form	No.	kg ai/ha	kg ai/hl	days		mg/kg	
California	EC	2	0.28		15	Whole fruit	<0.10 <0.10	PR No. 5228.96
Michigan	EC	-	0.28		14		0.11 <0.10	
Ohio	EC	2	0.28		14	Whole fruit	<0.10 <0.10	PR No. 5228

Tomatoes. GAP for tomatoes was reported for Belize, Bulgaria, Dominican Republic, Israel, Italy, Nicaragua, Spain and the USA, and pending GAP for Brazil. The maximum application rates are 0.12-0.28 kg ai/ha (0.108 kg/ha for the pending GAP) with PHIs of 7-30 days, "unrestricted" or unstated.

The residues from trials complying with the pending Brazilian, the Spanish and US GAP are underlined, double underlined and in italics respectively in Table 23.

Table 23. Supervised residue trials on tomatoes.

Location	Field/ Prot-ected	Application				PHI,	Sample	Residue,	Reference and Comment
		Form	No.	kg ai/ha	kg ai/hl	days		mg/kg	
Sitio Sao Joao, Itupeva Sao Paulo, Brazil	-	EC	1	0.108 0.216		20	Fruit	<0.05 <0.05	Report No. 23687/94
				0.108 0.216		40		<0.05 <0.05	
				0.108 0.216		60		<0.05 <0.05	
Curitiba/PR Brazil	-	EC	1	0.108 0.216		20	Fruit	<0.05 <0.05	Report No. 21929/94
				0.108 0.216		40		<0.05 <0.05	
				0.108 0.216		60		<0.05 <0.05	
Casal Nuovo, Italy	F	EC	1	0.24	0.04	30	Mature fruit	0.08 0.13	Report No. 207-88
						51		<0.030 <0.030	
Gava, Spain	F	EC	1	0.24	0.08	0 21 60	Fruit	<0.03 <0.03 <0.03	Report No. 0294-89 Last treatment at "beginning to mature" stage.
Viladecans, Spain	F	EC	1	0.18	0.06	0 21	Fruit	<0.03 <0.03	Report No. 225-90 Last treatment at "mature" stage.
Malgrat de Mar, Spain	F	EC	1	0.18	0.06	0 21	Fruit	0.05 0.03	Report No. 0226-90 Last treatment at "beginning to mature and flower" stage.

Location	Field/ Prot-ected	Application				PHI, days	Sample	Residue, mg/kg	Reference and Comment
		Form	No.	kg ai/ha	kg ai/hl				
Mataro, Spain	F	EC	1	0.18	0.06	0 21	Fruit	<0.03 <0.03	Report No. 0227-90 Last treatment at "beginning to mature" stage.
Viladecans, Spain	F	EC	1	0.18	0.06	0 22	Fruit	0.03 <0.03	Report No. 206202 Last treatment at "mature" stage.
43204 Reus, Spain	F	EC	1	0.18	0.06	0 21	Fruit	<0.03 <0.03	Report No. 206199 Last treatment at "mature" stage.
California, USA	-	EC	2	0.28-0.29	0.13-0.14	15	Mature fruit	0.85 1.4	Report No. V-10688-A Last treatment at "fruit, some 2 inches" stage.
						20		*0.76,0.71* *0.82,0.76*	
						30		0.54 0.64	
Florida, USA	-	EC	2	0.28	0.11	15	Mature fruit	<0.1 <0.1	Report No. V-10688-B Last treatment at "1st cluster, 2½-3½ inch diameter" stage.
						20		*0.35* *0.15*	
						30		<0.1 <0.1	
Ohio, USA	-	EC	2	0.28	0.10	15	Mature fruit	0.20 0.25	Report No. V-10688-C Last treatment at "ripening" stage.
						20		*0.34* *0.27*	
						30		0.18 0.18	
New Jersey, USA	-	EC	2	0.28	0.10	21	Mature fruit	*0.46* *0.43*	Report No. V-10688-D Last treatment at "fruiting" stage.
Indiana, USA	-	EC	2	0.28	0.22-0.26	20	Mature fruit	*0.12* *0.11*	Report No. 10688-E Last treatment at "bloom/first fruit" stage.
California, USA	-	EC	2	0.27-0.28	0.097-0.10	20	Mature fruit	*0.15* *0.16*	Report No. 10688-F Last treatment at "green-mature fruit" stage.
				1.38-1.40	0.50			1.1 1.2	
California, USA	-	EC	2	1.38-1.40	0.50	20	Mature fruit	0.81,0.87	Report No. V-10688-G Last treatment at "green fruit" stage.
							Wet pomace	0.78,0.79	
							Dry pomace	2.6,2.5	
							Paste	3.0,2.8	
							Juice	0.71,0.81	
South Carolina, USA	-	EC	2	0.28-0.29	0.11-0.14	20	Mature fruit	*0.35* 0.35	Report No. V-10688A-A Last treatment at "fruit 2.0 inches in diameter" stage.
Florida, USA	-	EC	2	0.28	0.12	20	Mature fruit	*<0.1* *<0.1*	Report No. V-10688A-B Last treatment at "mature green" stage.
California, USA	-	EC	2	0.28	0.15	20	Mature fruit	*0.65* *0.54*	Report No. V-10688-A-C Last treatment at "crop height 18-24 inches" stage.
Ohio, USA	-	EC	2	0.28	0.16	20	Mature fruit	*0.17* *0.21*	Report No. V-10688A-D Last treatment at "fruiting" stage.
				1.4	0.79			1.7 1.4	

Location	Field/ Prot-ected	Application				PHI, days	Sample	Residue, mg/kg	Reference and Comment
		Form	No.	kg ai/ha	kg ai/hl				
Ohio, USA	-	EC	2	1.4	0.79	20	Mature fruit	1.2	Report No. V-10688A-E
							Wet pomace	1.0	
							Dry pomace	4.0	
							Paste	3.6	
							Juice	0.92	
							Purée	2.6	
Pennsylvania, USA	-	EC	2	0.27-0.29	0.12-0.13	20	Mature fruit	*0.52* *0.50*	Report No. V-10688A-F Last treatment at “1% of fruit showing colour” stage.
Michigan, USA	-	EC	2	0.27-0.28	0.12	20	Mature fruit	*<0.10* <0.10	Report No. V-10688A-G Last treatment at “1% mature up to 3 inches diameter” stage.

Spinach. No GAP was reported.

Table 24. Supervised residue trials on spinach.

Location	Application				PHI, days	Sample	Residue, mg/kg	Reference
	Form	No.	kg ai/ha	kg ai/hl				
Puybarban, France	EC	1	0.12	0.024	15	Whole plants	0.14	Report No. TE-2315
			0.18	0.036	30		0.04	
			0.18	0.036	15		0.19	
			0.48	0.096	30		0.08	
					15		0.10	
					30		0.03	
					15		0.15	
					30		0.08	

Lettuce. GAP was reported for Australia and Israel. Both maximum application rates are 0.12 kg ai/ha. The PHIs are 28 days and unstated respectively.

The residues from trials according to the Australian GAP are underlined in Table 25.

Table 25. Supervised residue trials on lettuce.

Location	Field/ Prot-ected	Application				PHI, days	Sample	Residue, mg/kg	Reference and Comment
		Form	No.	kg ai/ha	kg ai/hl				
Grantham, Queensland, Australia	-	EC	1	0.12	0.087	0 14 28	Head	0.91 0.10 0.04	Report No. S/AU/H6/207/92 Last treatment at “30-50 cm diameter”.
				0.24	0.174	0 14 28		2.15 0.08 0.06	

Location	Field/ Prot-ected	Application				PHI, days	Sample	Residue, mg/kg	Reference and Comment
		Form	No.	kg ai/ha	kg ai/hl				
		Control				-		<0.02	Results corrected for mean recoveries
Bazas, France	-	EC	1	0.12	0.024	28	Whole heads	0.19 0.09	Report No. TE-2323
				0.18	0.036			0.13 0.11	
				0.18	0.036			0.15 0.27	
				0.48	0.096			0.34 0.29	
Stornara (FG), Italy	F	EC	1	0.24	0.04	0 10 15 20	Head	0.279 0.346 0.141 0.178 0.042 0.065 0.053 0.082	Report No. 0271-90

Beans, except soya beans. GAP was reported for beans for Belgium, Bolivia, Bulgaria, Paraguay, Peru, Spain and Turkey, for mung and fava beans (i.e. broad beans) for Australia and for legumes for Chile. The maximum application rates are 0.06-0.36 kg ai/ha for beans and 0.48 kg ai/ha for legumes, with PHIs of 0-65 days or unstated.

The residues in fresh beans from trials considered to comply with Belgian GAP are underlined in Table 26. Although some of the Belgian results were at shorter PHIs than GAP, the residues were all below the limit of determination. In addition one trial on "green beans" with a residue level of 0.21 mg/kg and one on "French beans" with a residue of <0.03 mg/kg were considered to comply with Spanish GAP; these results are double underlined. Although data on a number of US trials were also submitted, no GAP was reported for the North American continent.

Data from a trial on broad beans in Spain are shown in Table 27. The trial did not comply with relevant GAP.

Table 26. Supervised residue trials on beans.

Crop, Location	Field/ Prot-ected	Application				PHI, days	Sample	Residue, mg/kg	Reference and Comment
		Form	No.	kg ai/ha	kg ai/hl				
Fresh beans B2370 Arendonk, Belgium	-	EC	-	0.36	0.12	39	Pod	<0.05 <0.05	Report No. SELPV01 Last treatment at "BBCH 12".
Fresh beans, B3891 Mielen, Belgium	-	EC	-	0.36	0.12	40	Pod	<0.05	Report No. SELPV03 Last treatment at "BBCH60".
Fresh beans, B3680 Maaseik, Belgium	-	EC	-	0.36	0.12	40	Pod	<0.05	Report No. SELPV04 Last treatment at "BBCH55".

Crop, Location	Field/ Prot-ected	Application				PHI, days	Sample	Residue, mg/kg	Reference and Comment
		Form	No.	kg ai/ha	kg ai/hl				
Fresh beans, B3891 Mielen, Belgium	-	EC	-	0.36	0.12	39	Pod	<0.05	Report No. SELPV05 Last treatment at "BBCH55".
Beans (succulent), B8810 Beitem, Belgium	-	EC	1	0.09	0.012	51	Pod	<0.025 <0.025	Report No. 92clehar1 Last treatment at "Stage 1: vegetative".
Beans (succulent), B8810 Beitem, Belgium	-	EC	1	0.09	0.012	51	Pod	<0.025 <0.025	Report No. 92clehar2 Last treatment at "Stage 1: vegetative".
Beans (succulent), B7561 Esen, Belgium	-	EC	1	0.12	0.04	56	Pod	<0.025	
				0.36	0.12			<0.025	Report No. 93clehar1
Beans (succulent), Tennessee, USA	-	EC	2	0.28		21	Pod	0.60, 0.52 0.59, 0.58	PR No. 5205.94 TN02 Controls but not treated samples were corrected for interference with DME-OH peak
Beans (succulent), Oregon, USA	-	EC	2	0.28		21	Pod	0.22, 0.15, 0.24, 0.18, 0.29	PR No. 5205.93 OR34 Controls but not treated samples were corrected for interference with DME-OH peak
Beans (succulent), Florida, USA	-	EC	2	0.28		21	Pod	0.13, 0.27, 0.13 0.04, <0.05, 0.16 0.04, 0.41, 0.12 0.07, 0.11, 0.09	PR No. 5205.93 FL32 Controls but not treated samples were corrected for interference with DME-OH peak
Beans (succulent), Wisconsin, USA	-	EC	2	0.28		21	Pod	0.20, 0.17 0.18, 0.19 0.15, 0.18 0.21, 0.14, 0.15	PR No. 5205.93 WI07 Controls but not treated samples were corrected for interference with DME-OH peak
Beans (succulent), New York, USA	-	EC	2	0.28		22	Pod	<0.10 <0.10 <0.10	PR No. 5205.92 NY37 Controls but not treated samples were corrected for interference with DME-OH peak
Beans (succulent), Maryland, USA	-	EC	2	0.28		22	Pod	<0.10 0.59 0.65	PR No. 5205.92 MD09

Crop, Location	Field/ Prot-ected	Application				PHI, days	Sample	Residue, mg/kg	Reference and Comment
		Form	No.	kg ai/ha	kg ai/hl				
Beans (succulent), Wisconsin, USA	-	EC	2	0.28		23	Pod	<0.10 <0.10 <0.10	PR No. 5205.92 WI19
Beans (succulent), Michigan, USA	-	EC	2	0.28		21	Pod	<0.10 <0.10 0.11	PR No. 5205.92 MI19
Beans (succulent), California, USA	-	EC	2	0.28		19	Pod	<0.10 <0.10 <0.10	PR No. 5205.92 CA124
French beans, Villandraut (Gironde), France	-	EC	1	0.18	0.036	32	Pod	<0.03 <0.03	Report No. TE-2282
Green beans, Latina, Italy	F	EC	1	0.24	0.04	20	Bean	0.19 0.24	Report No. 0209-88
						24		0.17 0.21	

Table 27. Supervised residue trials on broad beans in Spain.

Location	Field/ Prot-ected	Application				PHI, days	Sample	Residue, mg/kg	Reference
		Form	No.	kg ai/ha	kg ai/hl				
Barcelona,	F	EC	1	0.139	0.03	57	Bean	<0.03 <0.03	Report No. 0070-88
Gava							Husk	<0.03 <0.03	

Lupins. GAP for lupins was reported for Australia, with a maximum application rate of 0.12kg ai/ha; no PHI was specified. Only one trial complied with this. The residue is underlined in Table 28.

Table 1. Supervised residue trials on lupins in Australia.

Location	Application				PHI, days	Sample	Residue, mg/kg	Reference and Comment
	Form	No.	kg ai/ha	kg ai/hl				
Cosgrove, Victoria,	EC	1	0.06	0.045	167	Grain (Dried Seed)	<0.1 <0.1 <0.1	Report No. T-7236
		-	-	-	-		<0.1	
		-	0.06 0.12 0.24	0.045 0.090 0.18	167	Straw	<0.1 <0.1 <0.1	
	Control						<0.1	

Lentils. GAP for lentils was reported to the current Meeting for Canada, New Zealand and Turkey, and to the 1994 Meeting for New Zealand and Spain. GAP for beans and legumes would include lentils. The maximum application rates are 0.06-0.19 kg ai/ha for lentils, 0.06-0.36 kg ai/ha for beans and 0.48 kg ai/ha for legumes with PHIs of 35 or 60 days for lentils and 15-65 days or unstated for beans and legumes.

Although two Spanish trials were reported, they could not be evaluated against Spanish GAP recorded in 1994 because the GAP did not include a PHI.

Table 29. Supervised residue trials on lentils.

Location	Field/ Prot-ected	Application				PHI, days	Sample	Residue, mg/kg	Reference and Comment
		Form	No.	kg ai/ha	kg ai/hl				
Poplar Point, Manitoba, Canada	-	EC	1	0.045	0.04	84	Seed	<0.02 <0.02	Report No. 94-102 DC Growth stage at last treatment: 84 days: 6-8 cm 70 days: 12-15 cm
				0.09	0.08			<0.02 <0.02	
				0.18	0.16			<0.02 <0.02	
				0.045	0.04	70		<0.02 <0.02	
				0.09	0.08			<0.02 <0.02	
				0.18	0.16			<0.02 <0.02	
Koral, Sask., Canada	-	EC	1	0.18	0.16	103	Seed	<0.02 <0.02	Report No. 94-102 DC Growth stage at last treatment: 103 days: 17 cm 118 days: 7 nodes, 6-7 cm
						118		<0.02 <0.02	
Haight, Sask., Canada	-	EC	1	0.18	0.16	92	Seed	<0.02 <0.02	Report No. 94-102 DC Growth stage at last treatment: 92 days: actively growing, 6 cm height 107 days: 15-18 cm height
						107		<0.02 <0.02	
Oakbluff, Manitoba, Canada	-	EC	1	0.045	0.04	91	Seed	<0.02 <0.02	Report No. 94-102 DC Growth stage at last treatment: 91 days: 6-8 cm 76 days: 12-15 cm
				0.09	0.08			<0.02 <0.02	
				0.18	0.16			<0.02 <0.02	
				0.045	0.04	76		<0.02 <0.02	
				0.09	0.08			<0.02 <0.02	
				0.18	0.16			<0.02 <0.02	
Rowatt, Sask., Canada	-	EC	1	0.045	0.04	89	Seed	<0.02 <0.02	Growth stage at last treatment: 89 days: 6-7 leaf 77 days: flowering
				0.09	0.08			<0.02 <0.02	
				0.18	0.16			<0.02 <0.02	
				0.045	0.04	77		<0.02 <0.02	
				0.09	0.08			<0.02 <0.02	
				0.18	0.16			<0.02 <0.02	

Location	Field/ Prot-ected	Application Form	No.	kg ai/ha	kg ai/hl	PHI, days	Sample	Residue, mg/kg	Reference and Comment
Okotaks, Atta, Canada	-	EC	1	0.18	0.16	75	Seed	<0.02 <0.02	Growth stage at last treatment: 75 days: flowering stage, 30-35 cm height 90 days: 9 node leaf stage 18-22 cm height
						90		<0.02 <0.02	
Koral, Sask., Canada	-	EC	1	0.04	0.04	58	Seed	<0.02 <0.02	Last treatment at “20 cm” stage.
				0.09	0.08			0.04 0.05	
				0.18	0.16			0.09 0.12	
Bristows, Sask., Canada	-	EC	1	0.04	0.04	62	Seed	0.05 0.05	Last treatment at “30 cm, 13 nodes” stage.
				0.09	0.08			0.15 0.12	
				0.18	0.16			0.22 0.31	
Airdrie, Alberta, Canada	-	EC	1	0.045	0.04	60	Seed	0.13 0.10	Last treatment at “pod filling” stage.
				0.09	0.08			0.27 0.25	
				0.18	0.16			0.22 0.35	
Morden, Manitoba, Canada	-	EC	-	0.18	0.13	67	Seed	<0.02 <0.02	Last treatment at “8 cm, 6 nodes” stage.
Richardson, Sask. Canada	-	EC	1	0.09	0.08	58	Seed	0.04 0.04	Last treatment at “12-26 cm height” stage. Suspect results owing to possible labelling error
				0.18	0.16			0.04 0.04	
Koral, Sask., Canada	-	EC	1	0.045	0.04	72	Seed	<0.02 <0.02	Last treatment at “15 cm” stage.
				0.09	0.08			<0.02 <0.02	
				0.18	0.16			<0.02 <0.02	
Bristows, Sask., Canada	-	EC	1	0.045	0.04	77	Seed	<0.02 <0.02	Last treatment at “25 cm” stage.
				0.09	0.09			<0.02 <0.02	
				0.18	0.16			0.05 0.03	
Airdrie, Alberta, Canada	-	EC	1	0.045	0.04	75	Seed	0.06 0.08	Last treatment at “flowering” stage.
				0.09	0.09			0.02 0.03	
				0.18	0.16			0.07 0.10	
Morden, Manitoba, Canada	-	EC	1	0.18	0.13	77	Seed	<0.02 <0.02	Last treatment at “8 cm, 6 nodes” stage.
Lentils, Richardson, Sask., Canada	-	EC	1	0.09	0.08	74	Seed	0.22 0.23	Last treatment at “3-12 cm height” stage. Suspect results owing to possible labelling error
				0.18	0.16			0.05 0.13	

Location	Field/	Application				PHI,	Sample	Residue,	Reference and Comment
	Prot-ected	Form	No.	kg ai/ha	kg ai/hl	days		mg/kg	
Castrobol, Spain	F	EC	1	0.18	0.06	0 21	Husk	2.2 1.4	Report No. 229-90 Last treatment at “before ripening” stage.
Monte Urones-Mayorage Valladolid, Spain	F	EC	1	0.18	0.06	0 21	Husk	2.2 1.1	Report No. 228-90 Last treatment at “before mature” stage.

Carrots. GAP for carrots was reported for Israel and Russia, and pending GAP for Brazil. The maximum applications rates are 0.108 (Brazil) to 0.24 kg ai/ha with PHIs of 40-75 days.

The residues from trials according to the pending Brazilian GAP are underlined in Table 30. None of the other trials accorded with reported GAP.

Table 30. Supervised residue trials on carrots.

Location	Application				PHI,	Sample	Residue,	Reference and Comment
	Form	No.	kg ai/ha	kg ai/hl	days		mg/kg	
Engenheiro, Coelho/SP, Brazil	EC	1	0.108 0.216		20	Root	<0.05 <0.05	Report No. 9402487
			0.108 0.216		40		<0.05 <0.05	
			0.108 0.216		60		<0.05 <0.05	
Engenheiro, Coelho/SP, Brazil	EC	1	0.108 0.216		20	Root	<0.05 <0.05	Report No. 96000098
			0.108 0.216		40		<0.05 <0.05	
			0.108 0.216		60		<0.05 <0.05	
Moscow region, Russia	EC	-	0.096	0.048	0 20 40 60 73	Root-crops	0.11 0.29 0.032 <0.1 <0.1	Last treatment at “4 leaves” stage.
			0.240	0.12	0 20 40 60 70 85		0.21 0.43 0.050 <0.1 <0.1 <0.1	
Leningrad region, Russia	EC	-	0.24	0.12	0 20 45 75		0.02 0.37 0.51 <0.1	
Kiev, Ukraine	EC	-	0.07- 0.29	-	-	Root	<0.01	
Florida, USA	EC	2	0.28	-	30	Root	<0.14 <0.14	PR No. 5217.94 FL74
Michigan, USA	EC	2	0.28	-	31	Root	<0.14 <0.14	PR No. 5217.95 MI02
California, USA	EC	2	0.28	-	29	Root	<0.14 <0.14	PR No. 5217.94 CA*91
California, USA	EC	2	0.28	-	31	Root	<0.14 <0.14	PR No. 5217.94 CA*92

Location	Application				PHI,	Sample	Residue,	Reference and Comment
	Form	No.	kg ai/ha	kg ai/hl	days		mg/kg	
California, USA	EC	2	0.28	-	29	Root	0.25 0.22	PR No. 5217.95 CA*48 .
California, USA	EC	2	0.28	-	31	Root	<0.14 <0.14	PR No. 5217.95 CA*49
Texas, USA	EC	2	0.28	-	29	Root	0.18 0.28	PR No. 5217.95 WA*32
Washington, USA	EC	2	0.28	-	31	Root	<0.14 <0.14	PR No. 5217.95 WA*32

Artichokes. No specific GAP for artichokes was reported, and the single reported trial in Italy did not comply with any relevant general GAP.

Table 31. Supervised residue trial on artichokes in the field.

Location	Application				PHI,	Sample	Residue,	Reference and Comment
	Form	No.	kg ai/ha	kg ai/hl	days		mg/kg	
Stornara, Italy	EC	1	0.24	0.03	20	Fruit	0.50 0.57	Report No. 0289-89
					25		0.26 0.33	
					30		0.18 0.29	

Celery. GAP for celery was reported for Australia, with a maximum application rate of 0.12 kg ai/ha and a PHI of 9 weeks.

The residue from the single trial under these conditions is underlined in Table 32.

Table 32. Supervised residue trials on celery.

Location	Application				PHI,	Sample	Residue,	Reference
	Form	No.	kg ai/ha	kg ai/hl	days		mg/kg	
Cranbourne, Victoria, Australia	EC	1	0.12 0.24 0.48	0.096 0.192 0.384	66	Stalk	<u>0.04</u> 0.13 0.15	Report No. 5/AU/H2/91
			Control-				<0.02	
Texas, USA	EC	2	0.28	-	30	Stalk	0.26 0.31	Report No. 5218.95-TX*44
California, USA	EC	2	0.28	-	39	Stalk	0.33 0.33	Report No. 5218.94-CA*32
California, USA	EC	2	0.28	-	31	Stalk	0.12 0.14	Report No. 5218.94-CA*31
Florida, USA	EC	2	Not stated	-	30	Stalk	0.14 0.14	Report No. 5218.94-FL46
Michigan, USA	EC	2	Not stated	-	31	Stalk	<0.10 <0.10	Report No. 5218.94-MI13

Flax (linseed). GAP was reported for Canada, Russia and the Ukraine. The maximum application rates are 0.09-0.24 kg ai/ha and PHIs 60-80 days or not specified.

None of the Canadian trials were in strict accordance with the Canadian GAP. The residues in several trials at exaggerated dose rates were <0.05 mg/kg at PHIs of 84-119 days, but the Canadian PHI is 60 days. The residues from the single trial in accordance with Russian GAP is double underlined in Table 33.

Table 33. Supervised residue trials on flax.

Location	Application				PHI, days	Sample	Residue, mg/kg	Reference and Comment
	Form	No.	kg ai/ha	kg ai/hl				
Birch Hills, Saskatchewan Canada	EC	1	0.12 0.24 -	0.108 0.216 -	116	Mature seed	<0.05 <0.05 <0.05	Report No. T-7095 Last treatment at "16 cm height".
Elfros, Saskatchewan Canada	EC	1	0.12 0.24 -	0.108 0.216 -	119	Mature seed	<0.05 <0.05 <0.05	Report No. T-7096 Last treatment at "16 cm height".
Manitoba, Canada	EC	1	0.12	0.096	88	Mature seed	<0.05	Report No. T-7097 Last treatment at "8 to 10 cm height".
			0.24	0.19			<0.05	
	Control						<0.05	
Elie, Manitoba, Canada	EC	1	0.105	0.105	84	Seed	<0.05	Tomen No. 489A
Indus, Alberta, Canada	EC	1	0.105	0.105	108	Seed	<0.05	Tomen No. 489A
Tverj region Russia	EC	-	0.075	0.04	80	Seed	<0.01	
			0.175	0.09	80		<0.01	
			0.250	0.13	80		<u><0.01</u>	
	Control				80		<0.01	
Kiev, Ukraine	EC	-	0.072 0.12 0.17 0.24 0.29	-	-	Seed	<0.01	

<u>Peanuts</u>. GAP was reported for Argentina, Australia, Bolivia, Israel, Taiwan and the USA, with maximum application rates of 0.09-0.336 kg ai/ha and PHIs of 40-70 days or not specified.

The residues from trials according to US GAP are underlined in Table 34.

Table 34. Supervised residue trials on peanuts.

Location	Application				PHI, days	Sample	Residue, mg/kg	Reference and Comment
	Form	No.	kg ai/ha	kg ai/hl				
Cordoba Provine, Argentina	EC	1	0.12	-	71	Kernels	<0.01	Tomen No. 527
			0.24	-			0.6	
Queensland, Australia	EC	1	0.24	0.20	49	Kernels + hulls	0.81	Report No. 223/AU/94/03/QU01
			0.48	0.40	-		1.3	
			Control				<0.02	
Lowood, Queensland,	EC	1	0.24	0.20	11 18 49	Foliage	17.0 11.0 2.3	Report No. 223/AU/94/03/QU01

Location	Application				PHI,	Sample	Residue,	Reference and Comment
	Form	No.	kg ai/ha	kg ai/hl	days		mg/kg	
Australia			0.48	0.40	11 18 49		32.0 24.0 5.6	Last treatment at “pegging and just covering between the rows. Height was 30 cm although the top of the plants had been slashed 3 weeks earlier to control the weed growth”.
			Control				<0.05	
Alabama, USA	EC	2	0.28	0.15	40	Kernels	1.3, 0.35	Report No. T-7408 Last treatment at “late pegging”.
						Hulls	0.18, 0.24	
						Vine[1]	0.27, 0.26	
						Hay	0.36, 0.38	
North Carolina, USA	EC	2	0.28	0.15	40	Kernels	1.3, 0.47	Report No. T-7408 Last treatment at “pod-fill”.
						Hulls	0.21, 0.60	
						Vine[1]	1.6, 1.7	
						Hay	2.6,2.5	
Georgia, USA	EC	2	0.28	0.14	25	Kernels	0.03, <0.05	Report No. T-7406 Growth stage at last treatment: 25 days: “early maturing” 40 days: “pegging”
						Hulls	0.40, 0.35	
						Vine[1]	0.58, 0.50	
						Hay	0.97, 1.2	
			0.28	0.14	40	Kernels	2.7, 0.15	
						Hulls	0.20, 0.17	
						Vine[1]	0.56, 0.21	
						Hay	0.43, 0.36	
Georgia, USA	EC	2	0.28	0.13-015	65	Kernels	0.08, 0.06	Report No. T-7406 Last treatment at “early pegging”.
						Hulls	0.08, <0.05	
						Vine[1]	0.60, 0.14	
						Hay	0.18, 0.22	
Texas, USA	EC	2	0.28	0.15	60	Kernels	0.39, 0.33	Report No. T-7407 Last treatment at “pegging”.
						Hulls	0.74, 0.46	
						Vine[1]	9.2, 13	
						Hay	1.0, <0.05	
Texas, USA	EC	2	0.28	0.15	20	Kernels	0.46, 0.51	Report No. T-7407 Growth stage at last treatment: 20 days: “pod fill” 35 days: “late pegging”
						Hulls	0.45, 0.59	
						Vine[1]	19, 21	
						Hay	1.4, 1.6	

Location	Application				PHI,	Sample	Residue,	Reference and Comment
	Form	No.	kg ai/ha	kg ai/hl	days		mg/kg	
			0.28	0.15	35	Kernels	<0.05, <0.05	
						Hulls	0.75, 0.59	
						Vine[1]	10, 12	
						Hay	1.9, 1.7	
Alabama, USA	EC	2	0.28	0.15	0	Plants[2]	3.8 7.4	Report No. T-7408
					12		0.67 0.89	Last treatment at "late pegging".
					19		0.53 0.73	
					26		0.39 0.45	
					33		0.35 0.40	
					40		0.19 0.22	
Georgia, USA	EC	2	1.4	0.75	40 + 3[3]	Kernels	3.5, 3.4	Report No. T-7409
						Meal (press-cake)	9.6	Last treatment at "pegging".
						Crude oil	1.7	
						Refined oil	0.32	
						Soap-stock	10	
, USA	EC	2	0.28	0.75	40	Kernels	1.8, 1.7	Report No. T-7409
						Hulls	0.31, 1.3	Last treatment at "pegging".
						Vine[1]	2.0, 1.8	
						Hay	2.2, 1.9	
			1.4	0.75	40	Kernels	3.9, 4.0	Mean of the duplicate analysis of the GAP results.
						Hulls	0.25, 0.62	was 1.75 mg/kg for Kernels and 0.81 mg/kg for the hull
						Vine[1]	20, 13	
						Hay	5.1, 13	
Georgia, USA	EC	2	0.28	0.15	41	Kernels	0.35, 0.33	Report No. V-1028A
						Hulls	0.17, 0.17	Last treatment at "pegging".
						Vine[1]	0.11, 0.11	Mean of the duplicate analysis of the GAP results.
						Hay	0.22, 0.23	was 0.34 mg/kg for Kernels and 0.17 mg/kg for the hull.
Texas, USA	EC	2	0.28	0.15	40	Kernels	0.60, 0.52	
						Hulls	0.31, 0.29	
						Vine[1]	2.1, 2.3	Mean of the duplicate analysis of the GAP results.

Location	Application				PHI,	Sample	Residue,	Reference and Comment
	Form	No.	kg ai/ha	kg ai/hl	days		mg/kg	
						Hay	0.71, 0.58	was 0.56 mg/kg for Kernels and 0.3 mg/kg for the hull.
Florida, USA	EC	2	0.25	0.15	40	Kernels	0.76, 0.82	Report No. V-1028C
						Hulls	0.23, 0.24	Last treatment at "pod development" stage.
						Vine[1]	0.26, 0.19	
						Hay	0.43, 0.42	Mean of the duplicate analysis of the GAP results. was 0.79 mg/kg for Kernels and 0.24 mg/kg for the hull.

[1]Described as immature plant
[2]Twelve individual plants collected
[3]Drying time

Alfalfa. GAP was reported for Argentina, Canada, Chile, Ecuador, Israel, Peru and the USA. The maximum application rates are 0.09-0.48 kg ai/ha with PHIs of 15-30 days or not specified.

The residues from trials according to Canadian and US GAP are underlined and double underlined respectively, in Table 35. US GAP allows 2 x 0.28 kg ai/ha. Where residues from single applications of 0.28 kg/ha exceeded those from the two higher levels have been regarded as being from treatment according to GAP.

Table 35. Supervised residue trials on alfalfa.

Location	Application				PHI,	Sample	Residue,	Reference and Comment
	Form	No.	kg ai/ha	kg ai/hl	days		mg/kg	
Yerong Creek, Australia	EC	1	0.12	0.15	0 3 14	Forage	6.6 1.2 0.13	Report No. 223/AU/95/08/SN01
			0.24	0.30	0 3 7 14		1.2 3.9 2.0 0.73	
Yerong Creek Australia	EC	1	0.48	0.60	0 14	Forage	27 0.60	Report No. 223/AU/95/08/SN01
Haight, Saskatoon, Saskachawan, Canada	EC	1	0.045	0.04	30	Forage	<0.02 <0.02	Report No. 95-040.DC Last treatment at "2-3 foliate".
			0.09	0.08			<0.02 <0.02	
							<0.02 <0.02	
			0.045	0.04	44		<0.02 <0.02	
			0.09	0.08			<0.02 <0.02	
							<0.02 <0.02	
Koral, Saskatoon, Saskachawan, Canada	EC	1	0.045	0.04	34	Forage	<0.02 <0.02	Report No. 95-040.DC Last treatment at "5-6 trifoliate".
			0.09	0.08			<0.02 <0.02	
			0.18	0.16			<0.02 <0.02	
			0.045	0.04	45		<0.02 <0.02	

Location	Application				PHI, days	Sample	Residue, mg/kg	Reference and Comment
	Form	No.	kg ai/ha	kg ai/hl				
			0.09	0.08			<0.02 <0.02	
			0.18	0.16			<0.02 <0.02	
Portage, Manitoba, Canada	EC	1	0.045	0.04	31	Forage	<0.02 <0.02	Report No. 95-040.DC Last treatment at "3-4 trifoliate".
			0.09	0.08			<0.02 <0.02	
			0.09	0.08			<0.02 <0.02	
			0.045	0.04	46		<0.02 <0.02	
			0.09	0.08			<0.02 <0.02	
			0.09	0.08			<0.02 <0.02	
Korael, Saskatoon, Saskawan, Canada	EC	1	0.045	0.04	47	Forage	<0.02 <0.02	Report No. 95-040.DC Last treatment at "3 trifoliate".
			0.09	0.08			<0.02 <0.02	
			0.18	0.16			<0.02 <0.02	
			0.045	0.04	29		<0.02 <0.02	
			0.09	0.08			<0.02 <0.02	
			0.18	0.16			<0.02 <0.02	
Oakbluff, Manitoba, Canada	EC	1	0.045	0.04	30	Forage	<0.02 <0.02	Report No. 95-040.DC Last treatment at "2-3 trifoliate".
			0.09	0.08			<0.02 <0.02	
			0.09	0.08			<0.02 <0.02	
			0.045	0.04	45		<0.02 <0.02	
			0.09	0.08			<0.02 <0.02	
			0.09	0.08			<0.02 <0.02	
Guelph, Ontario, Canada	EC	2	0.09	0.045	30	Forage	<0.02 <0.02	Report No. 95-040.DC Last treatment at "2-5 trifoliate".
					45		<0.02 <0.02	
Campbellville, Ontario, Canada	EC	2	0.09	0.045	30	Forage	<0.02 <0.02	Report No. 95-040.DC Last treatment at "0-4 trifoliate".
					46		<0.02 <0.02	
Airdrie, Alberta, Canada	EC	2	0.09	0.08	30	Forage	0.027 0.022	Report No. 95-040.DC Last treatment at "1-3 trifoliate". Mean of duplicate analyses for the GAP results were 0.0245 mg/kg.
		1			45		<0.02 <0.02	
Alfalfa, Minnesota, USA	EC	1	0.28	0.14	21	Forage	1.4, 1.5	Report No. T-7397 Growth stage at last treatment:
		2	0.28	0.14 to 0.14	20		0.55 0.50	

Location	Application				PHI,	Sample	Residue,	Reference and Comment
	Form	No.	kg ai/ha	kg ai/hl	days		mg/kg	
		2	0.28	0.14 to 0.15	61		<0.01 <0.01	21 days: "vegetative" 20 days: "50% bloom" 61 days: "50% bloom" Mean residue of duplicate analyses for GAP results was 0.525 mg/kg.
Nebraska, USA	EC	1	0.28	0.15	20 57	Forage	0.87 0.77	Report No. T-7398
							<0.01 <0.01	Last treatment at "vegetation".
		2	0.28	0.15	20		0.61 0.63	Mean residue of duplicate analyses for GAP results was 0.62 mg/kg.
		3			20		0.35 0.31	
					61		<0.10 <0.10	
Minnesota, USA	EC	1	0.28	0.14	21+4*	Hay	3.2 2.4	Report No. T-7397
		2		0.14 to 0.15	20+7*		1.0 1.1	*Drying time. Last treatment at "vegetative, 50% bloom".
					61+6*		<0.10 0.14	
Nebraska, USA	EC	1	0.28	0.15	20+3*	Hay	2.3 1.8	Report No. T-7398
					57+4*		0.40 <0.1	*Drying time. Last treatment at "vegetation".
Nebraska, USA	EC	2	0.28	0.15	20+2*	Hay	1.2 1.4	Report No. T-7398
		3			20+2*		0.56 0.47	*Drying time. Last treatment at "vegetation".
					61+6*		<0.10 0.16	
Alfalfa, Nebraska, USA	EC	1	-	0.16	20	Hay	1.1	
		2	0.28	0.15 to 0.16	20		1.6 1.6	Report No. T-7399
					63		<0.1 <0.1	Last treatment at "vegetative".
South Dakota, USA	EC	1	0.16	0.15	20	Hay	1.1 1.2	Report No. T-7400
		2			20		0.96 0.98	Last treatment at "vegetative".
					67		<0.1 <0.1	
Alfalfa, Michigan, USA	EC	1	175	0.15	20+3*	Hay	2.6	
		2	175- 187	0.15	20+3*		3.0 3.4	Report No. T-7399 *Drying time. Last treatment at "vegetative".
					63+3*		<0.10 <0.10	
South Dakota, USA	EC	1	0.16	0.15	20+2*	Hay	3.1 3.4	Report No. T-7400
		2			20+1*		1.4 1.6	*Drying time. Last treatment at "vegetative".
					67+3*		<0.1 <0.1	
North Dakota, USA	EC	1	0.28	0.16	15	Forage	1.4 1.2	Report No. T-7401
		2	0.28	0.15- 0.16	15		5.7 5.0	
		1	0.28	0.15	19	Forage	0.90 1.3	

Location	Application				PHI, days	Sample	Residue, mg/kg	Reference and Comment
	Form	No.	kg ai/ha	kg ai/hl				
		2	0.28	0.15	21		1.3 1.1	
North Dakota, USA	EC	1	0.28	0.16	15+8*	Forage	2.3 2.3	Report No. T-7401
		2	0.28	0.15-0.16	15+10*		2.1 3.7	*Drying time.
		1	0.28	0.15	19+8*	Forage	1.7 2.2	
		2	0.28	0.15	21+10*		1.2 1.4	
Wisconsin, USA	EC	1	0.28	0.14	15	Forage	2.8 3.0	Report No. T-7402
		2	0.28	0.14-0.15	15		1.7 2.3	Mean residue of duplicate analyses = 2.0 mg/kg
					44		<0.2 <0.2	
		1	0.28	0.14	20	Forage	0.52 0.72	
		2			20		1.2 1.5	Mean residue of duplicate analyses = 1.35 mg/kg
					49		<0.1 <0.1	
Wisconsin, USA	EC	1	0.28	0.14	15+4*	Hay	4.0 3.8	Report No. T-7402
		2	0.28	0.14-0.15	15+4*		2.0 1.7	Mean residue of duplicate analyses = 1.85 mg/kg
					44+5*		<0.2 <0.2	
		1	0.28	0.14	20+4*	Forage	1.5 1.3	
		2			20+5*		1.6 1.9	*Drying time.
					49+5*		<0.1 <0.1	
Iowa, USA	EC	1	0.28	0.15	15	Forage	1.4 1.5	Mean residue of duplicate analyses = 1.45 mg/kg
		2			17		1.2 1.1	
					63		<0.2 <0.2	Report No. T-7403
		1	0.28	0.15	20	Forage	0.82 0.73	
		2			22		0.91 0.79	Mean residue of duplicate analyses = 0.85 mg/kg
					68		<0.1 <0.1	
Iowa, USA	EC	1	0.28	0.15	15+2*	Hay	3.1 2.2	Mean residue of duplicate analyses = 2.65 mg/kg
		2			17+2*		1.7 2.2	
					63+3*		<0.2 <0.2	Report No. T-7403
		1	0.28	0.15	20+2*	Hay	1.4 1.5	*Drying time.
		2			22+2*		1.6 1.3	
					68+3*		<0.1 <0.1	
California,	EC	1	0.28	0.15	15	Forage	1.5 1.3	Mean residue of duplicate analyses = 1.4 mg/kg

Location	Application				PHI,	Sample	Residue,	Reference and Comment
	Form	No.	kg ai/ha	kg ai/hl	days		mg/kg	
USA		2			15		0.59 0.81	Report No. T-7404
					43		<0.2 <0.2	
					71		<0.02 <0.02	
		1	0.28	0.15	20	Forage	0.44 0.89	Mean residue of duplicate analyses = 0.67 mg/kg
		2			20		0.11 <0.10	
					48		<0.10 <0.10	
					76		<0.10 <0.10	
California, USA	EC	1	0.28	0.15	15+6*	Hay	4.4 4.3	Mean residue of duplicate analyses = 4.35 mg/kg
		2			15+2*		2.4 2.8	
					43+5*		0.13 <0.10	Report No. T-7404
					71+5*		0.64 0.36	
		1	0.28	0.15	20+6*	Hay	1.2 1.6	*Drying time.
		2			20+12*		0.56 0.71	
					48+5*		<0.10 <0.10	
					76+5*		0.12 <0.10	
Wisconsin, USA	EC	1	0.28	0.14	15	Forage	1.4 1.3	Mean residue of duplicate analyses = 1.35 mg/kg
		2	0.28	0.13-0.14			0.86 0.88	Report No. T-7477
New York, USA	EC	1	0.28	0.15	15	Forage	2.6 2.5	Mean residue of duplicate analyses = 2.55 mg/kg
		2					2.1 1.7	Report No. T-7478
Wisconsin, USA	EC	1	0.28	0.14	15+6*	Hay	2.7 2.7	Report No. T-7477
		2	0.28	0.13-0.14	15+5*		1.5 1.8	*Drying time.
New York, USA	EC	1	0.28	0.15	15+3*	Hay	3.5 3.2	Report No. T-7478
		2			15+2*		4.5 4.4	Mean residue of duplicate analyses = 4.45 mg/kg *Drying time.
California, USA	EC	1	0.28	0.61	15	Forage	0.29 0.25	Mean residue of duplicate analyses = 0.27 mg/kg
		2	0.28	0.58-0.61			0.19 0.13	Report No. T-7479 * Aerial application.
Idaho, USA	EC	1	0.28	0.13	15	Forage	2.1 1.8	Report No. T-7480
		2					2.8 3.2	Mean residue of duplicate analyses = 3.0 mg/kg
California, USA	EC	1	0.28	0.61	15+3*	Hay	0.45 0.76	Mean residue of duplicate analyses = 0.61 mg/kg
		2	0.28	0.58-0.61	15+6*		0.58 0.47	Report No. T-7479 *Drying time.
Idaho, USA	EC	1	0.28	0.13	15+5*	Hay	4.6 5.6	Report No. T-7480

Location	Application				PHI,	Sample	Residue,	Reference and Comment
	Form	No.	kg ai/ha	kg ai/hl	days		mg/kg	
		2			15+6*		9.2 8.6	Mean residue of duplicate analyses = 8.9 mg/kg *Drying time.
Idaho, USA	EC	1	0.28	0.13	15+11*	Processed	3.8	Report No. T-7480
		2			15+16*	Hay	6.1	*Drying time

White clover. GAP for clover was reported for Israel and New Zealand. Both maximum application rates are 0.12 kg ai/ha with a PHI of 63 days in New Zealand and no specified in Israel.

The two residues in trials complying with New Zealand clover GAP are underlined in Table 36 but the samples analysed were described as "young plants" and "silage".

Table 36. Supervised residue trials on clover.

Location	Application				PHI,	Sample	Residue,	Reference and Comment
	Form	No.	kg ai/ha	kg ai/hl	days		mg/kg	
White Clover,	EC	1	0.12	0.04	71	Young	0.07	
Methven,			0.36	0.12		Plant	0.24	Report No. 880410
Canterbury, New Zealand	Control						<0.03	Last treatment at “vegetative”.
Clover,	EC	1	0.12	0.04	62	Silage	0.26	
Methven,			0.36	0.12			0.93	Report No. 880410
Canterbury, New Zealand	Control						0.04	Last treatment at “vegetative”.
Clover, Oregon, USA	EC	1	0.28	-	15	Forage	5.8 6.1	Report No. 06218.95-OR29
		1	0.28	-	15+7*	Hay	15 12	*Drying time.
Clover, Oregon, USA	EC	1	0.28	-	15	Forage	5.2 4.6	Report No. 06218.95-OR30
		1	0.28	-	15+7*	Hay	11 12	*Drying time.
Clover, Oregon, USA	EC	1	0.28	-	15	Forage	3.2,3.2 3.2,3.0	Report No. 06218.95-OR31
		1	0.28	-	20	Hay	11,11 12,12	

Field peas (dry). GAP for field peas was reported for Australia and Canada, for chick peas for Australia. The maximum application rate in Canada is 0.09 kg ai/ha with a PHI of 75 days. The maximum rate reported by the Australian government, supported by a product label, was 0.06 kg ai/ha with no PHI and differed from that reported by the manufacturer.

The residues from trials considered to comply with Canadian GAP are underlined, and the single residue which was considered to result from Australian GAP is double underlined. All residues in the Australian trials were <0.1 mg/kg, even those from exaggerated doses.

Table 37. Supervised residue trials on field peas.

Crop, Location	Field/ Prot-ected	Application				PHI, days	Sample	Residue, mg/kg	Reference and Comment
		Form	No.	kg ai/ha	kg ai/hl				
Peas (field), Elmore, Victoria, Australia	-	EC	1	0.06	0.045	110	Grain (dried seed)	<0.10	Report No. T-7234
				0.12	0.09			<0.10	Last treatment at 4 to 8 leaves
				0.24	0.18		Straw	<0.10	
				0.06	0.045			<0.10	
				0.12	0.18			<0.10	
				0.24	0.24			<0.10	
Peas (protein) B9230 Mlelle, Belgium	-	EC	1	0.09	0.012	41	Seed	<0.025 <0.025	Report No. 92CLEPOI 1
				0.18	0.024			<0.025 <0.025	Last treatment at 12-15 cm.
Peas (field), Okotoks, Alta, Canada	-	EC	2	0.09	0.08	75	Seed	0.59 <0.02	Residues from duplicate analyses too different to use.
						90		<0.02 <0.02	Report No. 95.036.DC Growth stage at last treatment: 75 days: flowering, height 50-60 cm 90 days: 6 node leaf, height 20-24 cm
Peas (field), Rowatt, Saskatoon, Canada	-	EC	1	0.045	0.04	77	Seed	0.084 <0.02	Report No. 95.036.DC
				0.09	0.08			<0.02 0.059	Growth stage at last treatment:
			2	0.09	0.08			0.14 0.02	Mean residue of duplicate analyses = 0.08 mg/kg
			1	0.045	0.04	62	Seed	0.01 0.21	77 days: 4-8 expanded leaves 62 days: flowering, pod
				0.09	0.08			0.64 0.59	formation
			2	0.09	0.08			0.83 0.46	Mean residue of duplicate analyses = 0.65 mg/kg
Peas (field), Korae, Saskatoon, Canada	-	EC	1	0.045	0.04	51	Seed	0.27 0.17	Report No. 95.036.DC
				0.09	0.08			0.50 0.47	Growth stage at last treatment:
				0.18	0.16			0.50 0.33	51 days: 50 cm height 37 days: 70 cm height
			1	0.045	0.04	37	Seed	0.43 0.38	
				0.09	0.08			0.70 0.94	
				0.18	0.16			2.2 1.6	
Peas (field), Airdrie, Alberta, Canada	-	EC	1	0.045	0.04	75	Seed	0.16 0.18	Report No. 95.036.DC
				0.09	0.08			0.24 0.38	Mean residue of duplicate analyses = 0.31 mg/kg
			2	0.09	0.16			0.70 1.2	Growth stage at last treatment:
			1	0.045	0.04	60	Seed	0.38 0.54	75 days: flowering 60 days: pod filling
				0.09	0.08			1.5 0.69	
			2	0.09	0.16			2.0 1.5	Mean residue of duplicate analyses = 1.75 mg/kg
Peas (field) Richardson,	-	EC	1	0.09	0.08	73	Seed	0.05 0.06	Report No. 95.036.DC

Crop, Location	Field/ Prot-ected	Application Form	No.	kg ai/ha	kg ai/hl	PHI, days	Sample	Residue, mg/kg	Reference and Comment
Saskatoon, Canada				0.18	0.16			0.07 0.10	Growth stage at last treatment:
				0.09	0.08	58	Seed	0.71 0.81	73 days: 3-5 expended leaves 58 days: 10-12 expanded
				0.18	0.16			1.9 2.0	leaves and flowering
Peas (field) Polar Point, Manitoba, Canada	-	EC	1	0.18	0.16	82	Seed	0.19 0.15	Report No. 95.036.DC
						67		0.43 0.66	Growth stage at last treatment: 82 days: 3-6 nodes 67 days: 15 nodes
Peas (field), Polar Point, Manitoba, Canada	-	EC	1	0.045	0.04	85	Seed	<0.02 <0.02	Report No. 95.036.DC
				0.09	0.08			<0.02 <0.02	Growth stage at last treatment:
				0.18	0.16			<0.02 <0.02	85 days: 3-6 leaf 71 days: 15-20 cm
				0.045	0.04	71	Seed	<0.02 <0.02	
				0.09	0.08			<0.02 <0.02	
				0.18	0.16			<0.02 <0.02	
Peas (field), Oak bluff, Manitoba, Canada	-	EC	1	0.045	0.04	93	Seed	<0.02 <0.02	Report No. 95.036.DC
				0.09	0.08			<0.02 <0.02	Growth stage at last treatment:
				0.18	0.16			<0.02 <0.02	93 days: 6-12 cm 78 days: 10-20 cm
				0.045	0.04	78	Seed	<0.02 <0.02	
				0.09	0.08			<0.02 <0.02	
				0.18	0.16			<0.02 <0.02	
Peas (field), Edenuold, Saskatoon, Canada	-	EC	1	0.045	0.04	80	Seed	<0.02 <0.02	Report No. 95.036.DC
				0.09	0.08			<0.02 0.18	Residues from duplicate analyses too different to use. Growth stage at last treatment:
			2	0.09	0.08			0.14 0.02	80 days: 5-6 expanded leaves 68 days: flowering
			1	0.045	0.04	68	Seed	<0.02 <0.02	
				0.09	0.08			0.20 0.15	Mean residue of duplicate analyses =0.175 mg/kg
			2	0.09	0.08			0.34 0.61	
Peas (field), Aubeterre/ Pont Ste. Marie, France	-	EC	1	0.18	0.045	67	Seed	<0.03 0.05	Report No. TE-2301
				0.48	0.12			0.11 0.06	Last treatment at 15-20 cm high.
				0.96	0.24			0.29 0.18	
Peas (field), Voue/ Pont Ste. Marie, France	-	EC	1	0.18	0.045	72	Seed	0.03 <0.03	Report No. TE-2302
				0.48	0.12			0.08 0.04	Last treatment at 20 cm high.

Crop, Location	Field/ Prot-ected	Application				PHI, days	Sample	Residue, mg/kg	Reference and Comment
		Form	No.	kg ai/ha	kg ai/hl				
				0.96	0.24			0.15 0.12	
Peas (field), Warlus/ Airaines, France	-	EC	1	0.18	0.045	82	Seed	0.06 0.04	Report No. TE-2303
				0.48	0.12			0.20 0.28	Last treatment at 20 cm high
				0.96	0.24			0.75 0.43	
Peas (field), Flacey/ Bonneval, France	-	EC	1	0.18	0.045	72	Dry seed	<0.03 <0.03	Report No. TE-2304
				0.48	0.12			0.09 0.13	Last treatment at 20 cm high.
				0.96	0.24			0.10 0.17	
Peas (field), Amiens, France	-	EC	1	0.48 0.96	0.12 0.24	80	Dry seed	<0.03 0.04 0.05 0.08	Report No. TE-2305 Last treatment at 20 cm high.
Peas (field), Marboue Chateaudun, France	-	EC	1	0.18 0.48 0.96	0.45 0.12 0.24	79	Dry seed	<0.03 <0.03 0.04 <0.03 0.14 0.05	Report No. TE-2306 Last treatment at 15 cm high.
Peas (field), Thurston, Suffolk, UK	-	F	1	0.36	0.12	53	Pea green	<0.03 <0.03	Report Nos. 0432-88, 0434-88,
				0.72	0.24			0.08 0.10	0552-88, 0556-88
				0.36	0.12	53	Husk	<0.03 <0.03	Last treatment at 4-5 leaves
				0.72	0.24			<0.03 <0.03	
				0.36	0.12	85		<0.03 0.05	
				0.72	0.24			0.07 0.12	
Peas (dry), Washington, USA	F	EC	2	0.28	-	21	Dried peas	4.6, 3.5 3.7, 3.3 4.8, 4.1 5.8, 4.3	Report No. 5204.93-WA*07
Peas (dry), Washington, USA	F	EC	2	0.28	-	20	Dried peas	6.9, 7.0 6.5, 6.1 6.6	Report No. 5204.94-WA*22
Peas (dry), California, USA	F	EC	2	0.28	-	20	Dried peas	0.48 0.68	Report No. 5204.94 CA93

Peas. GAP for peas was reported for Belgium, the Czech Republic, Israel, New Zealand and Spain, and "proteaginous peas" for France. The maximum application rates are 0.06-0.36kg ai/ha with PHIs of 30 or 60 days, or not specified. None of the trials complied with the reported GAP.

Table 38. Supervised residue trials on peas.

Location	Application				PHI, days	Sample	Residue, mg/kg	Reference and Comment
	Form	No.	kg ai/ha	kg ai/hl				
Rawora Canterbury, New Zealand	EC	1	0.24	0.08	43	Peas + pods	0.29	Report No. 8804-09 Last treatment at early flowering.
			0.48	0.16			0.45	
			0.72	0.024			0.34	
	Control						<0.03	
Rawora Canterbury, New Zealand	EC	1	0.24	0.08	43	Silage	0.47	Report No. 8804-09 Last treatment at early flowering.
			0.48	0.16			2.24	
			0.72	0.024			0.93	
			-	-	-		0.07	
Michigan, USA	EC	2	0.28	-	22		6.19 7.25	PR No. 05202
		2	0.28	-	22	+ Pods	3.13 3.88	
Washington, USA	EC	2	0.28	-	21	+ Pods	0.84 0.70 0.88 0.82	PR No. 05202
New York, USA	EC	2	0.28	-	20	+ Pods	0.42 0.71 0.74 0.45	PR No. 05202
Wisconsin, USA	EC	2	0.56	-	20	+ Pods	1.24 1.49 1.39 1.69	PR No. 05202
Illinois, USA	EC	2	0.56	-	20	+ Pods	2.22 2.18 1.96 1.87	PR No. 05202
New York, USA	EC	-	0.28	-	-	+ Pods	<0.10	PR No. 5202.92
		2					0.78 0.84	
Wisconsin, USA	EC	-	0.28	-	-	+ Pods	<0.10	PR No. 5202.92
		2					1.59 2.18	
Washington, USA	EC	-	0.28	-	-	+ Pods	<0.10	PR No. 5202.92
		2					1.32 1.63	

Fodder beet. GAP was reported for Belgium, the Czech Republic, Germany, Italy, Russia and Switzerland. The maximum application rates are 0.14-0.36 kg ai/ha with PHIs of 60-90 days, or not specified.

The residues in the French trials which complied with Belgian GAP are underlined in Table 40.

Table 39. Supervised residue trials on fodder beet.

Location	Application				PHI,	Sample	Residue,	Reference and Comment
	Form	No.	kg ai/ha	kg ai/hl	days		mg/kg	
St. Pardoux du Breuilh, France	EC	1	0.36 0.72	0.072 0.14	129	Top	<0.03 <0.03	Report No. TE-2161
			0.36 0.72	0.072 0.14		Root	<0.03 <0.03	
Veigne, France	EC	1	0.36 0.72	0.06 0.12	109	Top	<0.03 <0.03	Report No. TE-2166
			0.36 0.72	0.06 0.12		Root	<0.03 <0.03	
Bernapre, France	EC	1	0.18 0.36 0.48 0.96	0.045 0.09 0.12 0.24	102	Top	<0.03 <0.03 <0.03 <0.03	Report No. TE-2298
			0.18 0.36 0.48 0.96	0.045 0.09 0.12 0.24		Root	<0.03 <0.03 <0.03 <0.03	

Peppers. No GAP for either sweet or Chilli peppers was reported to the current or the 1994 Meeting. The results of trials on the two types of pepper are shown in Tables 40 and 41.

Table 40. Supervised residue trials on sweet peppers.

Location	Field/ Prot-ected	Application				PHI, days	Sample	Residue, mg/kg	Reference and Comment
		Form	No.	kg ai/ha	kg ai/hl				
Borgo Piave, Italy	F	EC	1	0.24	0.04	18	Mature fruit	0.080 0.116	Report No. 0266-90
						28		0.030 0.075	
						38		0.041 0.049	
Bell peppers, New York, USA		EC	2	0.28		19	Mature fruit	0.89 0.68	PR No. 5226.93
Bell peppers, California, USA		EC	2	0.28		-	Mature fruit	0.62 0.58	PR No. 5226.94
Bell peppers, Florida, USA		EC	2	0.28		21	Mature fruit	0.54 0.51	PR No. 5226.93
Bell peppers, North Carolina, USA		EC	2	0.28		19	Mature fruit	0.45 0.38	PR No. 5226.93
Bell peppers, Georgia, USA		EC	2	0.28		20	Mature fruit	0.11 0.14	PR No. 5226.93
Bell peppers, Texas, USA		EC	2	0.28		21	Mature fruit	0.29 0.34	PR No. 5226.93

Table 41. Supervised residue trials on chilli peppers.

Location	Field/ Prot-ected	Application				PHI days	Sample	Residue, mg/kg	Reference and Comment
		Form	No.	kg ai/ha	kg ai/hl				
California, USA	-	EC	2	0.28		NA	Mature fruit	0.91 0.82	
New Jersey, USA	-	EC	2	0.28		22	Mature fruit	0.97 0.87	
Georgia, USA	-	EC	2	0.28		20	Mature fruit	0.12 0.12	
Texas, USA	-	EC	2	0.28		21	Mature fruit	0.40 0.39	
Florida, USA	-	EC	2	0.28		21	Mature fruit	0.43 0.48	
New Jersey, USA	-	EC	2	0.28		22	Mature fruit	0.97 0.87	
Zucchini, Borgo Piave, Italy	F	EC	1	0.24	0.04	28 33 43	Mature fruit	<0.03 <0.03 <0.03	

Livestock feeding trial

In the cow feeding study, reviewed in the 1994 monograph, fourteen dairy cows were used to show the distribution of clethodim residues in bovine tissues. Two were used as controls and the others were split into three groups of four. The treated cows received a daily capsule containing clethodim (5%) and clethodim sulfoxide (95%) for 28 days at 10, 30 and 100 nominal ppm in the feed. Table 42 reproduces the results reported in the 1994 monograph.

Table 42. Residues of ^{14}C expressed as clethodim in bovine tissues

Compound	Feeding level (nominal in diet)	Liver, mg/kg	Kidney, mg/kg	Muscle, mg/kg	Fat, mg/kg
DME	0	<0.05	<0.05	<0.05	<0.05
	10 ppm	0.059	0.051	<0.05	<0.05
	30 ppm	0.119	0.170	<0.05	0.052
	100 ppm	0.445	0.538	0.070	0.153
S-methyl DME	0	<0.05	<0.05	<0.05	<0.05
	10 ppm	<0.05	<0.05	<0.05	<0.05
	30 ppm	<0.05	<0.05	<0.05	<0.05
	100 ppm	0.087	0.078	<0.05	<0.05
DME-OH	0	<0.05	<0.05	<0.05	<0.05
	10 ppm	<0.05	<0.05	<0.05	<0.05
	30 ppm	<0.05	<0.05	<0.05	<0.05
	100 ppm	<0.05	<0.05	<0.05	<0.05

(Tomen Agro, 1997; Weissenburger *et.al.,* 1989)

FATE OF RESIDUES IN STORAGE AND PROCESSING

In processing

Cotton seed. The 1994 monograph described a study in 1987 in which cotton treated at eight times the normal rate in Mississippi was processed in Texas. Table 42 is adapted from the Table in the 1994 monograph but also includes residues in refined oil.

Table 42. Effect of processing on clethodim residues in cotton seed.

Sample	Residues, mg/kg[1]	Processing factor
Fuzzy cotton seed (unprocessed)	0.80	1.0
Meal	1.35	1.69
Hulls	<0.98	<1.23
Crude oil	<0.18	<0.23
Refined oil	<0.08	<0.10
Soapstock	<0.85	<1.06
Delinted cotton seed	0.88	1.1

[1]Sum of DME and DME-OH expressed as clethodim, means of 3 results

NATIONAL MAXIMUM RESIDUE LIMITS

The following national MRLs were reported to the Meeting.

Country	Crop	MRL, mg/kg	Reference
Argentina	Alfalfa (forage)	10	Tomen Agro, 1997
	Alfalfa (hay)	15	
	Cotton seed	0.5	
	Peanut	0.5	
	Soya bean (grain)	0.5	
	Soya bean (straw)	3.0	
	Sunflower (seed)	0.5	
	Sunflower (straw)	3.0	
Australia[1]	Asparagus	1	Coleman, 1996
	Beans, except broad and soya	0.1*	
	Broad bean	0.1*	
	Brassica vegetables	0.2	
	Cucurbits	0.1*	
	Celery	0.1	
	Cotton seed	0.2	
	Edible offal	0.05*	
	Eggs	0.05*	
	Endive	0.05	
	Fennel, Bulb	0.01*	
	Fruiting veg., cucurbits	0.1*	
	Leek	0.01*	
	Lettuce, Head	0.1	
	Lettuce, Leaf	0.1	
	Lupin, dry	0.2	
	Meat, mammalian	0.05*	
	Milks	0.05*	
	Onion, bulb	0.3	
	Peanut	2	
	Peanut oil, crude	2	
	Peas	0.1*	
	Poppy seed	0.2	
	Poultry, edible offal	0.05*	
	Poultry meat	0.05*	
	Pulses (except lupin dry)[2]	0.1*	
	Rape seed	0.5	
	Root and tuber veg.	1	
	Spinach	0.1*	
	Strawberry	0.1	
	Sunflower seed	0.1*	
	Tomato	0.1	

Country	Crop	MRL, mg/kg	Reference
Belgium	Beans	0.1	Tomen Agro, 1997
	Fodder beet	0.05	
	Onions	0.05	
	Peas	0.1	
	Potatoes	0.1	
	Sugar beet	0.05	
Brazil	Soya bean	1.0	Tomen Agro, 1997
	Garlic	0.05	
	Onion	0.05	
	Tomato	0.05	
	Dry bean	0.05	
	Potato	0.05	
	Carrot	0.05	
	Cotton	0.05	
Canada	Alfalfa seedlings	0.1	Tomen Agro, 1997
	Canola	0.1	
	Field peas	0.5	
	Flax (including low linolenic acid	0.3	
	varieties)	0.5	
	Lentil	0.5	
	Potato	10	
	Soya bean		
France	Proteaginous peas	0.1	Tomen Agro, 1997
	Sugar beet	0.05	
	Sun flower	0.1	
Italy	Sugar beet	0.2	Tomen Agro, 1997
	Fodderbeet	0.2	
	Tomato	0.2	
	Soya bean	1.0	
Mexico	Soya bean	10	Tomen Agro, 1997
Netherlands	All commodities[3]	0.03* - 0.05*	Olthof, 1997
New Zealand	White clover	<0.1	Tomen Agro, 1997
	Pea	<0.1	
	Lentil	<0.1	
	Oilseed rape	<0.1	
	Orchard crops	<0.1	
	Vegetables	<0.1	
Peru	Orange	5	Tomen Agro, 1997
	Apple	5	
	Bean	10	
	Alfalfa	1	
	Cotton	5	
Russia	Carrot	0.1	Tomen Agro, 1997
	Sugar beet	0.1	
	Fodder beet	0.1	
	Red beet	0.1	
	Onion	0.1	
	Soya bean	0.1	
	Flax seed	0.1	
	Potato	0.2	
Spain	Sunflower	0.05	Tomen Agro, 1997
	Beans	0.1	
	Field peas	0.1	
	Tomato	0.1	
	Onion	0.05	
	Garlic	0.05	
	Flax seed	0.1	
	Soya bean	0.3	
	Potato	0.05	
Switzerland	Potato	0.1	Tomen Agro, 1997
	Sugar beet	0.05	
	Fodder beet	0.05	
	Vegetables	0.1	

Country	Crop	MRL, mg/kg	Reference
Ukraine	Sugar beet Fodder beet Red beet Potato Flax seed Flax seed oil Onion Carrot Soya bean	0.1 0.1 0.1 0.2 0.1 0.1 0.1 0.1 0.1	Tomen Agro, 1997
UK		No MRLs established	UK, 1997
USA	Potato[4] Soya bean Soya bean soapstock Cotton seed Cotton seed meal Dry onions Garlic Shallots Sugar beet (tops) Sugar beet (roots) Sugar beet (molasses) Alfalfa (hay)[4] Alfalfa (forage)[4] Dry bean seed[4] Dry bean forage[4] Dry bean straw/hay[4] Peanut Kernels [4] Peanut hulls[4] Peanut hay[4] Peanut meal[4] Tomato[4]	0.5 10 15 1.0 2.0 0.2 0.2 0.2 0.5 0.2 2.0 15 10 2.0 5.0 7.0 3.0 2.0 5.0 10.0 1.0	Tomen Agro, 1997
Uzbekistan	Onion Carrot Soya bean Beet Potato	0.1 0.1 0.1 0.1 0.2	Tomen Agro, 1997

[1]In Australia residues arising from the use of clethodim are covered by MRLs for sethoxydim, defined as "sum of sethoxydim and metabolites containing the 5-(2-ethylthiopropyl) cyclohexene-3-one and 5-(2-ethylthiopropyl)-5-hydroxycyclohexene-3-one moieties and their sulfoxides and sulfones, expressed as sethoxyquin"

[2]Includes field peas, chickpeas, fava beans

[3]No specific MRLs have been established in The Netherlands so the limit of determination (0.03-0.05 mg/kg) applies

[4]Temporary

[5]Tolerance Petition at USA EPA awaiting approval

[5]The MRLs listed under reference Tomen Agro, 1997 were described as either "established or proposed"

APPRAISAL

Clethodim was first evaluated by the 1994 JMPR which recommended a number of MRLs. At the 28th Session of the CCPR opinions were expressed that the 1994 monographs were unclear and over-summarized. Detailed written comments were submitted by some governments, to which the manufacturer has provided an item-by-item response.

In response to the submitted comments, the Meeting evaluated the previously reviewed data in more detail. The comments and the responses of the Meeting are given below.

Metabolism

(i) "*There are no data on the kinds and quantities of metabolites in the goat study. Therefore, it cannot be established whether the definition of residues for cattle kidneys, liver, meat, milk is acceptable*."

The study was re-evaluated. Milk contained 0.02-0.05 mg/kg clethodim equivalents and the highest tissue concentrations were found in the liver (0.414 mg/kg clethodim equivalents) and kidneys (0.378 mg/kg). In milk, the extracted radioactivity was mostly associated with lactose and clethodim sulfoxide. In the blood and tissues the major compounds were clethodim sulfoxide (33-52% of the substrate radioactivity) and *S*-methyl-clethodim sulfoxide (6-37%). Clethodim was only found above 4% of the substrate radioactivity in blood (28%) and liver (28%).

(ii) "*There are no data on quantities concerning the metabolism in plants, i.e. there are no data indicating the determined quantities of the metabolites referred to*."

The metabolism studies on carrots, soya beans and cotton have been re-evaluated and information on the quantities of individual metabolites is provided in the monograph. The identified metabolites were clethodim sulfoxide, clethodim sulfone, the imine sulfoxide and sulfone, and 5-hydroxyclethodim sulfoxide and sulfone. Clethodim was not present or was found at very low levels. Clethodim sulfoxide and the imine sulfoxide were the major metabolites in both leaves and edible parts.

Methods of analysis

(iii) "*According to the method of residue analysis referred to, two compounds have to be determined simultaneously, therefore it is doubtful whether a determination limit of 0.05 mg/kg for both compounds is practicable, also in view of the dissolution of isomers into several peaks which is possible under certain circumstances.*"

In the "common moiety" method referred to, clethodim and its metabolites containing the 2-cyclohexen-1-one moiety are determined as dimethyl 3-[2-(ethylsulfonyl)propyl]pentanedioate (DME) and its 3-hydroxy analogue (DME-OH) as described in the 1994 monograph. The manufacturer has supplied several typical chromatograms which showed two resolved peaks with some tailing, for labelled DME and DME-OH standards in clean solvent at concentrations of 0.5 μg/ml and 0.75 μg/ml or 10 or 25 ng. The reports of the trials included data showing acceptable recoveries (generally 70-110%) for a range of crop commodities; these were usually at fortification levels above 0.2 mg/kg each of clethodim, clethodim sulfoxide and 5-hydroxyclethodim sulfone. Some acceptable recoveries of clethodim sulfoxide and sulfone at 0.05 mg/kg were submitted, e.g. for dried peas. Some of the residue trials (e.g. on succulent beans) reported “limits of quantification” of 0.1 mg/kg.

A revised confirmatory method was submitted to the present Meeting. The recovery data for the revised method, which is necessary to differentiate clethodim from related compounds such as sethoxydim, indicated that 0.05 mg/kg could not be achieved routinely. The Meeting noted that the lowest fortification level at which acceptable individual recoveries could be achieved was generally about 0.5 mg/kg. Acceptable recoveries were obtained from sugar beet, potatoes and liver at 0.1, 0.2 and 0.2 mg/kg respectively however. The Meeting agreed that it will be necessary for monitoring and enforcement laboratories to use the amended confirmatory method to differentiate residues of clethodim from those of sethoxydim if measurable residues are found with the "common moiety" method reviewed by the 1994 Meeting. The Meeting also agreed that the limit of determination appropriate for routine monitoring and enforcement should be that of the confirmatory method.

On the basis of the information on the revised confirmatory method, the Meeting concluded that the practical limit of determination appropriate for routine monitoring and enforcement should be 0.5 mg/kg, with lower levels only for sugar beet, fodder beat, potatoes, liver, kidneys and milk. For milk a practical limit of determination of 0.1 mg/kg was considered appropriate. Accordingly the Meeting recommended that some of the low maximum residue levels estimated by the 1994 Meeting be raised to 0.5* mg/kg and that these should be recommended as MRLs.

(iv) "*The method of analysis referred to does not make it possible to distinguish between residues from sethoxydim and a clethodim treatment. A verification method for the determination of clethodim and its metabolites has not been published and is thus not available for food inspection purposes.*"

The revised confirmatory method mentioned above is evaluated in the monograph. It is specific for the determination of clethodim and its metabolites in crops, animal tissues, milk and eggs, and can distinguish residues of clethodim from those of sethoxydim. The Meeting expressed concern that details of the revised confirmatory method were not currently in the public domain, but was informed that the manufacturer would make full details of the method available to monitoring and enforcement laboratories on request.

Supervised trials

(v) "*The residue trials for beans (dry), field peas (dry), potatoes and Sugar beet are summarized too strongly. Obviously, in some cases only summaries of trials have been available to the JMPR; we hold the view that an evaluation on such a basis should be refused.*"

The trials data for dry beans, dry peas, potatoes and Sugar beet reviewed by the 1994 Meeting are given in more detail in the monograph. The Meeting agreed that summaries of data should not be used when not accompanied by the full study reports, but full study reports were available to the Meeting on all the trials about which concern had been expressed except two potato trials, one each in the Ukraine and Belgium, for which only summaries were available.

(vi) "*It cannot be understood in all cases on which GAPs (use pattern) and which residue data the proposed MRLs are based. We hold the view that the residue data for potatoes are insufficient, irrespective thereof they do not justify an MRL of 0.2 mg/kg since the data from Canada cannot be used as a basis of comparison with the treatment in Belgium, Ecuador, Peru and Switzerland for climatic reasons.*" In addition, it was stated that "*The data is only available in summarized form. The number of trials that are within GAP is rather limited. The proposal is based on Canadian trials.*"

The Meeting agreed that outdoor trials data in Canada would not normally be related to GAP in Europe or South America. Additional information was provided to the current Meeting on GAP for potatoes in Australia, Belgium, Bulgaria, Canada, Czech Republic, Dominican Republic, Ecuador, Germany, Israel, Peru, Poland, Russia, Switzerland and Yugoslavia. This indicated slight changes from the GAP reported in 1994 for Belgium and Switzerland. The maximum application rates are 0.12-0.36 kg ai/ha with PHIs of 7-60 days. Canadian GAP was reported to the current Meeting. Although the Meeting agreed that the data were rather limited, a number of trials were available which indicated that residues resulting from a number of use patterns were low and often below the LOD. The Meeting confirmed that the previously estimated maximum residue level of 0.2 mg/kg was appropriate.

(vii) "*The MRL for sugar beet seems to be based on two Italian trials the results of which deviate from all other trials without any explanation being given.*"

GAP for sugar beet in Belgium, Morocco, Spain and Switzerland was reported to the 1994 Meeting. The maximum application rates were 0.20-0.36 kg ai/ha with PHIs from 50-90 days or not specified.

Nine French trials and one German trial were considered comparable to the German GAP reported to the current Meeting, with residue levels of <0.03 (9) and 0.05 mg/kg. Four Italian trials reported to the 1994 Meeting had originally been considered to comply with Spanish GAP, with reported residue levels of 0.06 (2) and 0.17 (2) mg/kg at 59 or 60 days. However, the Meeting was informed that treatment of sugar beet was at about the 2-8 leaf stage and that the minimum PHI was "about 90 days in practice."

In view of this new information the Meeting agreed to revise the previous recommendation and estimated a maximum residue level of 0.1* mg/kg, based on the trials according to German GAP. The Meeting concluded that the limit of determination in sugar beet was 0.1 mg/kg because acceptable recovery data for the revised confirmatory method had been submitted at this level.

(viii) "*The MRLs of 0.1 mg/kg cattle kidneys and liver are obviously based on a dosage of 10 mg/kg feed. But there are no reports on residues in potential feeding stuffs which would lead to such residues in everyday feed. Soya beans (MRL 10 mg/kg) usually only reach a percentage of 25-30 % in everyday feed: for cotton seed and rape seed an MRL of only 0.5 mg/kg has been envisaged. The MRL of 0.1 mg/kg cattle kidney, liver thus is unnecessarily high.*"

The Meeting observed that the highest residues (DME, *S*-methyl-DME and DME-OH) found in cows at the lowest dosing level were 0.059, <0.05 and <0.05 mg/kg, and 0.051, <0.05 and <0.05 mg/kg in liver and kidneys respectively. Since clethodim residues are calculated by the summation of the DME and DME-OH peaks in the common moiety method, the Meeting agreed that the maximum residue levels of 0.1 mg/kg estimated for cattle liver and kidneys by the 1994 Meeting had been appropriate. However, in view of the new information provided on the limit of determination of the revised confirmatory method, the Meeting agreed to increase the estimates to 0.2* mg/kg. The Meeting recognised that acceptable data on recoveries from kidneys by the revised confirmatory method were not available but considered that the limit of determination in kidneys was likely to be similar to that in liver, from which recoveries were satisfactory at 0.2 mg/kg.

(ix) The comment was made for beans (dry) "*The data is only available in summarized form. The number of trials is not specified. There are only trials from one country (Brazil) where clethodim is not registered. The trials are in accordance with GAP of other countries in the region. The proposal is based on a PHI of 65 days (pp. 358, 1994 Evaluations). Taking this PHI into account, 0.05 mg/kg is more appropriate.*"

In response, the supervised trials data for dry beans reviewed by the 1994 Meeting are given in more detail in the monograph. The Meeting reassessed the data which were available to the 1994 Meeting, concluded that they were insufficient to estimate a maximum residue level, and withdrew the previous recommendation for an MRL.

(x) "*Although a minor point, the table on pp 346 (1994 Evaluations) does not specify the levels in refined oil, and clarification is sought on the statement in the text (pp 347) that processing reduces levels to 10% in refined oil.*"

Additional information is provided in the monograph. The residue in the refined oil was <0.08 mg/kg and in the unprocessed cotton seed 0.8 mg/kg. A processing factor of <0.1 for cotton seed to refined oil is therefore appropriate.

(xi) Comments on dry field peas were that "*There is only registered use in Australia. The proposal is based on a PHI of 50-110 days (pp 358, 1994 Evaluations). On the basis of the Australian trial data (number of trials not specified, dosage 0.06-0.24 kg ai/ha, PHI 110 days) a limit of 0.05 mg/kg is sufficient. Also UK data (0.36 kg ai/ha [six times Australian registered dose], PHI 53 and 85 days) and Belgium data (up to three times registered dose in Australia, PHI 41 days) support this latter level. Only the French trials (0.18 kg ai/ha, PHI 67-82 days) points to a level of 0.1 mg/kg, but this is not in accordance with GAP.*"

A re-evaluation of the data on dry field peas has been carried out (see below), since new information on GAP and data from residue trials were reported to the present Meeting.

(xii) "*The proposal for sunflower seed is based on data from Argentina taking into account a PHI of 106 days. However, such a long PHI is not in accordance with the PHI reported for Argentina and other countries in table 4 of the Evaluations. The Netherlands therefore reserves its position for these proposals. For oil, crude and oil edible 0.05 mg/kg are reasonable when 0.2 mg/kg is a appropriate level for sunflower seed.*"

GAP for sunflower in Argentina, Bolivia, Ecuador, Israel, Morocco, Paraguay and Spain was reported in the 1994 monograph, where the maximum application rate in Spain was stated to be 0.2 kg ai/ha with an unspecified PHI. The manufacturer informed the Meeting that the use was post-emergence and that the Spanish PHI was "60 days in practice". Residues from applications 60-74 days before harvest were 0.03-0.13 mg/kg in three Italian trials.

The maximum application rate in South America was 0.12-0.34 kg ai/ha with PHIs of 5-56 days or not specified. The PHIs in all of the Argentinean trials were longer at 102-106 days. The manufacturer stated that "although the PHI from the Argentina trials exceeded the GAP of 75 days, we believe that we would not detect any greater than what we have observed at a 75 day PHI." The Meeting concluded that there were insufficient data from trials according to GAP to estimate a maximum residue level and withdrew the previous estimate.

GAP for such broad categories as "fruit" or "vegetables" has been ignored in evaluating the results of the other supervised trials reviewed below.

Peaches. Conflicting information on GAP in Spain had been reported, with application rates of 0.096-0.192 or 0.036-0.24 kg ai/ha. The timing of the application was also unclear. The Meeting was informed that the application was directed around the base of the tree.

GAP for “fruit trees” was reported for Chile, Ecuador and Saudi Arabia and for “orchard crops” for New Zealand. The maximum application rates were 0.06-0.24 kg ai/hl and 0.18-0.72 kg ai/ha with PHIs ranging from 15-60 days or not specified.

The Meeting noted that although all the residues in the trials on peaches were below the limit of determination of 0.03 mg/kg, only one trial included a PHI longer than 21 days. Since longer intervals between treatment and harvest might lead to determinable residues owing to uptake, and in view of the conflicting information on GAP, the Meeting concluded that there were insufficient data to estimate a maximum residue level.

Onions and garlic. Information on GAP for garlic in Saudi Arabia, Spain and the USA was reported. The maximum application rates were 0.192- 0.28 kg ai/ha with PHIs of 30, 45 or 60 days. The maximum number of applications was not stated for any country.

Only two trials on garlic were considered to comply with US GAP. Although the DME-OH residue levels were described by the manufacturer as “not considered to be clethodim-related, due to matrix interference peak” this could not be confirmed by the Meeting and the results were therefore included; the residues (sum of DME and DME-OH) were 0.36 and 0.1 mg/kg.

GAP for onions was reported for Australia, Belize, Dominican Republic, Guatemala, Honduras, Israel, New Zealand, Russia, Saudi Arabia, Turkey, the USA and Uzbekistan, and pending GAP in Brazil. The maximum application rates were 0.12-0.28 kg ai/ha (0.108 kg/ha in the pending Brazilian GAP) with PHIs of 7-65 days.

The residues in trials considered to comply with US GAP were <0.05, ≤0.1(4), 0.13 and 0.15 mg/kg. The residues in trials considered to be in accord with GAP in New Zealand or pending GAP in Brazil were all below the limit of determination, as were other residues from exaggerated application rates in some of these trials. The residues from the trials according to GAP were <0.01, <0.02, <0.03 (4) and <0.05 (6) mg/kg. Only one trial accorded with Australian GAP, with a residue of 0.05 mg/kg.

The Meeting agreed that the results of the garlic and onion trials could be combined, but noted that the US residues formed a different population from those in the Brazilian and New Zealand trials. The combined US residues in rank order were <0.05, <0.1 (4), 0.1, 0.13, 0.15 and 0.36 mg/kg. The Meeting estimated maximum residue levels of 0.5 mg/kg and STMRs of 0.1 mg/kg, based on US GAP, for both onion and garlic.

Cabbage. GAP was reported for Australia and Poland. The maximum application rates are 0.12 and 0.24 kg ai/ha with PHIs of 7 and 60 days respectively.

Only one residue trial was considered to comply with Australian GAP and one with Polish. The residues were 0.07 and 0.15 mg/kg respectively. There were insufficient data to estimate a maximum residue level.

Cauliflower. GAP was reported only for New Zealand, with a maximum application rate of 0.24 kg ai/ha and a PHI of 35 days. Only one trial was considered to comply with this, with a residue of 0.28 mg/kg. There were insufficient data to estimate a maximum residue level.

Cucumber. GAP for cucumbers was reported only for Poland, with a maximum application rate of 0.24 kg ai/ha and a PHI of 60 days, and for cucurbits in Paraguay with the same maximum application rate and an unstated PHI.

A single trial was considered to be comparable to Polish GAP, because although the PHI was shorter the residue level was <0.05 mg/kg. In six US trials all residues were <0.14 mg/kg at the short PHI of 13-14 days, but none of the trials was according to GAP. There were insufficient data to estimate a maximum residue level.

Summer squash. GAP for cucurbits was reported for Paraguay, but none of the three trials in the USA were considered to conform to it. There were insufficient data to estimate a maximum residue level.

Tomatoes. GAP for tomatoes was reported for Belize, Bulgaria, Dominican Republic, Israel, Italy, Nicaragua, Spain and the USA, and pending GAP for Brazil. The maximum application rates are 0.12-0.28 kg ai/ha (0.108 kg/ha for the pending GAP) with PHIs of 7-30 days, “unrestricted” or unstated.

The residues in two trials considered to comply with the pending Brazilian GAP were <0.05 mg/kg.

The residues in trials considered to comply with Spanish GAP were <0.03 (5), 0.03, 0.08 and 0.13 mg/kg, and with US GAP <0.1 (3), 0.11, 0.12, 0.15 (2), 0.16, 0.17, 0.21, 0.27, 0.34, 0.35 (2), 0.43, 0.46, 0.50, 0.52, 0.54, 0.65, 0.71, 0.76 (2) and 0.82 mg/kg. The Meeting estimated a maximum residue level of 1 mg/kg and an STMR of 0.35 mg/kg, based on the trials according to US GAP.

Lettuce. GAP was reported for Australia and Israel. The maximum application rates are 0.12 kg ai/ha with PHIs of 28 days and unstated respectively.

The residues in trials considered to comply with Australian GAP were 0.04 and 0.21 mg/kg. There were insufficient data to estimate a maximum residue level.

Beans (fresh). GAP was reported for beans for Belgium, Bolivia, Bulgaria, Paraguay, Peru, Spain and Turkey, for mung and fava beans for Australia and for legumes for Chile. The maximum application rates are 0.06-0.48 kg ai/ha with PHIs of 0-65 days or unstated.

The residues in fresh beans from trials considered to comply with Belgian GAP were <0.025 and <0.05 (4) mg/kg. Although some of the Belgian results were at shorter PHIs than GAP, the residues were all below the limit of determination. In addition one trial on "green beans" with a residue of 0.21 mg/kg and one on "French beans" with a residue of <0.03 mg/kg were considered to comply with Spanish GAP. Although data on a number of US trials were also submitted, no GAP was reported for the North American continent. A trial on broad (fava) beans in Spain did not conform to reported GAP.

The Meeting estimated a maximum residue level of 0.5* mg/kg and an STMR of 0.05 mg/kg for beans, except broad bean and soya bean, based on the trials according to Belgian GAP.

Lentils. GAP for lentils was reported to the current Meeting for Canada, New Zealand and Turkey, and to the 1994 Meeting for New Zealand and Spain. GAP for beans and legumes would presumably cover lentils.

Although two Spanish trials were reported, they could not be evaluated against the Spanish GAP recorded in 1994 because the GAP did not include the PHI. Neither of the trials was considered to comply with relevant GAP. There were insufficient data to estimate a maximum residue level.

Lupins. GAP for lupins was reported for Australia, with a maximum application rate of 0.12 kg ai/ha; no PHI was specified.

Only one trial was considered to comply with Australian GAP with a residue of <0.1 mg/kg. The Meeting could not estimate a maximum residue level.

Carrots. GAP for carrots was reported for Israel and Russia, and pending GAP for Brazil. The maximum application rates were 0.108 (Brazil) - 0.24 kg ai/ha with PHIs of 40-75 days.

The residues in two trials which complied with the pending Brazilian GAP were <0.05 mg/kg. None of the other trials were considered to accord with any other reported GAP. There were insufficient data to estimate a maximum residue level.

Celery. GAP for celery was reported for Australia. The maximum application rate is 0.12 kg ai/ha with a PHI of 9 weeks.

The only trial which complied with Australian GAP showed a residue of 0.04 mg/kg. There were insufficient data to estimate a maximum residue level.

Linseed (flax). GAP was reported for Canada, Russia and the Ukraine. The maximum application rates were 0.09-0.24 kg ai/ha. PHIs were 60-80 days or not specified.

One trial was considered to be in accord with Russian GAP, with a residue of <0.01 mg/kg. Several Canadian trials was reported for which exaggerated rates had been used with all residues below the limit of determination at PHIs of 84-119 days, but since no samples were taken at the Canadian PHI of 60 days the Meeting concluded that there were insufficient data to estimate a maximum residue level.

Peanuts. GAP was reported for Argentina, Australia, Bolivia, Israel, Taiwan and the USA. The maximum application rates were 0.09-0.336 kg ai/ha with PHIs of 40-70 days or not specified. GAP for "vegetables" was also reported for Chile, Ecuador, New Zealand, Paraguay and Peru.

The residues in trials considered to comply with US GAP were <0.05, 0.34, 0.56, 0.79, 1.3 (2), 1.8, 2.7 and 3.5 mg/kg in the kernels and 0.17, 0.20, 0.24, 0.24, 0.3, 0.60, 0.75 and 0.81 mg/kg in the hulls. The Meeting estimated a maximum residue of 5 mg/kg and an STMR of 1.3 mg/kg for peanut.

Alfalfa. GAP was reported for Argentina, Canada, Chile, Ecuador, Israel, Peru and the USA. The maximum application rates were 0.09-0.48 kg ai/ha with PHIs of 15-30 days or not specified.

Trials according to national GAP were carried out in Canada and the USA with residues of <0.02 (7) and 0.02 mg/kg in Canada, and 0.27, 0.53, 0.61, 0.62, 0.67, 0.85, 1.2, 1.4 (3), 1.5, 1.6, 1.9, 2.0, 2.6, 2.7

(2), 3.0, 4.4, 4.5, 5.4 and 8.9 mg/kg in the USA. The Meeting estimated a maximum residue level of 10 mg/kg and an STMR of 1.6 mg/kg, based on US GAP.

White Clover. GAP for clover was reported for Israel and New Zealand. The maximum application rates are both 0.12 kg ai/ha with a PHI of 63 days in New Zealand and not specified in Israel. GAP for "vegetables" was reported for Chile, Ecuador, New Zealand, Paraguay and Peru

The two residues in trials considered to comply with New Zealand GAP were 0.07 and 0.26 mg/kg. The samples analysed were described as "young plants" and "silage". There were insufficient data to estimate a maximum residue level.

Field peas (dry). GAP for field peas was reported for Australia and Canada. The maximum application rate in Canada is 0.09 kg ai/ha with a PHI of 75 days. The maximum rate reported by the Australian government, supported by a product label, was 0.06 kg ai/ha and differed from that reported by the manufacturer.

All the residues in six trials in Australia were <0.1 mg/kg after 110 days even at exaggerated doses. The residues in several further trials which were considered to comply with Canadian GAP were <0.02 (4), 0.06, 0.08, <0.10, 0.18 (2), 0.31, 0.65 and 1.8 mg/kg. The Meeting estimated a maximum residue level of 2 mg/kg and an STMR of 0.08 mg/kg, based on Canadian GAP.

Peas. GAP for peas was reported for Belgium, the Czech Republic, Israel, New Zealand and Spain, and for "proteaginous peas" for France. The maximum application rates are 0.06-0.36 kg ai/ha with PHIs of 30 or 60 days, or not specified. No trials were considered to comply with relevant GAP and no maximum residue level could be estimated.

Fodder beet. GAP was reported for Belgium, the Czech Republic, Germany, Italy, Russia and Switzerland. The maximum application rates are 0.14-0.36 kg ai/ha with PHIs of 60-90 days or not specified. The Meeting was informed that application is at about the 2-8 leaf growth stage.

The residues in three trials in France which were considered to comply with Belgian GAP were all below the LOD of 0.03 mg/kg in both roots and tops. In additional trials at the same sites with exaggerated application rates the residues were also all below 0.03 mg/kg. The Meeting estimated a maximum residue level of 0.1* mg/kg and an STMR of 0.03 mg/kg. The Meeting established the limit of determination for fodder beet at 0.1 mg/kg because acceptable recovery data for the revised confirmatory method had been submitted for sugar beet at this level.

Other commodities. Residue trials data were also submitted for leeks, spinach, artichokes, sweet peppers and "non-bell peppers" but no specific GAP was reported to the present or the 1994 Meeting. The Meeting agreed that it would be appropriate to evaluate these trials against a general GAP for vegetables in the case of this compound, since it is a post-emergence herbicide, but there were no trials on any of these commodities according to GAP from which to estimate maximum residue levels.

RECOMMENDATIONS

Definition of the residue for compliance with MRL and for the estimation of the dietary intake: sum of clethodim and its metabolites containing the 5-(2-ethylthiopropyl)cyclohexene-3-one and the 5-(2-ethylthiopropyl)-5-hydroxycyclohexene-3-one moieties and their sulfoxides and sulfones, expressed as clethodim.

The Meeting estimated the following maximum residue levels which are recommended for use as MRLs:

Commodity		Recommended MRL, mg/kg		STMR mg/kg	PHI on which proposal based
CCN	Name	New	Previous		
AL 1020	Alfalfa fodder	10	-	1.6	15
VD 0071	Beans (dry)	W	0.1	0.05	65
VP 0061	Beans, except broad bean and soya bean	0.5*	-	0.05	60
MO 1280	Cattle, kidneys	0.2*	0.1	-	-
MO 1281	Cattle, liver	0.2*	0.1	-	-
MM 0812	Cattle meat	0.5*	0.05*	-	-
ML 0812	Cattle milk	0.1*	0.05*	-	-
PE 0840	Chicken eggs	0.5*	0.05*	-	-
PE 0840	Chicken meat	0.5*	0.05*	-	-
OC 0691	Cotton seed oil, crude	0.5*	0.1	-	-
OR 0691	Cotton seed oil, edible	0.5*	0 05	-	-
VD 0561	Field pea (dry)	2	0.1	0.08	75
AM 1051	Fodder beet	0.1*	-	0.03	90
VA 0381	Garlic	0.5		0.1	45
VA 0385	Onion, bulb	0.5	-	0.1	45
SO 0697	Peanut	5	-	1.3	40
OC 0495	Rape seed oil, crude	0.5*	0.05	-	-
OR 0495	Rape seed oil, edible	0.5*	0.05	-	-
OR 0541	Soya bean oil, edible	0.5*	0.1	-	-
VR 0596	Sugar beet	0.1	0.2	-	-
SO 0702	Sunflower seed	W	0.2	-	-
OC 0702	Sunflower seed oil, crude	W	0.05	-	-
OR 5702	Sunflower seed oil, edible	W	0.05	-	-
VO 0448	Tomato	1	-	0.35	20

FURTHER WORK OR INFORMATION

Desirable

Data on residues occurring in commerce and/or at consumption (from 1994 Meeting).

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FENBUCONAZOLE (197)

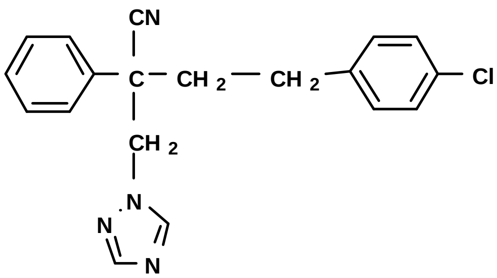
ISO common name: fenbuconazole

Chemical name
IUPAC: 4-(4-chlorophenyl)-2-phenyl-2-(1*H*-1,2,4-triazol-1-yl)methybutyronitrile
CA: α-[2-(4-chlorophenyl)ethyl]-α-phenyl-1*H*-1,2,4-triazole-1-propanenitrile

CAS registry no: 114369-43-6 (unstated steriochemistry)
119611-00-6 (with chiral properties specified)

Synonyms: Fenethanil, RH-7592, RH-57592.

Structural formula:



Molecular formula: $C_{19}H_{17}ClN_4$

Molecular weight: 336.8

Physical and chemical properties

Pure active ingredient

Physical state: solid

Appearance: white crystalline

Melting point: 126.5-127.0°C (Graves, 1991; Batra 1997)

Solubility: water, mg/l: 3.77/25°C; 7.01/40°C (bead elution/HPLC method, unbuffered water) 2.04-2.26 (22°C shake flask method, unbuffered water) (Jacobson, 1988)

3.6/pH 7; 3.6/pH9 (20°C, shake flask method) (Kellner, 1992) soluble in ketones, esters, alcohols and aromatic hydrocarbons; insoluble in aliphatic hydrocarbons

Vapour pressure: <10^{-5} Pa at 20°C (OECD method 104) (Kellner, 1992)

4.9 x 10^{-6} Pa. (Purity unstated but test carried out on “recrystallised fenbuconazole”) (Graves, 1991)

Hydrolysis:

Not hydrolysed 30 days under sterile conditions in the dark at pH 5, 7, and 9. Estimated minimum half-life 2210 days at pH 5, 3740 days at pH 7, 1340 days at pH 9 (O’Dowd, 1990d)

Photolysis:

Stable in pH 7 aqueous buffer at 25ºC for 30 days with 12-hour light/dark cycles (Wang,1991b)

Degraded at a concentration of 1.5mg/l in natural pond water. Assuming pseudo-first-order kinetics, calculated half-life was 86.7 days. (Baur 1994)

Octanol/water partition coefficient:

log P_{ow} = 3.22 at 25ºC. Determined from 14C-fenbuconazole (radiochemical purity 99.5%) solutions of 100, 500 and 1,000 ppm which were partitioned in triplicate between octanol and water, and the concentration in each phase was determined by radioassay. (O’Dowd, 1987).

Henry’s Law Constant: 7.63 x 10^{-9} [atm.m^3/g.mol] at 25ºC (Chong, 1992)

Technical material

Purity: 98.3% (94% minimum)
Physical state: Off-white to white powder
Melting point: 124°-126°C

Solubility in water: 3.8 mg/l at 25°C, 7.0 mg/l at 40°C (unspecified pH) (Graves, 1991; Costlow, 1997b)

Solubility, g/l 25°C: (by HPLC)
231 in acetonitrile
77 in aromatic 200 (C9 & C10 hydrocarbons)
445 in cyclohexanone
159 in ethyl acetate
39 in ethanol
1 in heptane
13 in n-octanol
(Graves, 1991)

Fat solubility at 37°C: 1052.2 mg/100g (MacDonald *et al.,* 1990a; Batra, 1997)

Surface tension:
69.5 mN/m (as a 90% saturated water solution at 22°C. (fenbuconazole purity stated as “94 to 99.5%”). Test carried out according to EU method A5. (MacDonald *et al.*, 1990c; Costlow, 1997b)

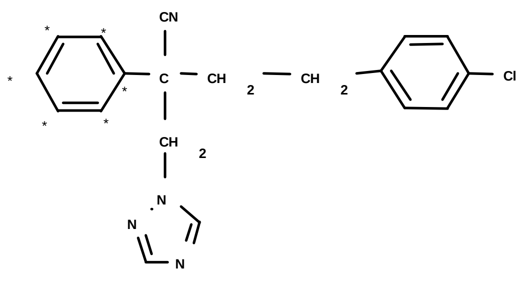
Relative density: D^{20} = 1.27 (EU method A3) (MacDonald *et al.*, 1990b)

Formulations

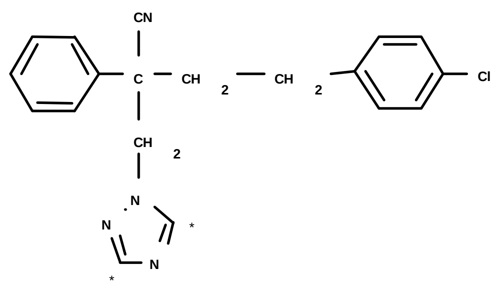
Fenbuconazole is a triazole fungicide and is formulated mainly as an EC or an EW (oil in water emulsion).

METABOLISM AND ENVIRONMENTAL FATE

The studies were carried out with [^{14}C]fenbuconazole uniformly incorporated in the unsubstituted phenyl ring (phenyl-labelled) or the triazole ring ("triazole-labelled"), as shown by the asterisks in the structures shown below.



Phenyl-labelled



Triazole-labelled

Animal metabolism

Rats. In a 1987 study (Hanauer, 1991), four groups of 4 Crl:CD BR rats were orally dosed with phenyl-labelled [^{14}C]fenbuconazole (radiochemical purity >99%). A 0.5% suspension of methyl cellulose was used as the vehicle. Dosing was by gavage at 100 mg/kg bw. All animals were killed 7 days after dosing.

In group 1 (four males) whole blood samples were taken for liquid scintillation counting 0.25, 1, 3, 6, 24, 48, 72, 96 and 168 hours after dosing. In group 2 (four males) urine and faeces were collected at 0, 6, 24, 48, 72, 96 and 168 hours, and ^{14}C was determined in the expired air and in selected tissues and organs after 7 days. In groups 3 and 4 (four animals of each sex) urine and faeces collected as in group 2 were frozen over liquid nitrogen for subsequent analysis.

The average total recovery of ^{14}C from the animals in group 2 was 67% of the administered dose, about 62% in the faeces and 4% in the urine. Radioactivity detected in expired CO_2 accounted for only 0.05% of the administered dose. Most of the excretion had occurred by 48 hours. Peak blood and plasma levels of the radiolabel were detected at 6 hours. A biphasic elimination pattern was seen with a rapid first phase (half-life 7 hours) followed by a slower second phase (half-life about 50 hours for plasma and 187 hours for whole blood). After 7 days <0.5% of the administered dose was detected in the tissues, where the radiolabel levels were all lower than 2.5 mg/kg fenbuconazole equivalents (liver levels were highest at 2.48 mg/kg or 0.13% of the dose). Of the average total of 0.53% of the dosed radiolabel found in the bodies after 7 days, 0.24% was found in the carcase.

The combined faeces and urine samples of each sex were analysed, using a variety of extractions, solvent partitions and chromatographic separations, and the purified components of the residue were identified by mass spectrometry or chromatographic comparison with synthesized samples of the suspected metabolites. The metabolite profile in the excreta is shown in Table 1.

Table 1. Metabolite profile of fenbuconazole in excreta of rats.

Compound	Percentage of total activity in excreta	
	Male	Female
Fenbuconazole	7.9	13.0
Lactones (both diasterioisomers)	12.9	8.1
Iminolactone	2.5	0.9
Benzylic alcohols (including non-sulfate conjugates)	7.1	3.3
Benzylic sulfates (both diasterioisomers)	4.1	11.2
Phenols (3-OH and 4-OH isomers, including conjugates)	9.3	14.3
Phenol lactones (both isomers, including conjugates)	6.5	5.8
Ketoacid	2.4	1.0
Phenol ketoacid	5.1	0.0
RH-7968[1]	<1	<1
Triazole	1.9[2]	1.5[2]

[1]4-(4-chlorophenyl)-2-hydroxymethyl-2-phenylbutyronitrile
[2]Percentage of dose

In a 1992 study, groups of male and female Crl:CD BR rats were dosed by gavage with phenyl-labelled [^{14}C]fenbuconazole (98.7% radiochemical purity) using a 0.5% suspension of methyl cellulose as the vehicle. Three groups of 5 males and 5 females were given single doses of 1 or 100 mg/kg bw [^{14}C]fenbuconazole or a labelled dose of 1 mg/kg bw after a 14-day treatment with 10 ppm of the unlabelled compound in the diet. Urine and faeces samples were taken at 0, 6, 24, 48, 72 and 96 hours after dosing and the animals were then killed. Two groups of 3 males and 3 females were dosed with 1 mg/kg bw, and excreta samples were taken at the time of dosing and when the rats were killed after 3 or 12 hours. One group of 5 males and 5 females were biliary canulated and dosed at 1 mg/kg bw. Excreta samples were taken up to 72 hours, when the animals were killed, and blood samples at 0, 6, 24, 48 and 72 hours. Tissues, organs and the carcases except those from the last group were analysed for radioactivity, but only the whole carcases of the canulated group were analysed.

The average total recoveries of ^{14}C in the groups killed after 4 days were 96-104.7% of the administered doses, mainly in the faeces and within the first 48 hours (Table 2).

Table 2. Recovery of [^{14}C] from tissues and excreta of rats 4 days after gavage administration of [^{14}C]fenbuconazole.

Dose, mg/kg bw	Sex	^{14}C, mean % of administered dose				
		Faeces	Urine	Tissues	Carcase	Total[1]
1	Male	85.3	9.8	0.5	0.3	96.0
1	Female	92.2	11.4	0.8	0.1	104.7
100	Male	90.2	7.0	0.2	0.2	97.6
100	Female	78.7	11.1	0.2	0.2	90.4
1[2]	Male	94.4	8.7	0.6	0.3	104.3
1[2]	Female	89.4	11.1	0.8	2.9	104.5

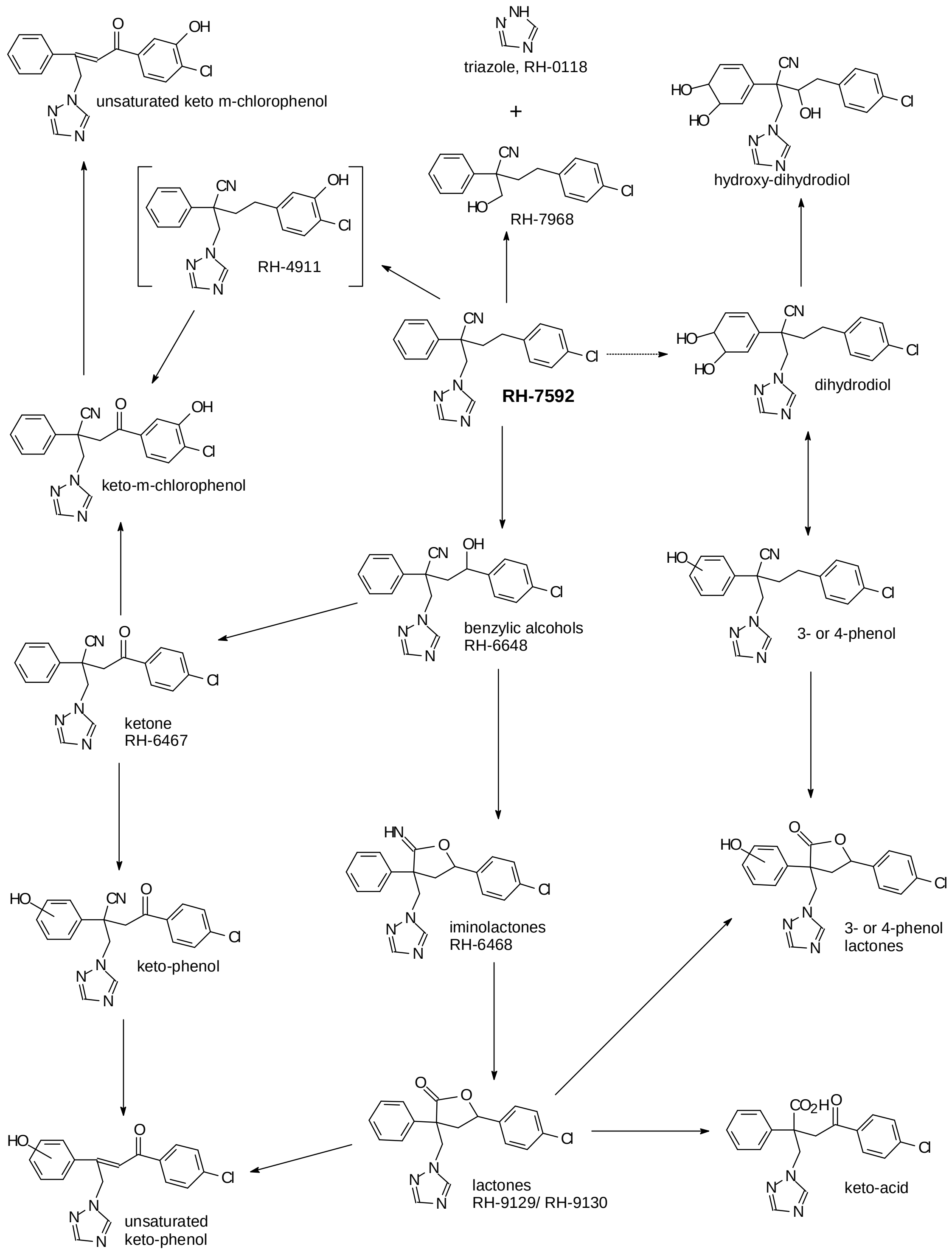
[1]Including cage wash
[2]After treatment for 14 days with 10 ppm unlabelled fenbuconazole in the diet

The results did not show any significant sex- or dose-related differences between treatments. In the canulated animals during the 3 day collection period, averages of 79-87% of the total dosed radiolabel were found in the bile (mainly within 24 hours), 6-7% in the faeces, 3-7% in the urine and 1-2% in the carcase, with total recoveries of 95-98%; 88-91% of the doses were absorbed.

The average total ^{14}C in the tissues of animals killed 3 and 12 hours after dosing was 8-12% and 3-4% of the administered dose respectively, of which >90% was found in the liver. The total residues of radiolabel in the carcases 4 days after dosing were low. At 1 mg/kg bw (both with and without prior dietary administration) the average residues varied from 0.001 mg/kg fenbuconazole equivalents in the muscles to maxima of 0.13 mg/kg in the liver (females) and 0.19 mg/kg in the thyroid (males). At 100 mg/kg bw ^{14}C levels varied from 0.09 mg/kg in the muscles to 0.7-1.4 mg/kg in the thyroids of males and 3.1-4.2 mg/kg in the livers of females (Donato and Hazelton, 1993).

On the basis of these studies the manufacturer proposed the metabolic pathways for fenbuconazole in rats shown in Figure 1.

Figure 1. Metabolism of fenbuconazole in rats.



The hydroxylated compounds may also be conjugated with sulfate, but were mainly present as glucuronide (Batra, 1997; Ross, 1997b).

Goats. Nine lactating goats each received seven daily doses of [^{14}C] fenbuconazole, five with the phenyl label at rates equivalent to 1, 10 and 100 ppm in the feed and four with the triazole label at a rate equivalent to 100 ppm. The goats were slaughtered 24 after the last dose. All samples were analysed by LSC after combustion. The samples were also extracted with methanol and then partitioned successively with ethyl acetate, n-butanol, hexane and water. The resulting organic and aqueous phases were analysed by TLC and HPLC.

The overall recoveries of ^{14}C were 72.7-87.3% from the phenyl label and 74.3-81.2% from the triazole labels. Most of the radioactivity was excreted (79.7-86.0% of the phenyl label and 72.3-80.0% of the triazole), with less than 0.1% of the phenyl label and 0.2-0.4% of the triazole label in the milk and 0.8-1.2% of the phenyl and 1.1-1.6% of the triazole label in the tissues.

The total ^{14}C in the milk, expressed as fenbuconazole equivalent, was less than 0.01 mg/kg at the 1 and 10 ppm feeding levels. At the 100 ppm level it reached plateaux of 0.07 mg/kg after 4 days with the phenyl label and 0.4 mg/kg after 5 days with the triazole label.

The triazole-labelled compound yielded two major metabolites in the milk, triazole (0.24 mg/kg) and triazolylalanine (0.15 mg/kg). The parent compound and six other metabolites were also identified, but were present at levels below 0.02 mg/kg (Table 3).

Table 3. Fenbuconazole and its metabolites in goat milk.

Compound	^{14}C, mg/kg as fenbuconazole	
	Phenyl label	Triazole label
Fenbuconazole	0.02	<0.01
Lactones and phenols	0.02	<0.01
Triazole	-	0.24
Triazolylalanine	-	0.15
RH-7968	<0.01	<0.01
Benzylic glucuronides	0.02	<0.01
Total	0.06	0.40

The total ^{14}C in the tissues (expressed as fenbuconazole) from the phenyl label was less than 0.05 mg/kg from the 1 and 10 ppm feeding levels, except in liver which contained 0.10 and 0.62 mg/kg. The 100 ppm feeding level produced 0.07 mg/kg in the muscle, 0.16 mg/kg in the fat, 0.89 mg/kg in the kidneys and 7.9 mg/kg in the liver. The triazole label at 100 ppm gave average total ^{14}C residues in the tissues of 0.23 mg/kg in the muscle, 0.02 mg/kg in the fat, 0.94 mg/kg in the kidneys and 12.1 mg/kg in the liver.

Table 4 shows the averages of the residues from the two labels for the 100 ppm feeding level.

Table 4. Fenbuconazole and its metabolites in goat tissues.

Compound	^{14}C, mg/kg as fenbuconazole			
	Liver	Kidneys	Muscle	Fat
Fenbuconazole	0.95	0.10	0.02	0.02
Lactones[1]	0.84	0.06	<0.01	<0.01
Phenol	0.47	0.13	<0.01	<0.01
Iminolactones[1]	0.56	<0.01	<0.01	<0.01
Ketoacid	0.16	<0.01	<0.01	<0.01
Benzylic alcohols[1]	0.13	0.05	<0.01	<0.01
Benzylic sulfates[1]	0.40	0.03	<0.01	<0.01
Triazole	1.79	0.11	0.09	<0.01
Triazolylalanine	4.95	0.24	0.07	<0.01
RH-7968	0.95	0.13	0.01	0.04
Benzylic glucuronides[1]	1.23	0.13	<0.01	0.01
Total	12.43	0.97	0.22	0.11

[1]Diasterioisomers

The six major components identified in the liver were the parent (0.95 mg/kg), the lactones (0.84 mg/kg), 4-(4-chlorophenyl)-2-hydroxymethyl-2-phenylbutyronitrile (RH-7968, 0.95 mg/kg), the benzylic glucuronides (1.23 mg/kg), triazole (1.79 mg/kg) and triazolylalanine (4.95 mg/kg). Five other metabolites were identified at levels below 0.6 mg/kg.

In the kidneys the six main components were the parent (0.10 mg/kg), RH-7968 (0.13 mg/kg), the phenol (0.13 mg/kg), glucuronides (0.13 mg/kg), triazole (0.11 mg/kg) and triazolylalanine (0.24 mg/kg). Five other metabolites were identified at levels of [0.06 mg/kg.

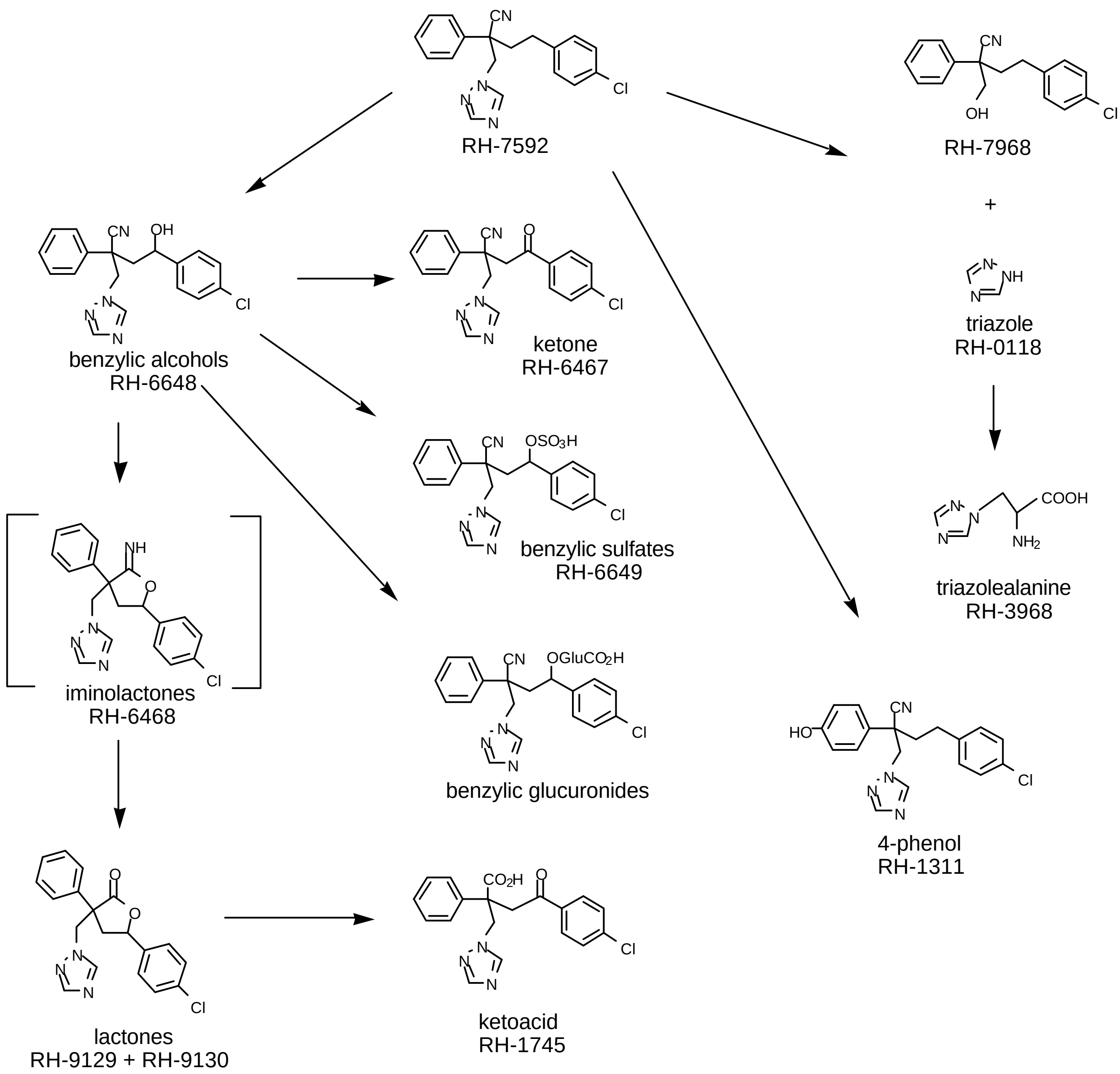
In muscle the main components were the parent (0.02 mg/kg), RH-7968 (0.01 mg/kg), triazole (0.09 mg/kg) and triazolylalanine (0.07 mg/kg). Six other metabolites were identified at levels below 0.01 mg/kg.

In fat all the compounds except the parent (0.02 mg/kg), RH-7968 (0.04 mg/kg) and the glucuronides (0.01 mg/kg) were at levels below 0.01 mg/kg (Jameson, 1989a; Predmore, 1990; Sharma 1992a).

The conversion of the two isomers of the iminolactones RH-6468 to the corresponding isomers of the lactones RH-9129 and RH-9130 during the analysis of cow liver samples was demonstrated. Samples fortified with RH-6468 were processed by the analytical method of Filchner (1994) and the conversion was shown to occur during clean-up on silica gel and C-18 SPE columns (Staurowsky and Wu, 1994).

The manufacturer has proposed the metabolic pathways in lactating goats shown in Figure 2.

Figure 2. Metabolic pathways of fenbuconazole in lactating goats (Batra, 1997).



Hens. Thirty five laying hens each received seven daily doses of [^{14}C]fenbuconazole at rates equivalent to 100 ppm in the feed, ten with the phenyl label and 25 with the triazole. The hens were killed 24 hours after the last dose. Samples were analysed by LSC after combustion, and were extracted with methanol and then partitioned into ethyl acetate, n-butanol and water. The resulting organic and aqueous phases were analysed by TLC and HPLC.

The overall recoveries of radioactivity were 94.9-99.4% from the phenyl label and 87.1-95.8% from the triazole label. Excretion accounted for 93.4-97.8% of the phenyl label and 85.1-93.8% of the triazole label, with only 0.4-0.5% of the phenyl label and 0.6-0.7% of the triazole label in the eggs and 0.6% of the phenyl label and 0.6-0.8% of the triazole label in the tissues.

The total ^{14}C in the eggs reached levels after 6 days of 2.0 mg/kg expressed as fenbuconazole with the phenyl label and 2.7 mg/kg with the triazole label. The major components identified in the eggs were the parent (0.9 mg/kg), its lactone (0.6 mg/kg) and, from the triazole label, RH-7968 and triazole (0.54 mg/kg). Four other metabolites were identified at levels below 0.2 mg/kg.

Table 5. Fenbuconazole and its metabolites in eggs.

Compound	^{14}C, mg/kg as fenbuconazole	
	Phenyl label	Triazole label
Fenbuconazole	0.89	0.88
Lactones	0.56	0.61
Iminolactones	0.16	0.16
Phenol	0.10	0.10
Benzylic sulfates	0.02	0.09
Triazole	-	0.54
RH-7968	0.08	0.77
Benzylic glucuronides	0.05	0.19
Total	1.86	2.61

The total ^{14}C residues in the tissues expressed as fenbuconazole equivalent from the phenyl label were 0.20 mg/kg in muscle, 1.04 mg/kg in the fat, 3.00 mg/kg in the kidneys and 11.6 mg/kg in the liver. The triazole label yielded 0.76 mg/kg in muscle, 0.96 mg/kg in the fat, 2.83 mg/kg in the kidneys and 11.1 mg/kg in the liver.

The highest residues in the liver were the glucuronides (3.69 mg/kg) and triazole 1.25 mg/kg). The parent and seven other metabolites were identified at levels below 1 mg/kg.

The two major components identified in muscle were the triazole (0.28 mg/kg) and triazolylalanine (0.06 mg/kg). The other identified compounds were at levels below 0.05 mg/kg.

In fat fenbuconazole (0.43 mg/kg) and the lactones (0.17 mg/kg) were the main residues. The other metabolites were at levels of <0.01-0.09 mg/kg (Jameson, 1989b; Sharma, 1992b, 1994a).

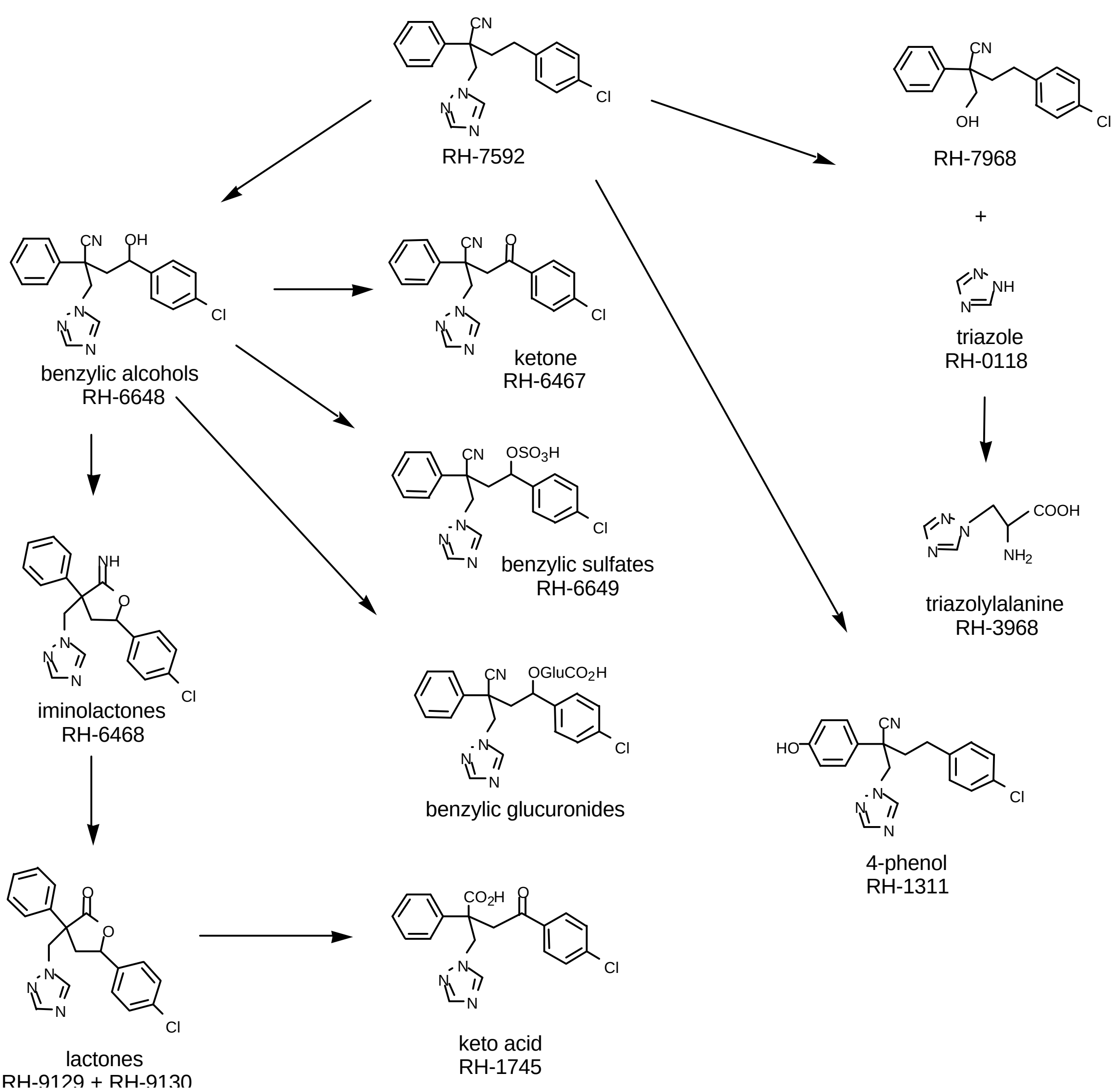
Table 6 shows the averages of the residues from the two labels.

Table 6. Fenbuconazole and its metabolites in hen tissues.

Compound	^{14}C, mg/kg as fenbuconazole		
	Liver	Muscle	Fat
Fenbuconazole	0.27	0.03	0.43
Lactones	0.90	0.02	0.17
Phenol	0.97	0.02	<0.01
Iminolactones	0.97	0.02	0.06
Ketoacid	0.32	<0.01	<0.01
Benzylic sulfates	0.64	0.03	<0.01
Triazole	1.25	0.28	<0.01
Triazolylalanine	<0.01	0.06	<0.01
RH-7968	0.87	<0.01	0.09
Benzylic glucuronides	3.69	0.02	0.07
Total	9.26	0.52	0.98

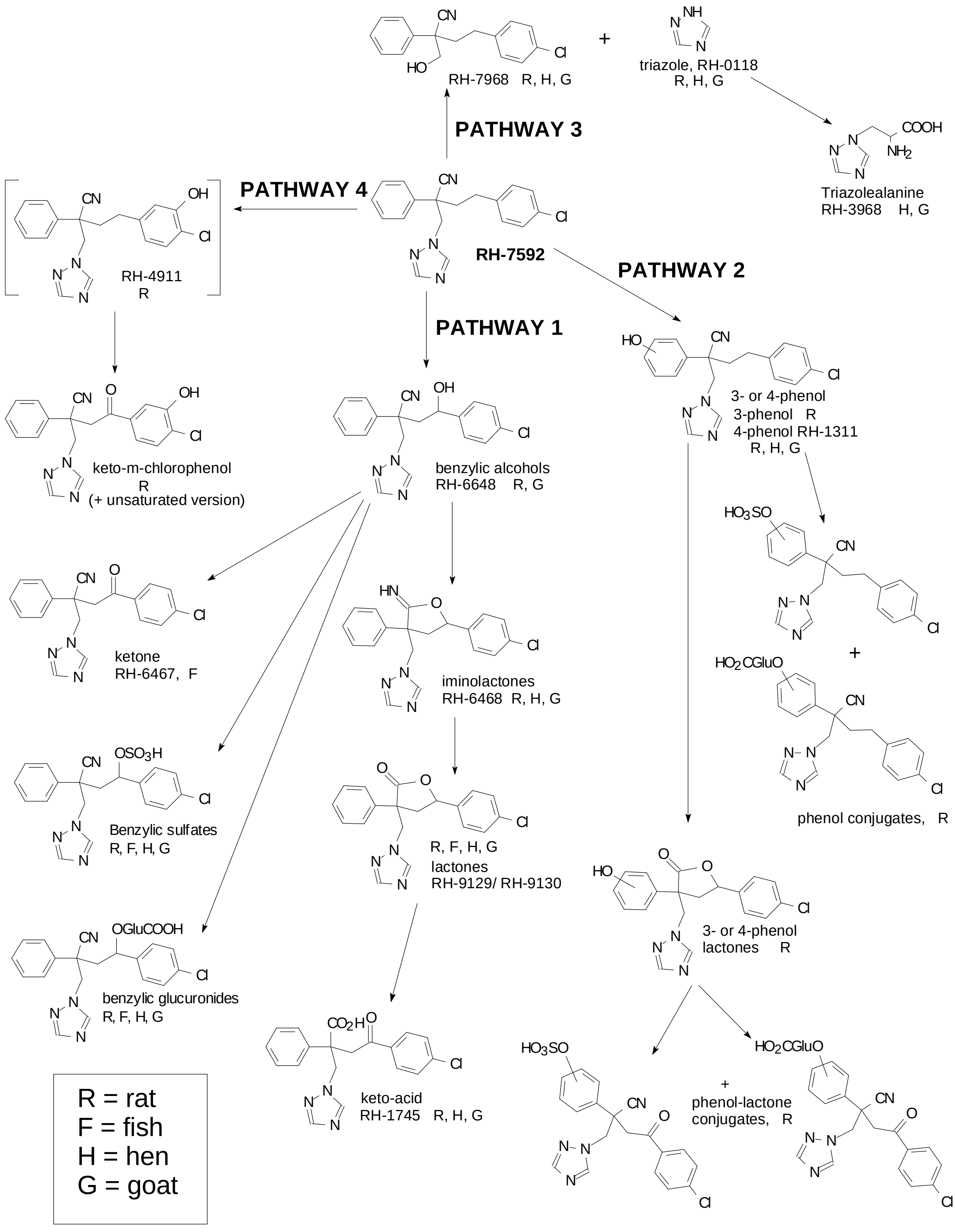
The metabolic pathways for fenbuconazole in laying hens proposed by the manufacturer are shown in Figure 3.

Figure 3. Metabolic pathways of fenbuconazole in laying hens (Batra,1997).



Fenbuconazole appeared to be extensively metabolised in all the animal species studied. Residues have been found which result from four metabolic pathways. The first is oxidation at the benzylic carbon linked to the chlorophenyl ring to produce the isomeric benzylic alcohols and their sulfate and glucuronide conjugates, the iminolactones, lactones, and the keto acid. The second pathway is oxidation of the unchlorinated benzene ring to produce the 3- and 4-phenols and their conjugates. Combinations of paths 1 and 2 produce phenol-lactones and their conjugates. The third path is cleavage of the triazole moiety which produces free triazole and its conjugates, and the alcohol RH-7968. The fourth pathway consists in hydroxylation of the chlorophenyl ring in the 3- position, followed by oxidation to the ketone of the chlorophenol and elimination of HCN to yield the α-unsaturated ketone. An overview of the metabolism of fenbuconazole in animals is shown in Figure 4.

Figure 4. Overview of the metabolic pathways of fenbuconazole in animals (Costlow 1997a; Batra 1997; Ross 1997b).



Plant metabolism

Wheat. In a study carried out in the USA (Pennsylvania) in 1987 phenyl- and triazole-labelled fenbuconazole (radiochemical purities 99.0 and 98.5%) were applied as emulsifiable concentrates twice to field-grown winter wheat at 0.4 kg ai/ha 218 and 226 days after sowing (growth stages 45 and 55). Samples were taken at harvest, 39 days after the last treatment. All samples were analysed by LSC after combustion, and were also soxhlet-extracted with methanol. Straw and chaff were further extracted with sodium hydroxide/methanol, and grain with HC1/methanol. The extracts were evaporated to dryness and reconstituted in ethyl acetate/water and the aqueous layers were partitioned with butanol, chloroform and hexane. The resulting organic and aqueous phases were analysed by TLC, HPLC and GC-MS. The unextractable material was combusted and analysed by LSC.

The total ^{14}C residues at harvest (expressed as fenbuconazole equivalent) were 0.44 mg/kg in the grain, 10.6 mg/kg in the straw and 6.1 mg/kg in the chaff from the phenyl label, and 0.14 mg/kg in the grain, 9.8 mg/kg in the straw and 6.1 mg/kg in the chaff from the triazole. On re-analysis of the samples after drying and freezer storage before extraction, the residues found were 0.05 mg/kg in the grain, 18.3 mg/kg in the straw and 7.6 mg/kg in the chaff from the phenyl label, and 0.53 mg/kg in the grain, 13.5 mg/kg in the straw and 6.4 mg/kg in the chaff from the triazole label.

The percentage recoveries of radiolabelled material in the separate extracts are shown in Table 7 and the identities of components of the residues in Table 8. Five components were identified. In the grain the two major compounds identified with the triazole label were triazolylalanine (0.25 mg/kg) and triazolylacetic acid (0.11 mg/kg); the parent was detected, but at less than 0.01 mg/kg. In the straw and chaff the three components identified were fenbuconazole, the lactone and the ketone at respective levels of 8.8-11.8, 1.1-1.4 and 0.59-0.62 mg/kg in the straw, and 3.7-4.5, 0.45-0.49 and 0.16-0.19 mg/kg in the chaff (all residues expressed as fenbuconazole).

Because two sugar conjugates of the chlorophenol RH-4911 were identified in peanut vines in another metabolism study the straw from the wheat treated with the triazole label was re-examined. After soxhlet extraction with methanol the extract was evaporated to dryness, re-constituted in water and the aqueous solution partitioned with ethyl acetate and butanol. The ethyl acetate extract was then partitioned with chloroform to remove the parent compound and the lactone metabolite, and the resulting ethyl acetate and butanol extracts were analysed by TLC. One of the two sugar conjugates, identified as the glucoside of RH-4911, was present at a level of 0.43 mg/kg expressed as fenbuconazole (Hawkins 1989, 1994; Sharma, 1993b).

Table 7. Distribution of extractable radioactivity in wheat at harvest.

Extract	^{14}C, % of total in sample					
	Grain		Straw		Chaff	
	Phenyl label	Triazole label	Phenyl label	Triazole label	Phenyl label	Triazole label
Soxhlet						
Ethyl acetate	25.3	5.4	85.5	79.8	69.2	77.6
Butanol	15.4	6.8	2.4	1.6	6.6	1.8
Aqueous	4.9	32.6	0.4	1.0	0.2	0.9
Blender						
Ethyl acetate	{11.6	1.1	-	-	-	-
Aqueous	{	45.5	-	-	-	-
Base						
Hexane	-	-	0.7	-	-	-
Chloroform	-	-	1.1	1.5	3.2	-
Ethyl acetate	-	-	4.1	6.0	2.8	3.7
Butanol	-	-	1.2	1.8	2.8	-
Aqueous	-	-	4.9	5.7	2.7	8.9
Unextractable	43.7	6.9	-	2.8	12.1	7.2
Total	100.9	98.3	100.3	100.2	99.6	100.1

Table 8. Distribution of fenbuconazole and its identified metabolites in wheat.

Compound	^{14}C, % of total in sample (mg/kg as fenbuconazole)					
	Phenyl label			Triazole label		
	Grain	Straw	Chaff	Grain	Straw	Chaff
Fenbuconazole	12.4(<0.01)	64.9 (11.8)	58.6 (4.49)	1.4 (<0.01)	60.2 (8.81)	57.9 (3.67)
Lactone	1.6 (<0.01)	7.7 (1.40)	6.5 (0.49)	0.1 (<0.01)	7.5 (1.10)	7.1 (0.45)
Ketone	0.0 (<0.01)	3.2 (0.59)	2.2 (0.16)	0.0 (<0.01)	4.2 (0.62)	3.0 (0.19)
Triazolylalanine	-	-	-	48.4 (0.25)	-	-
Triazolylacetic acid	-	-	-	20.1 (0.11)	-	-

Wheat and grapes. In a laboratory study of uptake and translocation under ambient temperatures and fluorescent lighting, wheat (Fielder variety) and grape plants (Delaware variety) were grown hydroponically in Hoagland nutrient solution for 14 days. Plants at the 3 to 4 leaf growth stage were treated with phenyl-labelled fenbuconazole (radiochemical purity >98%) either through the roots by adding 2.55 mg/l fenbuconazole to the solution or by applying 1 µl of 0.8% EC formulated fenbuconazole to one of the leaves of the plant with a microcapillary tube. Samples were taken at intervals for 14 days. The whole plants were autoradiographed and the combusted foliage and roots, the residual nutrient solution and the root washings were radioassayed. The results are given in Table 9. TLC analyses indicated that the test substance was not degraded significantly. The assays and autoradiograma indicated that uptake from the solution through the roots to the aerial plant parts was rapid, and that the radioactivity migrated rapidly to the tip of the leaf which had been treated. There was evidence of movement of the radioactivity downwards and to adjacent leaves from day 5 in wheat. The same pattern of movement was observed in grapes, but was generally slower. The radioactivity was shown to be highly mobile in the xylem and slightly mobile in the phloem (Sharma, 1993a).

Table 9. Distribution of radioactivity in wheat and grape plants grown hydroponically after treatment of roots or leaves with phenyl-labelled [^{14}C]fenbuconazole.

Crop	Days	^{14}C, mg/kg as fenbuconazole					
		Root treatment			Leaf treatment		
		Foliage	Roots	Nutrient solution	Root	Treated leaf	Untreated leaf
Wheat	1	173	867	1.49	0.06	85	0.7
	3	302	940	0.69	0.13	223	3.1
	5	352	208	0.25			
	7	269	319	0.15	0.36	137	3.8
	14	301	425	0.09	0.13	203	2.5
Grapes	1	1	938	1.95	0.05	167	0.08
	3	37	596	0.51	0.05	90	0.7
	5	112	219	0.25	0.16	119	1.9
	7	53	726	0.21	0.17	104	0.9
	14	51	284	0.14	0.54	107	1.3

Peanuts. In a field experiment in 1990 in North Carolina, peanuts (Florigiant variety) were treated four times at monthly intervals with either phenyl- or triazole-labelled fenbuconazole (formulated as 6% EC formulations) at 0.57 kg ai/ha. The field plots were irrigated throughout the study. Nuts (separated into kernels and shells) and vines were harvested 28 days after the last treatment. Each sample was ground to a fine powder and radioassayed after combustion to determine the total radioactive residues (Table 10).

Table 10. Total radioactive residues in peanuts and vines after treatment with either phenyl- or triazole-labelled fenbuconazole.

Sample	^{14}C, mg/kg as fenbuconazole	
	Phenyl label	Triazole label
Vine	13.68	13.49
Shells	1.04	1.30
Kernels	0.064	3.98

Each sample was extracted with methanol/water. Vines and shells were further extracted with KOH/methanol and kernels also with HCl/methanol. The total extraction efficiencies were >88% for all samples except kernels with the phenyl label from which only 44% of the initial activity could be extracted even after treatment with both acid and base.

Vine extracts were analysed by TLC using various solvent systems and the spots were autoradiographed to identify metabolites by comparison with reference standards. Some extracts were purified on C-18 columns and by TLC and HPLC. The structures of the sugar conjugates were established by β-glucosidase and acid hydrolysis, acetylation, and examination of the products by mass spectrometry. The shell extracts were passed through C-18 columns which were eluted with water and acetonitrile. The acetonitrile fraction was co-chromatographed with standards by TLC.

Because of the low levels of residue in the kernels from the phenyl label compared with the triazole label, it was postulated that the kernels would contain only polar residues. When the kernels were further extracted with KOH/methanol/water there was no further release of radioactivity. It was concluded that the remaining 56% of the phenyl-labelled residue was bound. The extractable residue was reconstituted in water and partitioned with ethyl acetate and butanol. These extracts were shown to contain similar components to those from the conjugates found in the vines.

The extractable triazole-labelled residues in the kernels and shells were analysed by TLC and cation-exchange column chromatography (which separated triazolylacetic acid and triazolylalanine.

The distributions of the identified compounds are shown in Table 11.

Table 11. The nature of radioactive residues in peanuts treated with triazole-labelled fenbuconazole.

Compound	^{14}C, % of TRR and mg/kg as fenbuconazole					
	Vine		Shell		Kernel	
	%	Mg/kg	%	mg/kg	%	mg/kg
Fenbuconazole	48.3	6.73	33.5	0.45	0	<0.002
Lactone A, RH-9129	4.48	0.62	0.97	0.013	0	<0.002
Ketone, RH-6467	8.82	1.23	7.51	0.10	0	<0.002
Iminolactones, RH-6468	0	0	2.45	0.033	0	<0.002
Sugar conjugates of RH-4911	23.8	3.32	17.2	0.23	0.50	0.019
(glucoside and malonylglucoside)						
Triazolylalanine, RH-3968	6.57	0.91	22.2	0.30	91.8	3.50
Triazolylacetic acid, RH-4098	0	0	4.24	0.057	1.9	0.07
Unknown	2.08	0.028	1.36	0.052	-	-
Bound	8.00	1.11	9.9	0.13	4.4	0.17

The identified compounds accounted for 88-94% of the total radioactivity recovered from all of the samples except the kernels containing the phenyl label.

The vines and shells contained mainly compounds in which the framework of the original molecule remained intact, namely fenbuconazole, small quantities of the lactone RH-9129, the ketone RH-6467 and the iminolactones RH-6468, and larger quantities of the sugar conjugates (mainly glucoside with some malonylglucoside) of the phenol RH-4911. The kernels contained traces of these but the main residue consisted of triazolylalanine and triazolylacetic acid in which only the triazole ring remained (Sharma, 1992c, 1993b).

Peaches. A study was carried out in the USA (Pennsylvania) in 1987. Phenyl- and triazole-labelled fenbuconazole (radiochemical purities 98.5 and 98.8%), formulated as emulsifiable concentrates were sprayed five times on outdoor peach trees, from blossom until 22 days before harvest (no rainfall on the days of application, except the first where 3.8mm fell, average temperature 21°C), at a rate of 0.2 kg ai/ha (0.7 for apples). Samples were taken at harvest and analysed by LSC after combustion. The samples were also extracted with methanol in Soxhlets, 'polytrons' and blenders. The extracts were evaporated to dryness and reconstituted in chloroform/water, and the resulting aqueous layers were partitioned with ethyl acetate, ether and butanol. The organic and aqueous phases were analysed by TLC, HPLC and GC-MS. The unextractable material was combusted and analysed by LSC.

The total ^{14}C residues in the peaches at harvest (expressed as fenbuconazole) were 0.08 mg/kg from the phenyl label and 0.12 mg/kg from the triazole label.

The distributions of the radiolabels are shown in Tables 12 and 13. The two main components of the residue were triazolylalanine and the parent compound, which were present at levels of 0.06 and 0.02-0.04 mg/kg (as fenbuconazole) respectively. The lactone and triazolylacetic acid were also identified. Five unknown compounds were present at levels below 0.01 mg/kg.

TLC of the ethyl acetate extract showed that a component of the residue was similar to the conjugates of RH-4911 found in peanuts. Co-chromatography with the conjugates identified in the peanut metabolism study showed that they were identical (Hawkins, 1988; Sharma, 1993b).

Table 12. Distribution of extractable radioactivity in peaches at harvest.

Extract	% of extractable ^{14}C in fruit	
	Phenyl label	Triazole label
Chloroform	70.5	21.5
Ethyl acetate	15.7	-
Ether	-	0.3
Butanol	5.8	7.9
Aqueous	1.6	64.6
Unextractable	6.4	4.6
Total	100.0	98.9

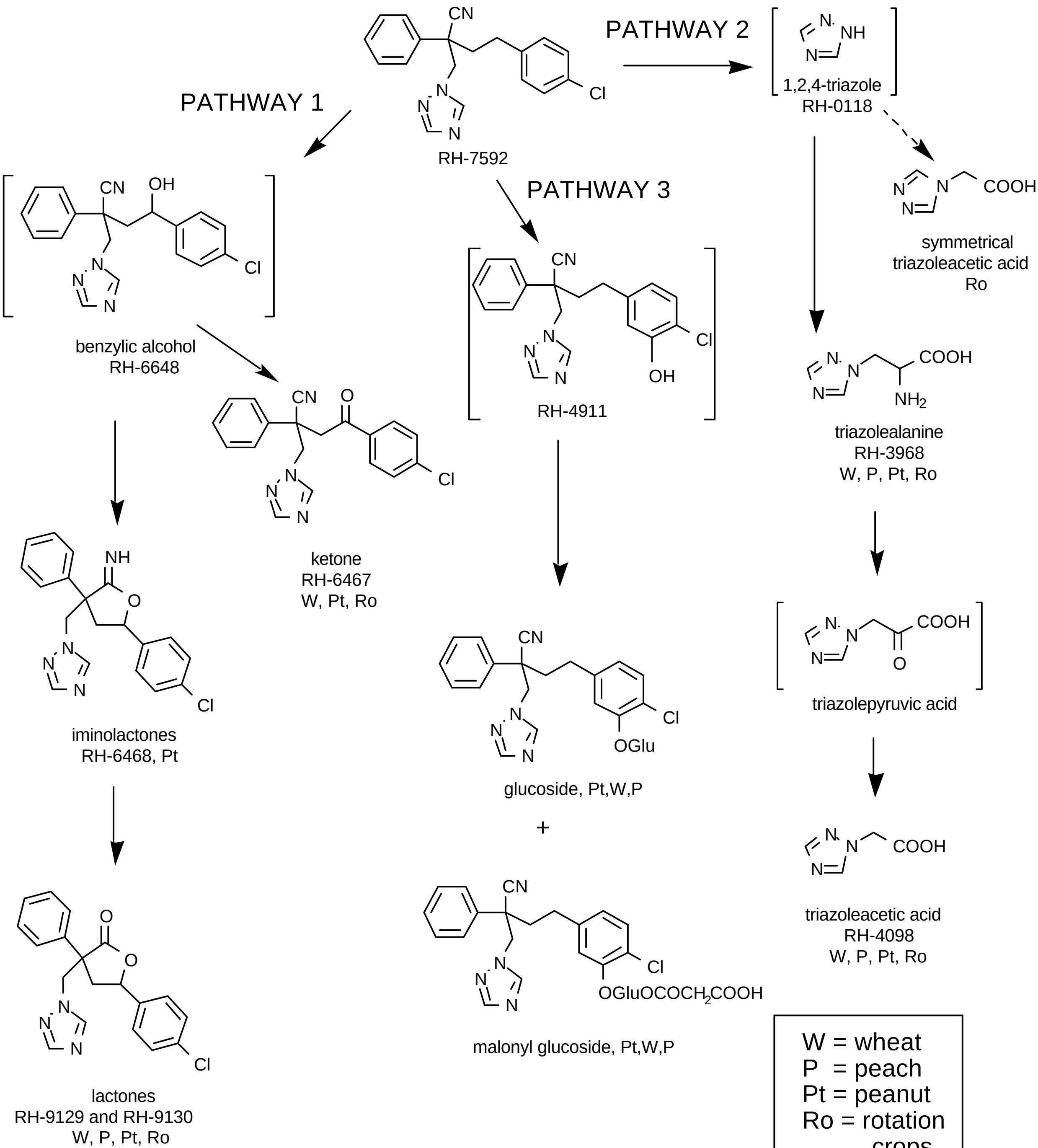
Table 13. Fenbuconazole and its metabolites in fruit at harvest.

Compound	^{14}C, % of TRR (mg/kg as fenbuconazole)	
	Phenyl label	Triazole label
Parent	45.0 (0.04)	15.5 (0.02)
Lactone	14.2 (0.01)	4.3 (<0.01)
Triazolylalanine	-	47.5 (0.06)

Compound	^{14}C, % of TRR (mg/kg as fenbuconazole)	
	Phenyl label	Triazole label
Sugar conjugates of RH-4911	-	4.4 (<0.01)
Triazolyl acetic acid	-	6.7 (<0.01)

A further study on sugar beet in 1997 was referenced by the manufacturer but not submitted for review. The manufacturer has proposed the metabolic pathway in plants shown in Figure 5.

Figure 5. Metabolic pathways of fenbuconazole in plants (Costlow, 1997a).



Metabolism and distribution in rotational crops

In a field trial in 1987 in the USA (Pennsylvania) wheat, turnips and collards were grown in bare soil treated with either phenyl- or triazole-labelled fenbuconazole (formulated as emulsifiable concentrate, radiochemical purities >98.5%) at a rate of 8.96 kg ai/ha. The crops were planted 30, 99 and 365 days after application and sampled at various times up to harvest. The samples were analysed by LSC after combustion, and extracted as in the wheat metabolism study. The resulting organic and aqueous phases were analysed by TLC and HPLC. The unextractable material was combusted and the ^{14}C determined by LSC.

The distribution of radioactivity in the plants is shown in Table 14.

Table 14. Radioactive residues from phenyl- and triazole-labelled fenbuconazole in rotational crops following application to bare soil.

Sample	Days between application and planting	Days between planting and sampling	^{14}C, mg/kg (as fenbuconazole)	
			Phenyl label	Triazole label
Collards	30	47	0.43	4.3
		56	0.32	5.5
		83	0.19	7.4
	99	52	0.59	15.0
		61	0.38	18.0
		88	0.26	25.0
	365	77	0.24	37.0
		94	0.30	28.0
		132	0.24	17.0
Turnips				
Tops/Roots	30	31	1.78/-	4.1/-
		56	0.78/0.48	3.5/3.4
		75	0.86/0.39	16.0/5.1
	99	52	0.45/-	11.0/-
		61	0.42/0.31	25.0/58.0
		88	0.36/0.10	30.0/18.0
	365	77	-/-	34.0/-
		94	-/-	20.0/-
		152	0.21/0.29	6.9/4.8
Wheat				
Straw = S	30	258	0.63(S)/0.30(H)	5.7(S)/14.0(H)
Head = H		273	1.4(S)/0.69(H)	11.0(S)/17.0(H)
Grain = G		281	1.6(S)/0.78(C)/0.52(G)	14.0(S)/13.0(C)/29.0(G)
Chaff = C	99	314	49.0(S)/0.57(C)/0.46(G)	47.0(S)/70.0(C)/122.0(G)
	365	282	0.5(S)/0.92(H)	6.6(S)/16.0(H)
		302	0.58(S)/1.4(H)	7.4(S)/21.0(H)
		318	1.2(S)/0.88(C)/2.3(G)	9.1(S)/8.2(C)/43.0(G)

In collards the total ^{14}C from the phenyl label residue expressed as fenbuconazole at harvest increased slightly from 0.19 mg/kg in crops grown in soil aged for 30 days to 0.24 mg/kg in crops grown in soil aged for 365 days, and that from the triazole label from 7.4 to 17 mg/kg (25 mg/kg for crops grown in soil aged for 99 days).

The total ^{14}C from the phenyl label decreased in turnip tops at harvest from 0.86 mg/kg from the 30-day planting to 0.21 mg/kg from the 365-day, and in the roots from 0.39 to 0.29 mg/kg. The ^{14}C levels from the triazole label at harvest were 16.0, 30.0 and 6.9 mg/kg in the tops and 5.1, 18.0 and 4.8 mg/kg in the roots from the 30-, 99- and 365-day plantings.

In wheat the total ^{14}C at harvest from the phenyl label was equivalent to 1.6, 49 and 1.2 mg/kg in the straw and 0.52, 0.46 and 2.3 mg/kg in the grain from the successive plantings. The corresponding levels from the triazole label were 14, 47 and 9.1 mg/kg in the straw and 29, 122 and 43 mg/kg in the grain. The manufacturer stated that the high levels in the wheat planted 99 days after treatment were possibly due to the low yield and near crop failure two weeks before harvest.

The recoveries of ^{14}C from the triazole label are shown in Table 15. Only samples at harvest with this label were completely analysed: analysis of selected samples with the phenyl label did not show any compound that was not seen with the triazole label. Two major metabolites were identified as triazolylacetic acid and triazolylanine, which accounted respectively 4.4-19.0 and 68-76% of the total radioactivity in collards, 19-21 and 58-62% in turnip tops, 1.7-7.8 and 81-90% in turnip roots, 27-34 and 56-65% in wheat grain, and 49-68 and 8.1-29% in wheat straw. Three other components were identified as the parent compound and the ketone and lactone metabolites, none of which accounted for more than 4% of the total radioactivity in any of the crops (the highest residue was 0.45 mg/kg fenbuconazole in wheat straw). The distributions of the identified compounds are shown in Table 16 (O'Dowd, 1990b; Hawkins, 1992).

Table 15. Distribution of ^{14}C at harvest in rotational crops treated with thiazole-labelled fenbuconazole.

Sample	Days between application and planting	^{14}C, % of TRR		
		Ethyl acetate phase	Aqueous phase	Unextractable
Collards	30	2.3	91.6	6.1
	99	1.0	92.1	6.9
	365	0.8	91.1	8.1
Turnips	30	3.0/2.7	83.8/91.6	13.2/5.7
Tops/Roots	99	1.1/0.7	89.7/96.1	9.2/3.2
	365	1.1/0.9	84.9/92.0	14.0/7.1
Wheat				
Grain/Straw	30	0.2/9.6	94.8/87.9	5.0/2.5
	99	0.3/2.7	94.5/92.2	5.2/5.1
	365	0.1/4.5	94.5/87.0	5.3/8.5

Table 16. Distribution of identified compounds in residues from treatments with triazole-labelled tebuconazole.

Sample	Days between application and planting	^{14}C, % of TRR and (mg/kg as fenbuconazole)				
		Parent	Lactone	Ketone	TAA	TA
Collards	30	0.8 (0.06)	-	-	4.4 (0.62)	76.0 (5.6)
	99	-	-	-	18.5 (4.6)	67.7 (17.0)
	365	-	-	-	14.4 (2.4)	71.4 (12.0)
Turnips						
Tops	30	1.6 (0.24)	1.2 (0.17)	0.4 (0.06)	18.8 (2.8)	58.3 (8.8)
	99	-	-	-	21.3 (6.6)	62.4 (19.0)
	365	-	-	-	19.6 (1.4)	60.7 (4.2)
Roots	30	1.5 (0.08)	0.4 (0.02)	-	2.6 (0.13)	83.9 (4.3)
	99	-	-	-	1.7 (0.31)	89.8 (16.0)
	365	-	-	-	7.8 (0.37)	80.9 (3.9)
Wheat						
Grain	30	-	-	-	26.8 (7.8)	64.8 (19.0)
	99	-	-	-	31.8 (39.0)	57.6 (70.0)
	365	-	-	-	34.2 (15.0)	55.8 (24.0)
Straw	30	3.4 (0.45)	0.9 (0.11)	0.5 (0.06)	55.5 (7.8)	26.0 (3.6)
	99	-	-	-	68.1 (32.0)	8.1 (3.8)
	365	0.2 (0.05)	0.2 (0.03)	-	49.0 (4.4)	29.2 (2.7)

TAA: triazoleylacetic acid
TA: triazolylalanine

Two further studies were carried out with phenyl-labelled fenbuconazole (radiochemical purities >99% and >96%). In one a 3% EC formulation was applied to sandy loam soil at 4 x 0.28 kg ai/ha. Lettuce (Buttercrunch variety), radishes (White icicle), and sorghum (Pioneer 8222) were planted 210 days after treatment and harvested at maturity (lettuce and radishes 291 days, sorghum 399 days after treatment).

In the other trial a 2% EC formulation was applied to sandy loam soil at 3 x 0.07 kg ai/ha. Lettuce (Waldmans Greenleaf), carrots (Imperator 58) and barley (BB82-425) were planted 35 and 260 days after treatment. Samples of lettuce and barley from the 35-day plantings were taken 133 days after treatment and the three crops were harvested at maturity: lettuce 210, carrots 288 and barley 253 days after treatment (DAT). Barley planted 260 DAT was sampled immediately after treatment, and all three crops were sampled at maturity: 360 DAT for lettuce, 260 DAT for carrots and barley].

All the samples were assayed for ^{14}C after combustion. Those with total radioactive residues of >0.01 mg/kg as fenbuconazole were Soxhlet-extracted with methanol and partitioned with sodium chloride and dichloromethane. After clean-up on silica gel, Florisil and C-18 columns the extracts were analysed by GLC. The analyses revealed no quantifiable residues of fenbuconazole (one sample of barley forage showed 0.008 mg/kg) or the metabolites RH-9129, RH-9130 and RH-6467. The limits of determination were 0.01 to 0.05 mg/kg depending on the sample. The samples were therefore analysed by TLC with radiometric detection and co-chromatography with reference standards. The TLC spots were characterized as either “non-polar” (radioactivity in the region of metabolite standards) or “polar” according to their Rf values. The extracts from the above extraction procedure were radioassayed by LSC, and the crop samples remaining after extraction were radioassayed after combustion.

The total radioactive residues and the characteristics of the residue components are given in Tables 17 and 18.

Table 17. Radioactive residues from phenyl-labelled fenbuconazole in rotational crops after application to bare soil at 4 x 0.28 kg ai/ha.

Sample	TRR, mg/kg as fenbuconazole	Extracted		Bound		Extracted, non-polar		Extracted, polar	
		%	mg/kg	%	mg/kg	%	mg/kg	%	mg/kg
Lettuce	0.039	55	0.022	41	0.016	35	0.013	20	0.008
Radish roots	0.008								
Radish leaves	0.033	51	0.017	42	0.014	27	0.009	27	0.009
Sorghum forage	0.039	79	0.030	23	0.009	40	0.015	39	0.015
Sorghum grain	0.0047								
Sorghum stover	0.033	28	0.009	43	0.017				

After application at 4 x 0.28 kg ai/ha the TRR in all samples was below 0.04 mg/kg. The residues in radish roots and sorghum grain were below 0.001 mg/kg, and in lettuce, radish leaves and sorghum forage between 0.03 and 0.04 mg/kg and sorghum stover residue was 0.033 mg/kg.

Table 18. Radioactive residues from phenyl-labelled fenbuconazole in rotational crops after application to bare soil at 3 x 0.07 kg ai/ha.

Sample	TRR, mg/kg as fenbuconazole	Extracted		Bound		Extracted, non-polar		Extracted, polar	
		%	mg/kg	%	mg/kg	%	mg/kg	%	mg/kg
35-day planting									
Lettuce	0.004								
Carrot root	0.008								
Carrot leaf	0.008	44	0.004	38	0.003				
Barley forage	0.016	46	0.007	19	0.003				
Barley grain[1]	0.008								
Barley straw	0.019	52	0.01	16	0.003	38	0.007	14	0.003
260-day planting									
Lettuce	0.009	81	0.007	33	0.003				
Carrot root	0.005								
Carrot leaf	0.016	69	0.011	31	0.005	13	0.002	61	0.009
Barley forage	0.082	73	0.06	16	0.013	21	0.017	52	0.042
Barley grain[1]	0.033								
Barley straw	0.162	70	0.113	32	0.051	23	0.037	47	0.075

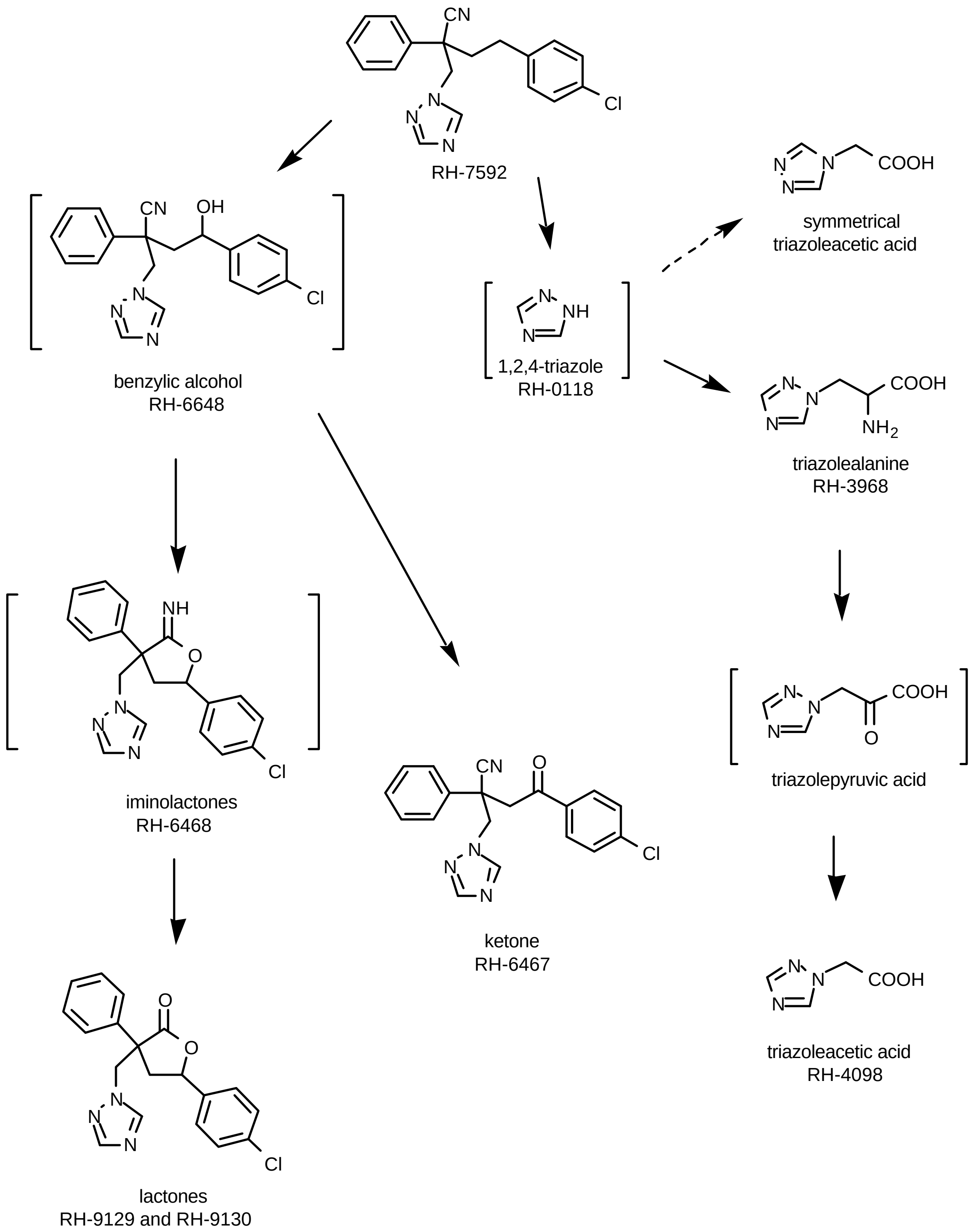
[1] Sample was not sufficient for further analysis

After application at 3 x 0.07 kg ai/ha, the residues in lettuce and carrot roots were below 0.01 mg/kg. The TRR in the grain samples was much lower from the 35-day than the 260-day planting. The company postulated that this may be because fenbuconazole is not translocated well via the roots, whereas degradation products formed in the soil may be taken up by the plants after a planting interval of 260 days. The highest residue found was 0.16 mg/kg, in barley straw. Only two samples (barley straw and forage) in the two studies had residues above 0.05 mg/kg.

TLC analyses confirmed the GLC results. No individual component was found above 0.03 mg/kg equivalents. The non-polar components altogether accounted for 13-38% of the TRR in each sample; the residues were equivalent to 0.002-0.037 mg/kg of fenbuconazole. Another 14-61% of the TRRs was characterized as polar and comprised 2 or 3 components in each sample, the range of residues being equivalent to 0.003-0.076 mg/kg. No single polar component exceeded 0.029 mg/kg fenbuconazole equivalents. The polar components were thought by the manufacturer to be the glucoase conjugates of RH-4911 (Sharma, 1994b).

The manufacturer has proposed the metabolic pathways for fenbuconazole in rotational crops shown in Figure 6 (Batra, 1997).

Figure 6. Metabolic pathways of fenbuconazole in rotational crops



Environmental fate in soil and water/sediment systems

Degradation on soil

An aerobic degradation study was conducted according to the German BBA guidelines. Triazole-labelled fenbuconazole (0.1 mg/kg dry soil, radiochemical purity 99.1%, specific activity 835 MBq/g) was added to Itingen III silt loam, Sisseln I sandy loam and Speyer 2.2 sandy loam soil (100 g dry weight) at 40% field capacity. Samples of each soil were incubated in the dark at 20°C and CO_2 and organic volatiles were trapped in NaOH and ethylene glycol respectively. Samples were taken after 0, 5, 7, 14, 33, 50, 70 and 96 days and extracted several times with acetonitrile and after the later times also with acetonitrile/water, and subsequently with methanol by Soxhlet. Radioactivity in liquid samples was measured directly by LSC, soil extracts were analysed by LSC and TLC and the residual radioactivity in the soil after extraction was determined by combustion and LSC.

Over the course of the experiment (96 days) the recovered ^{14}C for all the soils was always >95% of the applied radioactivity (AR). Volatile compounds were <0.1% of the AR and the evolved $^{14}CO_2$ increased up to 2.3% of the AR after 96 days (in Sisseln I soil). Up to five degradation products were observed in each of the three soils during the course of the experiment but only one was identified, as RH-6467, and constituted <10% of the AR. One product from the Speyer 2.2 incubation and one from the Sisseln I incubation reached 15% of the AR towards the end of the experiment but it is not clear whether this was the same compound in both cases. No other compound accounted for >10 % of the AR. Unextractable radioactivity reached only 15.1% after 96 days in the Itingen III soil but amounted to 45.5 and 36.3% in the Sisseln I and Speyer 2.2 soils respectively. This was consistent with the half-lives where first order kinetics gave 269 (r^2=0.79), 38 (r^2=0.93) and 74 (r^2=0.96) days for Itingen III, Sisseln I and Speyer 2.2 respectively (Mamouni, 1992).

A second aerobic and anaerobic degradation study was conducted according to US EPA guidelines. [^{14}C]Fenbuconazole (radiochemical purity 98 or 98.6%, specific activity 771 or 775 MBq/g) labelled in the phenyl or triazole ring was added to Lawrenceville silty clay loam (200 g, moisture content 15%) or Pasquotank sandy loam (200 g, moisture content 20%) in cellulose at a rate of 1 mg/kg (and at 30 mg/kg to identify degradation products). Some samples were sterilized and all were incubated in the dark at 25°C. After 30 days some samples were purged with nitrogen and then flooded with water to provide anaerobic conditions. CO_2 evolved from samples under aerobic conditions was trapped in NaOH. Samples of soil (10 g) incubated under aerobic conditions were taken after 7, 14, 21, 28, 44, 61, 90, 120, 181, 240 and 363 days, and 25-ml samples of slurry were taken 17, 30 and 60 days after the establishment of anaerobic conditions. The samples were extracted with acetonitrile/acetic acid and after later times with NaOH (the anaerobic slurry was first filtered and dried) and analysed by TLC. Overall radioactivity balances were generally 90-105% during the experiment.

The products RH-9129, RH-9130 and RH-6467 were confirmed by TLC, HPLC and GC-MS as present in both the aerobic and anaerobic incubations with both labels. In the aerobic incubations the concentrations of RH-9129, RH-9130 and RH-6467 reached maxima of <10%, <4.5% and <7.9% respectively. In the aerobic incubations with the triazole label free triazole was also identified at levels up to 13.6%. Levels of $^{14}CO_2$ reached 1.5% from the triazole label and 37% from the phenyl label after 365 days. Apart from this there was no difference between the two label positions and both were used to calculate half-lives for fenbuconazole, assuming first-order kinetics. They were as shown below.

	Lawrenceville (silty clay loam)	Pasquotank (sandy loam)
Aerobic conditions	258 days	367 days
Anaerobic conditions	464 days	655 days

No degradation was observed under sterile conditions during the 363 days of the study (Schieber, 1988a).

In a photolysis study according to US EPA guidelines phenyl-labelled fenbuconazole (10 mg/kg, specific activity 753 MBq/g, 96.2% pure) was applied to the surface of a Camden county sandy loam soil layer (2 g) which was maintained at 26°C and irradiated with a xenon lamp (approx. 150 W/m², of similar intensity to sunlight in New Jersey) for periods of 12 h followed by 12 h darkness for 30 days. Volatile compounds were trapped with polyurethane plugs, KOH and sulfuric acid. Duplicate soil samples were extracted with acetonitrile/acetic acid after 0, 3, 7, 14, 21, 30 days and the extracts analysed by TLC. After extraction the soil was combusted and the unextracted radioactivity quantified by TLC. At all sampling times the overall recovery of radioactivity was >90%.

The levels of trapped volatiles reached 0.02% after 30 days, at which time the unextractable radioactivity reached its maximum of 4%. Fenbuconazole was the only compound identified in the extract and had a half-life of 79 days according to first order kinetics. No degradation observed in the dark control (Wang, 1991a).

Adsorption and desorption

In a study according to US EPA guidelines triazole-labelled fenbuconazole (specific activity 775 MBq/g, purity 98%) was dissolved in 0.01M $CaCl_2$ (0.33, 0.22, 0.11 and 0.03 µg/l) and 10-40 ml aliquots were added in duplicate to five different soils (2-2.5 g). The soils were equilibrated for 24 h at 25°C. After centrifugation the supernatant was removed and desorption was measured by equilibrating the soil with fresh 0.01M $CaCl_2$ (10-40 ml) for 72 h. The ^{14}C in the solutions was quantified by LSC and that in the soils by combustion and LSC. TLC analysis of the solutions showed that fenbuconazole was the only source of radioactivity. The results are shown in Table 19 (Schieber, 1988c).

Table 19 Freundlich adsorption/desorption coefficients for fenbuconazole in five soils.

	Adsorption			Desorption		
	n[1]	K_d	K_{oc}	n	K_d	K_{oc}
Cecil (clay)	0.93	5.1	2185	0.95	7.1	3087
Keeton (loam)	1.01	75.2	5402	1.01	147.7	10625
Lakeland (sand)	1.22	7.6	2607	0.93	2.3	793
Pasquotank (sandy loam)	1.00	115.4	9042	0.96	132.2	10328
Lawrenceville (silty clay loam)	0.85	20.1	2884	0.87	33.0	4714

[1] Slope of Freundlich adsorption isotherm

<u>Mobility in soil</u>

In a study according to US EPA guidelines Pasquotank sandy loam soil was treated with triazole- or phenyl-labelled fenbuconazole (1 mg/kg, specific activity 775 or 771 MBq/g, purity 98%) and aerobically aged at 25 °C for 30 days. At this time <1% of the AR had been lost as $^{14}CO_2$ and >90% was fenbuconazole. Duplicate columns, 5.5 cm diameter, were filled to a height of 30 cm with untreated Pasquotank soil, wetted from the bottom until saturated and allowed to drain overnight. Aged treated soil (40 g) was added and the columns were leached for 7-14 days with 1000 ml water (42 cm depth). The leachate was assayed for radioactivity by LSC and the soil column divided into segments, extracted with acetonitrile/acetic acid and analysed by LSC and TLC.

The average ^{14}C balance at the end of the leaching was 98%. Radioactivity in the leachate accounted for 0.2% of the AR and the leachate was not analysed further. About 97% of the radiolabel was located on the top 6 cm segment of the column and was almost all due to fenbuconazole; trace amounts of RH-9129, RH-9130 and RH-6467 were also identified (Schieber, 1988b)

In a study according to German BBA guidelines sieved air-dried German standard soils (2.1, 2.2 and 2.3) were used to fill duplicate glass columns (5 cm diameter) to a height of 30 cm. The soils were saturated from the top and fenbuconazole (15 µg) was added after draining. Water (393 ml, equivalent to 200 mm depth) was passed through the columns for two days and the leachate collected. The leachate was analysed by GLC (recovery 76%, limit of detection 0.75 µg/l) but no fenbuconazole was detected in any sample (Specht, 1992).

Soil dissipation under field conditions

In a study conducted according to German BBA guidelines fenbuconazole (75 g/ha) was sprayed onto bare soil plots (5 m x 5 m) at four sites in Northern Germany (Klein-Offenseth, Bad Oldesloe, Hamburg and Walsrode) in April 1989. Samples (20 per plot) taken to a depth of 15 cm were bulked at 0, 7, 28, 56, 112, 224, 365 and 504 days after application. The soils were extracted with methanol and after clean-up the extracts were analysed by GLC. The limit of detection was 0.01 mg/kg. In the Bad Oldesloe soil RH-9129 was identified after 8 weeks at 5 µg/kg dry soil, but at no other time was RH-9129, RH-9130 or RH-6467 detectable.

Table 20. Fenbuconazole concentrations in German soils at intervals after field application.

Days after application	Fenbuconazole, mg/kg dry soil			
	Bad Oldesloe	Klein-Offenseth	Walsrode	Hamburg
Before application	nd	nd	nd	nd
0	45	29	30	65
7	42	38	34	23
48	22	16	17	17
56	26	19	19	8
112	13	15	11	11
224	9	7	18	19
365	5	6	16	15
504	10	1	15	13

Graphically estimated half-lives for fenbuconazole in these soils were approximately 56, 70, 84 and 28 days. Weather conditions were generally within the 30 year maxima and minima for monthly temperature and precipitation (Bieber *et al.*, 1990).

In a study according to EPA guidelines undertaken at four US locations fenbuconazole was applied twice or five times. At Minnesota (loamy soil) 2 x 0.14 kg/ha were applied to bare soil on 14 and 31 May 1990. At Georgia (loamy sand) 2 x 0.14 kg/ha were applied to bare soil on 19 July and 2 August 1989. At the Northern California site (clay) 5 x 0.22 kg/ha were applied to bare soil on 7 and 21 August, 5 and 21 September and 5 October 1989. At the Southern California site (sandy loam, 5 x 0.22 kg/ha) were applied to wheat on 1, 15 and 29 August and 12 and 26 September.

At all sites there were three treated plots and one control plot (30 x 6 m). Samples were taken at 0, 14, 30, 60, 90, 120, 150, 180, 210, 240, 300, 360, 420, 510 and 550 days after the last application. Five samples were taken from the control and each treated plot, divided into 0-15, 15-30, 30-45, 45-60, 60-90 and 90-120 cm sections, (0-7.5 and 7.5-15 cm in Minnesota) and composited. The soils were extracted with methanol and the residues determined by GLC with a quoted limit of determination of 0.01 mg/kg and recoveries of >90%. The residues found are shown graphically in Figure 7, and the calculated DT-50 and DT-90 values in Table 21.

Figure 7. Concentrations of fenbuconazole in 0-15 cm soil layers (USA, 1989-90).

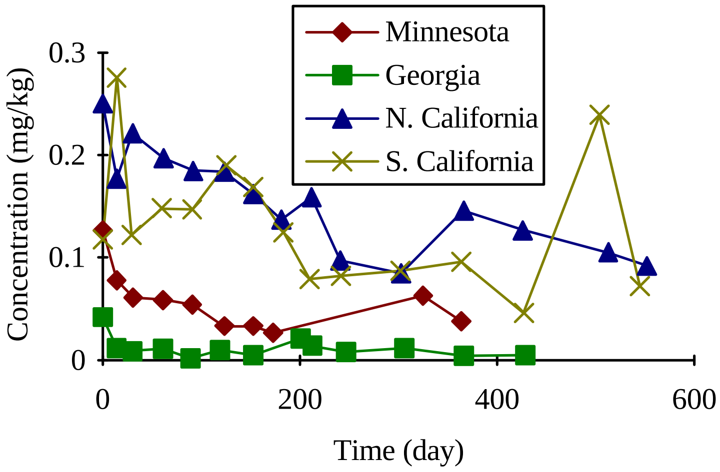


Table 21. DT-50 and DT-90 values for fenbuconazole in 0-15 cm soil layers.

Location	DT-50, days	DT-90, days
Minnesota	30	>364
Georgia	10	360
N. California	425	>550
S. California	200	>545

RH-9130, RH-9129 and RH-6467 were consistently found in the 0-15 cm layer up to the end of sampling in Southern California at levels up to 0.01, 0.031 and 0.016 mg/kg respectively. RH-9129 was found consistently, but at lower levels, in Northern California where fenbuconazole was regularly found at 15-30 cm at varying concentrations up to 0.021 mg/kg throughout the trial. In the trials in Georgia and Minnesota fenbuconazole was not identified below 15 cm and no degradation products were detected at any depth. The soil temperatures were recorded and are shown in Table 22.

Table 22. Soil temperatures at 5 cm depth averaged over 60-day periods in the US trials.

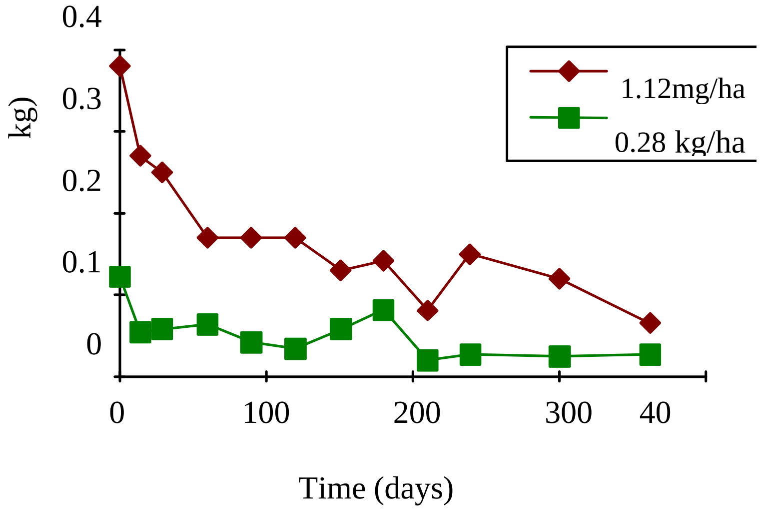
Location	Soil temperature, °C				
	0-60 days	60-120 days	120-180 days	180-240 days	240-300 days
Minnesota	23.3	21.9	5.7	-2.8	-2.4[1]
Georgia[2]	30.6	19.4	10.6	16.7	24.4
N. California	12	7.2	10.9	19.7	22.9[1]
S. California	16.5	7.7	12.2	22.2	35.8

[1]Temperatures at 10 cm depth
[2]Days 240-270 only

No evaporation data were available for the Georgia site but data for the other sites indicated that the only net downward flow of water would be in the 60-120 day period in both the California trials and the 180-270 day period in the Minnesota trial (Deakyne and Stavinski, 1991)

In another study according to EPA guidelines by the same authors fenbuconazole was applied at 0.28 or 1.12 kg/ha to bare plots of 5 x 20 m in Southern California (the same location as above) on 16 May 1991, with a control plot of the same size. Five samples from each plot taken at 0, 14, 29, 60, 90, 120, 151, 180, 210, 239, 300 and 362 days were divided into 0-15 or 0-7.5 and 7.5-15 cm depth segments and the corresponding groups of segments bulked. The samples were analysed for fenbuconazole, RH-9130, RH-9129 and RH-6467 as in the previous study. Recoveries were >88% with a limit of determination of 0.01 mg/kg.

Figure 8. Concentrations of fenbuconazole in 0-15 cm soil layers after applications of 1.12 kg/ha and 0.28 kg/ha (USA, 1991).



At both rates the half-lives of fenbuconazole were approximately 50 days and the DT90s >362 days. The concentrations in the upper layer were generally at least 20 times those in the lower layer and the highest level found in the lower layer at day 60 was approximately 7% of the applied dose.

In the upper layer at the high application rate, RH-9130 reached a peak concentration of 0.063 mg/kg after 14 days, RH-6467 0.047 mg/kg after 29 days and RH-9129 0.05 mg/kg after 90 days. All were still above the limit of detection after 300-362 days. None were detected in the lower layer at any time.

The plots received a total of 10 cm water by precipitation and irrigation during the first 160 days of the experiment, when the average soil temperature at a 20 cm depth was 29.4°C (Deakyne and Stavinski, 1993).

Sterile hydrolysis

In a study conducted according to EPA guidelines triazole-labelled fenbuconazole (0.1 µg/ml final concentration, specific activity 775 MBq/g, purity 99.1%) was incubated at 25°C in the dark in buffered solutions (10 ml) at pH 5, 7 and 9. After 0, 1, 2, 4, 8, 15, 22 and 30 days duplicate samples were extracted with ethyl acetate and analysed by TLC.

The ^{14}C balance averaged 99.4% during the study. Fenbuconazole was the only compound identified and accounted for >97% of the AR at all times. No degradation was detected (O'Dowd 1990a,d).

Aqueous photolysis

In a study according to EPA guidelines phenyl-labelled fenbuconazole (1.5 µg/ml final concentration, specific activity 753 MBq/g, purity 96.2%) in pH 7 phosphate buffer (40 ml) was irradiated with a xenon arc lamp (150 W/m^2, similar to natural sunlight in New Jersey) fitted with a filter to remove wavelengths below 290 nm. The system was maintained at 25°C and subjected to cycles of 12 h light followed by 12 h darkness. Volatile compounds were trapped and duplicate samples were taken at 0, 3, 7, 14, 21 and 30 days, extracted with ethyl acetate and analysed by TLC or HPLC. The overall ^{14}C balance averaged 105% of the AR and the only compound detected was fenbuconazole (always >99% of the AR over the 30-day period). At no time was radioactivity detectable in the traps. Dark controls also showed no degradation (Wang, 1991b).

The photolysis of triazole-labelled fenbuconazole (>99% radiochemical purity, 1.5 mg/l) was also examined in natural pond water. It was moderately unstable in the presence of simulated sunlight (xenon arc lamp) at 24.1±0.6°C. Irradiation was intermittent with light and dark cycles of 12 hours each day for 30 days. Samples were taken at 0, 3, 7, 14, 21, and 30 days and analysed by solvent-solvent extraction, LSC, TLC, and HPLC with radiometric detection. Overall recoveries were high at 101-107% from the irradiated samples and 104-109% from the control samples. In addition to fenbuconazole, which accounted for 75-94% of the total applied radioactivity after 30 days of irradiation, at least eight photodegradation products were detected but none exceeded 10% of the TRR. Radioactivity in water-soluble compounds gradually increased with time, reaching about 18% of the applied radioactivity at day 30. Less than 0.1% of the applied radioactivity was volatile. The identified products included RH-6467 (2.8%), triazole (3.3%) and RH-1311 (2.7%). A mixture of polar compounds totalling 17.9% was stated to be an artifact of the co-solvent used in the sample preparation. Assuming pseudo-first-order kinetics, the half-life of fenbuconazole under the test conditions was calculated to be 86.7 days (Baur, 1994).

Sediment/water systems

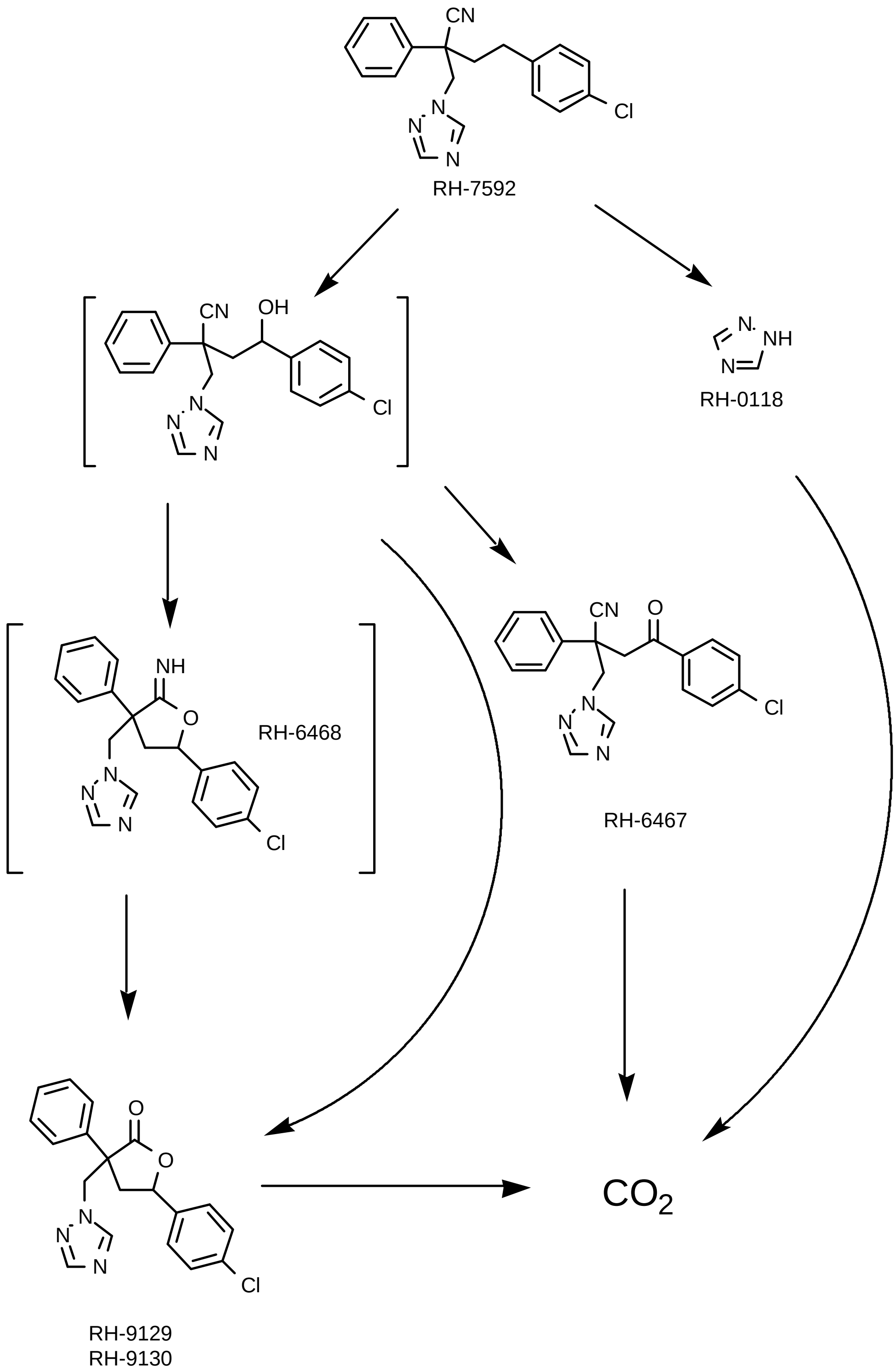
In a study conducted according to BBA guidelines Rhine and pond systems containing water (550 ml, approximately 6 cm depth) and sediment (250 g, approximately 2.5 cm depth) were allowed to equilibrate for approximately 20 days. Triazole-labelled fenbuconazole (0.13 or 0.0134 mg, specific activity 835 MBq/g, purity 97.3%) was then added and traps for volatile compounds were connected to the flasks (NaOH solution to trap CO_2, ethylene glycol to trap organic volatiles). The flasks were incubated in the dark at 20°C while CO_2-free air was continuously passed through and the water phase was agitated. Duplicate samples of water and sediment were taken at 0, 7, 30, 62 and 105 days, and from the higher rate systems also at 6 h, 1, 2 and 14 days. Water samples were passed through solid-phase extraction columns for analyses by HPLC. Sediment samples were extracted with acetonitrile/acetic acid and the extracts analysed by TLC. After extraction, soil was combusted to determine the unextractable radioactivity.

At all time intervals the total ^{14}C balance for both systems and both rates was >93%. There was very little difference between the results from the high and low rates and only results at the high rate are reported here. Volatile compounds accounted for 0.1% of the AR) and evolved $^{14}CO_2$ reached 0.3% after 105 days. Unextractable residues reached 6.6 and 12% of the AR after 105 days in the Rhine and pond sediments respectively. In both systems fenbuconazole was the major source of the radioactivity in the water and sediment and only very small amounts of other unidentified compounds were detected in either system (<0.7% in water, <4% in sediment). The fenbuconazole partitioned from the water to the sediment with a calculated first-order dissipation half-life from the water phase of 3.4 and 1.2 days in the Rhine and pond systems respectively and after 105 days the fenbuconazole in the sediment accounted for 79.4% of the AR in the Rhine system and 80.3% in the pond system (Volkl, 1992).

Biodegradability

In a study conducted according to OECD guidelines standard BOD bottles filled with nutrient medium (280 ml, containing inorganic salts), activated sludge bacterial inoculum and fenbuconazole (2 mg/l, 94-99.5% purity) were incubated in the dark at 20°C, together with negative and positive controls. Duplicate bottles were analysed for dissolved oxygen at days 0, 5, 15, and 28. The chemical oxygen demand (COD) was determined by sample digestion. After 28 days the degradation was 17%. The manufacturer has proposed the degradation pathways for fenbuconazole in soil shown in Figure 9 (Costlow, 1997a; Douglas,1990).

Figure 9. Degradation pathways of fenbuconazole in soil.

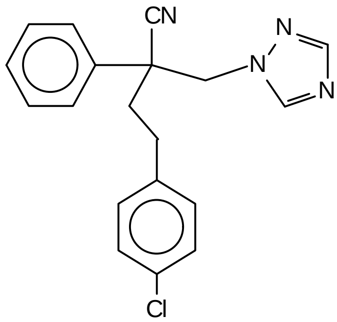
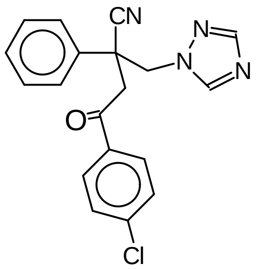
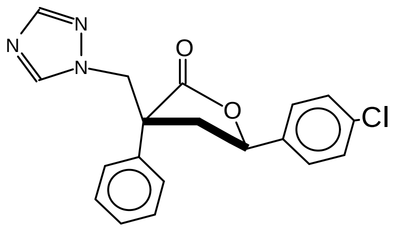
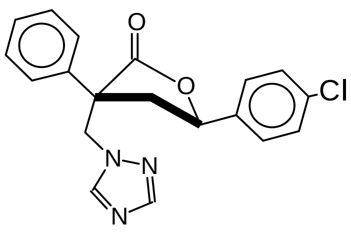
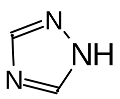
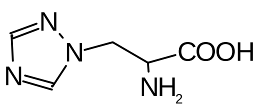


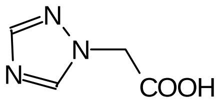
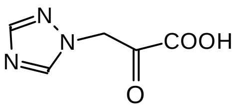
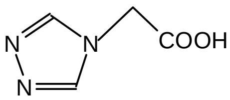
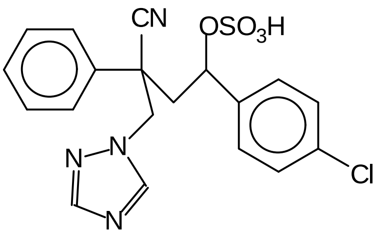
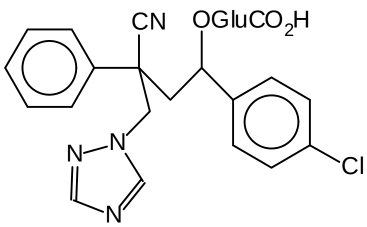
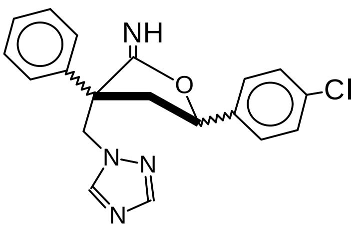
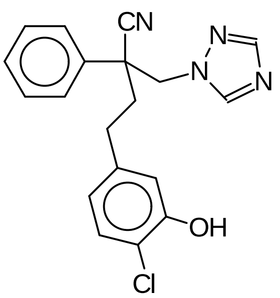
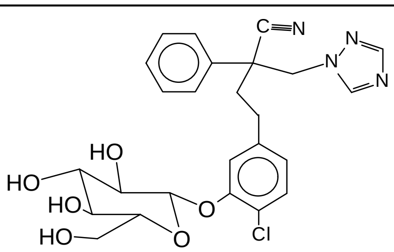
A number of studies on bioaccumulation in fish were also submitted but not reviewed (Forbis 1987; O'Dowd 1988, 1990c).

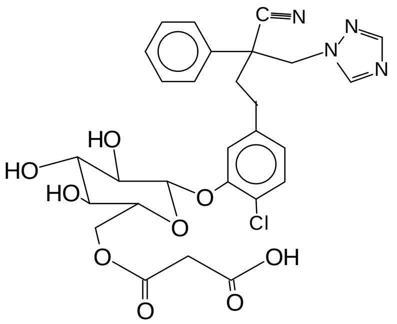
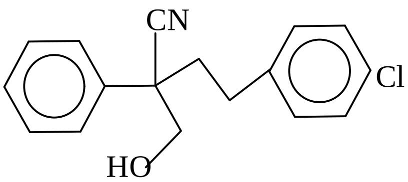
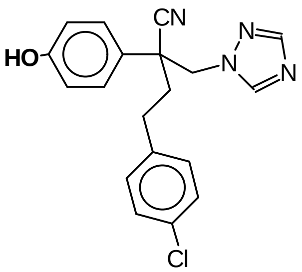
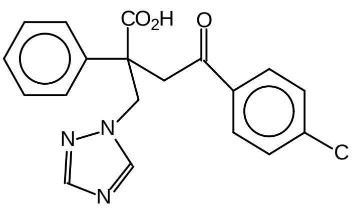
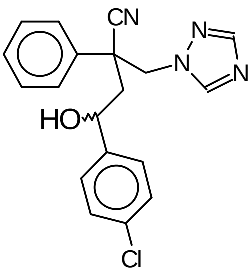
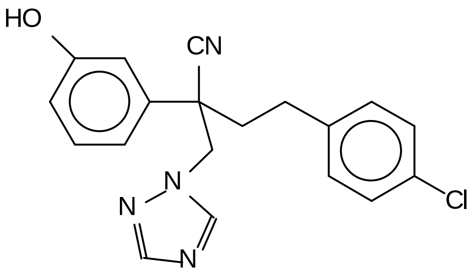
The names and structures of fenbuconazole and its degradation products and metabolites are given in Table 23.

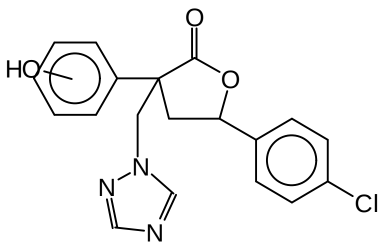
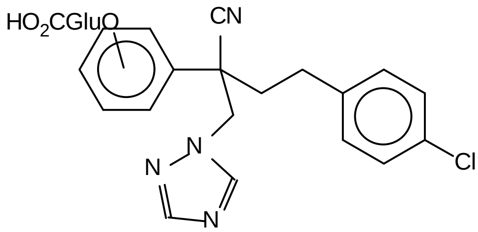
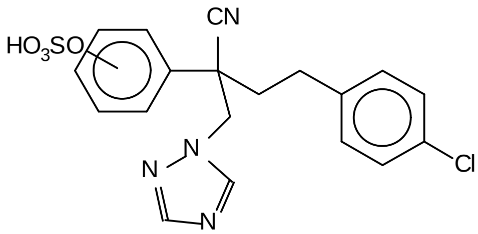
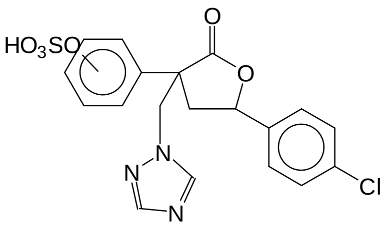
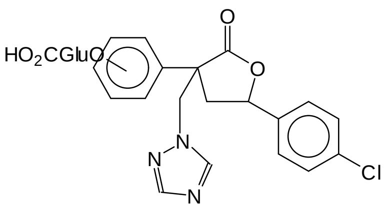
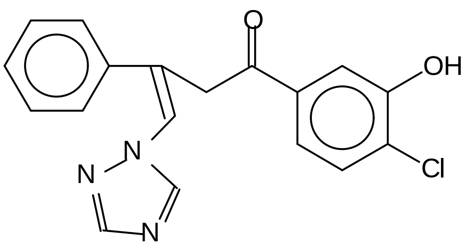
Table 23. Names and structures of fenbuconazole and its degradation products and metabolites.

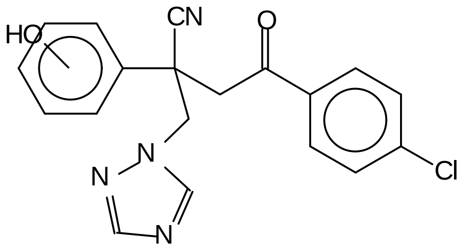
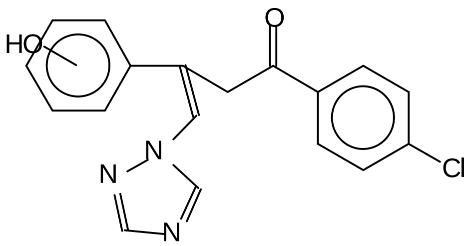
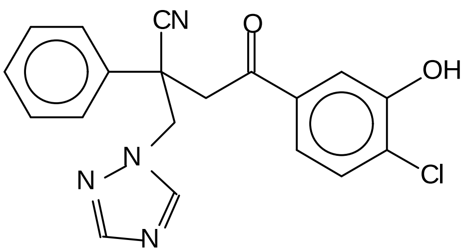
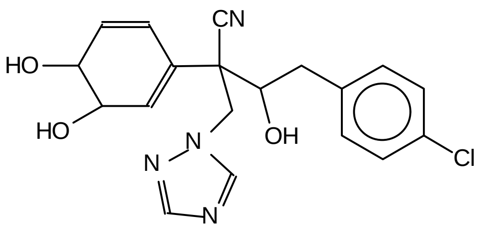
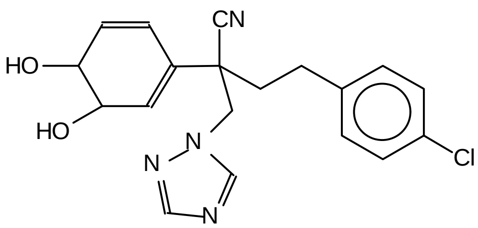
Table 23. Names and structures of fenbuconazole and its degradation products and metabolites.

Number	Structure	Name (code name/ chemical name)	Occurrence
1		fenbuconazole (RH-7592)	Soil, wheat, peach, peanut, rotation crops, hen, goat, fish, rat
2		ketone (RH-6467)	Soil, wheat, peanut, rotation crops, fish, rat
3		lactone A (RH-9129)	Soil, wheat, peach, peanut, rotation crops, hen, goat, fish, rat
4		lactone B (RH-9130)	Soil, rotation crops, hen, goat, rat
5		triazole (RH-0118)	Soil, hen, goat, rat
6		triazolylalanine (RH-3968)	Wheat, peach, peanut, rotation crops, hen, goat

Number	Structure	Name (code name/ chemical name)	Occurrence
7		triazol-1-ylacetic acid (TAA, RH-4098)	Wheat, peach, peanut, rotation crops
8		triazolylpyruvic acid	Peach
9		triazol-4-ylacetic acid symmetrical TAA	Rotational crops
10		benzylic sulfates (RH-6649)	Hen, goat, fish, rat
11		benzylic glucuronides	Hen, goat, fish, rat
12		iminolactones (RH-6468)	Peanut, hen, goat, rat
13		4-chloro-3-hydroxyphenyl derivative (RH-4911)	Rat
14		glucoside of RH-4911	Wheat, peach, peanut

Number	Structure	Name (code name/ chemical name)	Occurrence
15		malonylglucoside of RH-4911	Wheat, peach, peanut
16		intermediate I (RH-7968)	Hen, goat
17		4-phenol (RH-1311)	Hen, goat, rat
18		keto acid (RH-1745)	Hen, goat, rat
19		benzylic alcohols (RH-6648)	Goat, rat
20		3-phenol	Rat

Number	Structure	Name (code name/ chemical name)	Occurrence
21		phenol lactone	Rat
22	 	phenol conjugates	Rat
23	 	phenol lactone conjugates	Rat
24		unsaturated keto m-chlorophenol	Rat

Number	Structure	Name (code name/ chemical name)	Occurrence
25		keto phenol	Rat
26		unsaturated keto phenol	Rat
27		keto *m*-chlorophenol	Rat
28		hydroxy dihydrodiol	Rat
29		dihydrodiol	Rat

METHODS OF RESIDUE ANALYSIS

Analytical methods

Methods have been described for the determination of fenbuconazole, the lactones, the alcohol RH-7968 and the ketone RH-6467 by GLC. The glucose conjugate of the chlorophenol RH-4911 has been determined by HPLC.

Animal products

a) Several studies were submitted, all with the same method for the determination of fenbuconazole, the lactones and 4-(4-chlorophenyl)-2-hydroxymethyl-2-phenylbutyronitrile (RH-7968) in meat, fat, milk and eggs. Samples (apparently from goats and hens, although this was not clear) were first homogenised and then extracted by blending with methanol (hexane for fat). The extract was partitioned with hexane/water (methanol/water for fat). After adding 10% sodium chloride, the aqueous layer was partitioned with dichloromethane, the dichloromethane extract evaporated to dryness and the residual material dissolved in toluene for clean-up and separation on a silica gel column. The first eluate containing RH-7968 was further cleaned up on a Florisil column, and the second fraction containing the parent compound and the lactones on a C-18 solid-phase extraction column. The separate fractions were analysed by capillary GLC with NP detection. The limit of determination was 0.01 mg/kg for fenbuconazole and the lactones and 0.05 mg/kg for RH-7968. Acceptable recoveries were demonstrated at fortification levels of 0.02-0.2 mg/kg (0.06-0.6 mg/kg for RH-7968). Recoveries were higher (124-138%) at the lowest fortification level of 0.01 mg/kg. Acceptable chromatograms were submitted (Filchner *et al.*, 1992,1994a; Haines *et al.*, 1992).

The results of validation experiments are shown in Table 24.

Table 24. Validation of residue method a) for products of animal origin.

Substrate	Analyte	Recovery, %	Limit of determination, mg/kg
Milk	fenbuconazole	94 (mean)	0.01
	lactones	92 (mean)	0.01
	RH-7968		0.05
Muscle	fenbuconazole	98 (mean)	0.01
	lactones	95 (mean)	0.01
	RH-7968		0.05
Liver	fenbuconazole	103 (mean)	0.01
	lactones	102 (mean)	0.01
	RH-7968	79 (mean)	0.05
Kidneys	fenbuconazole	82 (mean)	0.01
	lactones	84 (mean)	0.01
	RH-7968	85 (mean)	0.05
Fat	fenbuconazole	104 (mean)	0.01
	lactones	107 (mean)	0.01
	RH-7968	106 (mean)	0.05
Egg	fenbuconazole	95 (mean)	0.01
	lactones	96 (mean)	0.01
	RH-7968		0.05
Milk	fenbuconazole	80-114	0.01
	RH-9130	74-113	0.01
	RH-9129	75-116, 142	0.01
Egg	fenbuconazole	84-138	0.01
	RH-9130	85-128	0.01
	RH-9129	83-124	0.01

Some additional recoveries by method a) were reported for cow and hen tissues. Fortification levels were 0.01 to 0.05 mg/kg. The results are shown in Table 25 (Stavinski, 1994a).

Table 25. Recoveries of fenbuconazole and the lactones from cow and hen tissues. Limit of determination 0.01 mg/kg for all samples.

Substrate	Analyte	Recovery, % Fortification level		
		0.01 mg/kg	0.02 mg/kg	0.05 mg/kg
Cow muscle	fenbuconazole	103-113	97-104	90-93
	RH-9130	106-131	96-102	92-96
	RH-9129	92-117	102-113	93-101
Cow liver	fenbuconazole	97-108	99-140	92-102
	RH-9130	92-108	83-101	93-100
	RH-9129	84-102	93-96	88-92
Cow kidney	fenbuconazole	83-100	82-110	81-94
	RH-9130	89-116	88-113	85-100
	RH-9129	93-138	90-114	98-101
Cow fat	fenbuconazole	31-99	50-92	58-90
	RH-9130	83-117	55-100	56-116
	RH-9129	56-114	65-94	62-116
Hen muscle	fenbuconazole	93-120	80-117	88-97
	RH-9130	112-155	87-119	90-100
	RH-9129	109-166	84-120	92-96
Hen liver	fenbuconazole	98-108	90-111	90-106
	RH-9130	103-137	93-139	98-105
	RH-9129	91-112	93-109	92-102
Hen fat	fenbuconazole	110-128	103-112	96-104
	RH-9130	110-131	105-114	100-110
	RH-9129	101-125	108-115	101-107

In a study by Sharma and Robinson (1996) samples of liver and fat from a goat metabolism study were analysed both by method a) and by TLC with radiometric detection. The results were well correlated indicating that method a) adequately extracts and quantifies the residues. The results are summarized in Table 26.

Table 26. Comparison of analyses of liver and fat by radio-TLC and analytical method a).

Analyte	Residues, mg/kg			
	Liver		Fat	
	Analyses by radio-TLC	Analyses by method a)[1]	Analyses by radio-TLC	Analyses by method a)[1]
Fenbuconazole	1.43	1.09	0.17	0.13
RH-7968	0.05	0.03	0.04	0.04
RH-9129	0.18	0.26	0.11	0.04
RH-9130	0.44	0.24	0.04	0.02

Analyte	Residues, mg/kg			
	Liver		Fat	
	Analyses by radio-TLC	Analyses by method a)[1]	Analyses by radio-TLC	Analyses by method a)[1]
RH-6468	0.12	quantified as RH-9129/RH-9130	not detected	Quantified as RH-9129/RH-9130
Sum of RH-9129, RH-9130, and RH-6468	0.74	0.50	0.16	0.05

[1]Means of triplicate analyses, except RH-7968

<u>Plant commodities</u>

Several minor variants of a basic method have been described for individual commodities. Most of the commodities in the supervised trials were analysed by a method validated for stone fruit or one for almonds.

b) This method was developed for the determination of fenbuconazole and the lactones RH-9129 and RH-9130 in stone fruit but was used for all the fruit and vegetables in the supervised trials except sugar beet. Fenbuconazole is extracted with methanol and partitioned with sodium chloride and dichloromethane. The dichloromethane is evaporated and the sample redissolved in toluene/acetone and cleaned up by silica gel and Florisil chromatography before analysis by GLC with NP detection. The linearity of response and sample chromatograms was acceptable. The limit of determination was 0.01 mg/kg. The recoveries are shown in Table 27 (Martin, 1998a, 1990, 1993a). A confirmatory method, with GLC on a different column, has been validated for stone fruit by Burnett (1991i).

Table 27. Recoveries of fenbuconazole and lactones from stone fruit.

Sample	Fortification, mg/kg	Recovery, %	No	Reference
Peaches/fenbuconazole	0.04	81	1	Martin, 1988a
	0.08	78	1	
	0.10	82, 114	2	
	0.20	75-101	4	
	0.40	62-116	4	
	0.65	115	1	
	0.80	94	1	
	1.0	95-103	3	
	1.6	94	1	
Cherries/fenbuconazole	0.10	110	1	
	0.20	84, 94	2	
	0.30	95-109	3	
	0.40	92	1	
	0.45	75	1	
	0.50	85, 92	2	
Plums/fenbuconazole	0.01	75-93	3	
	0.10	93-97	3	
	0.20	94	1	
	0.50	91	1	
Cherries/fenbuconazole	0.01	93, 100	2	Martin, 1990, 1993a
	0.04	91	1	
	0.08	95	1	

Sample	Fortification, mg/kg	Recovery, %	No	Reference
	0.10	87	1	
	0.40	84	1	
	2.0	90	1	
	3.0	78, 83	2	
	4.0	93	1	
Cherries/RH-9130	0.01	88, 99	2	
	0.04	90	1	
	0.08	90	1	
	0.10	85	1	
	0.40	93	1	
	2.0	95	1	
	3.0	81, 83	2	
	4.0	98	1	
Cherries/RH-9129	0.01	86, 94	2	
	0.04	77	1	
	0.08	88	1	
	0.10	74	1	
	0.40	90	1	
	2.0	87	1	
	3.0	60, 71	2	
	4.0	85	1	

c) This method has been validated for the determination of fenbuconazole and the lactones RH-9129 and RH-9130 in almonds, but also applied to fruits, vegetables, cereals and oilseed in the supervised trials. Almond hulls are Soxhlet-extracted with methanol, and kernels with toluene/methanol. The analysis is completed as above, except that clean-up on a C-18 column is included. Recoveries, linearity of response and sample chromatograms were acceptable. The limit of determination in almonds was 0.01 mg/kg for all the analytes. Recoveries are shown in Table 28 (Ross, 1996).

Table 28. Recoveries of fenbuconazole and lactones from almonds (Ross, 1996).

Sample/analyte	Fortification, mg/kg	Recovery, %	No.
Hull/fenbuconazole	0.01	86	1
Nut/fenbuconazole	0.01	82, 86	2
Hull/fenbuconazole	0.02	97, 101	2
Kernel/fenbuconazole	0.05	103	1
Hull/fenbuconazole	0.10	90-105	3
Kernel/fenbuconazole	0.10	94, 112	2
Nut/fenbuconazole	0.25	104	1
Hull/fenbuconazole	0.25	102, 107	2
Nut/fenbuconazole	0.5	99	1
Hull/fenbuconazole	0.5	96, 107	2
Kernel/fenbuconazole	1.0	103	1
Hull/fenbuconazole	1.0	72	1
Hull/RH-9129	0.01	125	1
Nut/ RH-9129	0.01	75	1
Hull/ RH-9129	0.02	97, 98	2

Sample/analyte	Fortification, mg/kg	Recovery, %	No.
Kernel/ RH-9129	0.05	103	1
Hull/ RH-9129	0.10	93-107	3
Kernel/ RH-9129	0.10	105, 115	2
Hull/ RH-9129	0.25	105, 110	2
Kernel/ RH-9129	0.5	97	1
Hull/ RH-9129	0.5	99, 108	2
Kernel/ RH-9129	1.0	107	1
Hull/ RH-9129	1.0	78	1
Hull/RH-9130	0.01	107	1
Nut/ RH-9130	0.01	77, 83	2
Hull/ RH-9130	0.02	76, 97	2
Kernel/ RH-9130	0.05	108	1
Hull/ RH-9130	0.10	97-102	3
Kernel/ RH-9130	0.10	93, 105	2
Nut/ RH-9130	0.25	105	1
Hull/ RH-9130	0.25	100, 109	2
Nut/ RH-9130	0.5	99	1
Hull/ RH-9130	0.5	93, 109	2
Kernel/ RH-9130	0.5	93	1
Kernel/ RH-9130	1.0	96	1
Hull/ RH-9130	1.0	72	1

The same method (Ross, 1996) has been validated for the determination of fenbuconazole, the lactones, and the ketone RH-6467 in wheat. It was also applied to sugar beet and oilseed in the supervised trials. The limit of determination for all the analytes was 0.05 mg/kg in straw and 0.01 mg/kg in grain. The recoveries are shown in Table 29. The identities of the analytes were confirmed by GLC on a different column (Burnett *et al.*, 1994a,b; Martin, 1991c).

Table 29. Recoveries of fenbuconazole, RH-9129, RH-9130 and RH-6467 from wheat (Burnett *et al.*, 1994a,b; Martin, 1991c).

Sample/analyte	Fortification, mg/kg	Recovery-%	No.	Reference
Grain/fenbuconazole	0.01	77-106	3	Burnett *et al.*, 1994a
	0.02	63-101	4	
	0.03	69-81	3	
	0.04	78	1	
	0.05	68-104	6	
	0.10	90-106	6	
Grain/RH-9130	0.01	92-101	3	
	0.02	68-110	4	
	0.03	74-83	3	
	0.04	81	1	
	0.05	70-106	6	
	0.10	92-109	6	
Grain/RH-9129	0.01	75-96	3	
	0.02	63-103	4	
	0.03	69-73	3	

Sample/analyte	Fortification, mg/kg	Recovery-%	No.	Reference
	0.04	72	1	
	0.05	66-106	6	
	0.10	80-108	6	
Grain/RH-6467	0.01	79-111	3	
	0.02	73-118	4	
	0.03	80-94	3	
	0.04	91	1	
	0.05	72-112	6	
	0.10	83-117	6	
Straw/fenbuconazole	0.05	80-91	6	
	0.10	66-109	3	
	0.20	70-93	4	
	0.50	82-109	7	
	1.0	79-99	4	
	2.0	89-113	5	
Straw/RH-9130	0.05	79-92	6	
	0.10	67-101	3	
	0.20	82-102	4	
	0.50	76-109	7	
	1.0	79-90	4	
	2.0	89-113	5	
Straw/RH-9129	0.05	79-96	6	
	0.10	69-100	3	
	0.20	85-95	4	
	0.50	77-123	7	
	1.0	75-92	4	
	2.0	93-114	5	
Straw/RH-6467	0.05	81-94	6	
	0.10	62-112	3	
	0.20	78-115	4	
	0.50	78-114	7	
	1.0	79-92	4	
	2.0	89-122	5	
Straw/fenbuconazole	1.0	78-100	4	Burnett *et al.*, 1994b
	5.0	88-99	3	
	10.0	80-108	6	
	15.0	79-114	6	
Straw/RH-9130	1.0	79-97	4	
	5.0	92-98	3	
	10.0	78-107	6	
	15.0	77-114	6	
Straw/RH-9129	1.0	81-100	4	
	5.0	79-93	3	
	10.0	74-109	6	
	15.0	81-114	6	
Straw/RH-6467	1.0	75-93	4	
	5.0	82-88	3	
	10.0	56-94	6	

Sample/analyte	Fortification, mg/kg	Recovery-%	No.	Reference
	15.0	60-106	6	

d) A method developed for the determination of the same compounds in pecans was identical except that samples were Soxhlet-extracted with hexane/2-propanol and concentrated to an oily residue before dissolution in toluene/acetone. The limit of determination was 0.01 mg/kg for all the analytes with the recoveries shown in Table 30 (Martin, 1991a,b; Wu, 1994). A confirmatory procedure, with GLC on a different column has been validated (Burnett, 1991i).

Table 30. Recoveries of fenbuconazole, the lactones and the ketone from pecans.

Compound	Fortification, mg/kg	Mean (±sd)	Recovery,-%	No.
fenbuconazole	0.01	85 (21)	49-104	7
	0.02	98 (8)	84-108	6
	0.04	92 (19)	70-122	8
RH-9130	0.01	91 (26)	55-123	7
	0.02	101 (14)	75-113	6
	0.04	91 (17)	67-114	8
RH-9129	0.01	89 (27)	51-122	7
	0.02	91 (18)	67-112	6
	0.04	79 (14)	64-99	8
RH-6467	0.01	100 (33)	84-153	7
	0.02	105 (25)	71-134	6
	0.04	93 (15)	61-111	8

e) The same method, but with GLC on a capillary column, has been used for the determination of fenbuconazole, the ketone and the lactones in wheat, and fenbuconazole and the lactones in apples. The limit of determination was 0.01 mg/kg for wheat grain and 0.05 mg/kg for straw and apples. Acceptable chromatograms were submitted. The recoveries are shown in Table 31 (Burnett *et al.*, 1992a,b; Martin, 1989).

Table 31. Recoveries of fenbuconazole and metabolites from wheat and apples.

Sample	Fortification, mg/kg	Recovery, %		
		fenbuconazole	ketone	lactones
Wheat grain	0.02-1.0	63-111 mean 91	72-118	63-110 mean 89
Straw	0.05-5.0	67-118 mean 94	62-122 mean 91	54-123 mean 89
Apples	0.1-1.0	71-114	-	65-109 mean 84

f) A somewhat simpler method has been used for the determination of fenbuconazole and the lactones RH-9129 and RH-9130 in wheat and grapes. Samples were extracted with acetone/petroleum ether or hexane, cleaned up on a Florisil column, and analysed by GLC with an FID. The limit of determination was stated to be 0.01 mg/kg. Recoveries from wheat grain and grapes fortified at 0.25 mg/kg were 92 ± 8%. Recoveries from grapes fortified with fenbuconazole at 0.1 to 0.25 and 1 mg/kg and “its lactone” at 0.2 to 0.4 and 2 mg/kg were about 100 ± 10%. A satisfactory sample chromatogram was submitted (Mestres, 1989).

g) A preliminary method for the determination of RH-7905, the glucose conjugate of the phenol RH-4911, in wheat grain, bananas and apples has been described. The conjugate was hydrolysed to its phenol with acid in methanol and the hydrolysate partitioned with sodium chloride solution and dichloromethane. The dichloromethane was evaporated and the residue redissolved in acetonitrile before LC-18 SPE clean-up and analysis by HPLC-MS. Satisfactory sample chromatograms were provided. The average recoveries were 87% (sd ± 2.5) from wheat grain, 84% (sd ± 1.9) from apples, and 90% (sd ± 7.3) from bananas. Only summary information was provided on recoveries; the report states that the limit of determination will be determined in further experiments (Staurowsky and Novak, 1996).

The methods of analysis used in the residue trials on the individual commodities are listed in Table 32.

Table 32. Summary of the methods of analysis used in supervised trials (Ross, 1997a).

Commodity	Method		
	b)	d)	c)
Citrus fruits			
Grapefruit	yes		
Orange	yes		
Pome fruits			
Apple	yes		yes
Loquat	yes		
Pear	yes		yes
Stone fruits			
Apricot	yes		
Cherries	yes		
Nectarine	yes		
Peach	yes		
Plum / prune	yes		yes
Berries			
Grape	yes		yes
Strawberry	yes		yes
Fruit, inedible peel			
Banana	yes		
Mango	yes		
Fruiting veg., Cucurbits			
Melons not watermelon	yes		yes
Cucumber	yes		yes
Squash, summer	yes		
Fruiting veg., other than cucurbit			
Peppers	yes		yes
Tomato	yes		yes
Root and tuber vegetables			
Sugar beet			yes
Cereal grains			
Barley			yes

	Method		
Maize (corn)			yes
Rye			yes
Wheat			yes
Tree nuts			
Almond			yes
Pecan		yes	
Oilseeds			
Rape seed			yes
Sunflower seed			yes

Stability of pesticide residues in stored analytical samples

Apples, wheat grain and straw. Samples were fortified at 0.5 mg/kg with fenbuconazole and the lactone metabolites, and wheat grain and straw also with the ketone, and stored in a freezer at -10^{o}C for 36 months. Samples taken at intervals were analysed by method c). Uncorrected recoveries are shown in Tables 33-35. They show that all the analytes were stable for 36 months in wheat and apples. Procedural recoveries generally varied between 60 and 120% being somewhat higher for apples (Batra, 1995c).

Table 33. Stability of residues in fortified wheat grain stored at -10°C.

Storage period, months	Recoveries, %		
	Fenbuconazole	Lactone	Ketone
0	74.9	73.5	76.7
3	66.3	66.4	82.9
6	80.0	77.9	81.1
12	74.9	69.0	86.4
18	79.0	81.1	100
24	94.9	92.4	90.7
30	98.3	98.1	119
36	90.4	86.2	90.3

Table 34. Stability of residues in fortified wheat straw stored at -10°C.

Storage period, months	Recoveries, %		
	Fenbuconazole	Lactone	Ketone
0	70.3	61.8	71.0
3	93.0	94.6	100
6	83.0	87.4	97.8
12	80.7	86.6	96.8
18	103	101	113
24	79.9	77.1	71.8
30	80.0	79.1	94.7
36	93.7	95.6	98.7

Table 35. Stability of residues in fortified apples stored at -10°C.

Storage period, months	Recoveries, %	
	Fenbuconazole	Lactone
0	110	111
3	94.1	93.6
6	81.7	80.4
12	79.1	74.8
14	94.2	87.9
18	84.7	78.2
24	86.7	82.7
30	78.0	73.1
36	102	96.0

Another study on the stability of residues in frozen wheat grain, bran and shorts/germ showed that residues of fenbuconazole were stable in these substrates for up to 29 months (Burnett, 1992g).

Wheat grain, bran, middlings, shorts, red dog, low grade flour, patent flour and bread were analysed by, the method of analysis of Burnett (1994a) before and after frozen storage for approximately 4.6 years. Residues did not decrease significantly in cleaned grain, bran, shorts/germ, and red dog, but losses were substantial in middlings, flour and bread. Recoveries of 70 to 125% were reported (Burnett, 1994c).

Table 36. Stability of total residues of fenbuconazole and its lactone and ketone metabolites in processed fractions of wheat stored at -10°C for approximately 4.6 years.

Sample	Residues, mg/kg	
	Before storage	After storage
Cleaned grain	0.094	0.077 (-18%)
Bran	0.536	0.611 (+14%)
Middlings	0.108	0.074 (-31%)
Shorts/germ	0.270	0.262 (-3%)
Red dog	0.125	0.112 (-10%)
Flour (low grade)	0.066	0.042 (-36%)
Patent flour	0.104	0.045 (-57%)
Bread	0.110	0.072 (-34%)

Stone fruit. Peaches were fortified with fenbuconazole and its lactone metabolites at 0.5 mg/kg and stored in a freezer at -10^{o}C for 54 months. Samples taken at intervals were analysed by the method of Martin (1990). Uncorrected recoveries are recorded in Table 37 and show that fenbuconazole and the lactones were stable for 54 months. Procedural recoveries of 65-120% were generally acceptable. CLICK HERE to continue

Table 37. Stability of residues in fortified peaches stored at -10°C.

Storage Period, months	Recoveries, %	
	Fenbuconazole	Lactone
0	110	110
3	82.2	81.6
6	84.3	86.3
7	94.7	91.4
12	81.0	74.3
18	93.6	89.6
24	83.2	79.1
30	81.1	74.2
36	86.7	84.4
42	99.5	94.0
48	97.1	90.6
54	99.3	94.2

(Batra, 1994a; 1996b)

Tree nuts. Pecans fortified at 0.5 mg/kg with fenbuconazole and its ketone and lactone metabolites were stored in a freezer at -10°C. Duplicate samples taken after 0, 3, 6, 12, 18, 24, 30, 36, 42, 48 and 54 months were analysed by the method of Martin (1991b). A control and two freshly fortified samples were, two samples each of the processed pecans were analysed for fenbuconazole and metabolites by the analytical methods for parent and metabolites in pecans (Martin, 1991b). A control and two freshly fortified samples were run concurrently at each sampling interval to validate the analytical procedure. Uncorrected recoveries are shown in Table 38. Procedural recoveries were 75 -115% (Batra, 1996c).

Table 38. Stability of residues in fortified pecans stored at -10°C.

Storage period, months	Recoveries, %		
	Fenbuconazole	Lactone	Ketone
0	78.4	74.1	84.8
3	79.9	72.3	78.2
6	72.9	70.2	62.7
12	69.1	61.3	77.8
18	108	94.2	110
24	84.3	74.2	76.7
30	102	85.8	122
36	87.4	82.8	81.3
42	97.3	83.3	93.0
48	72.5	76.7	93.4
54	99.7	93.0	95.1

In a similar study on almonds, kernels were fortified with either fenbuconazole or the lactones at 1 mg/kg. Duplicate samples taken after 0. 7, 14, 28, 60 and 90 days, 6 months and 1 year were analysed for fenbuconazole and the metabolites by the method of Martin (1989). Positive and negative control samples were run as before. Uncorrected recoveries are shown in Table 39. Procedural recoveries were 75-125%.

Table 39. Stability of residues in fortified almond kernels stored at -10°C.

Storage period, days	Recoveries, %	
	Fenbuconazole	Lactone
0	80	100
6	79	90
13	73	75
27	82	79
60	73	80
91	-	85
181	76	77
c.365	90	98

Samples from residue trials on almonds were reanalysed by the method of Martin (1989) after 1035 days frozen storage. There was no significant difference between the fenbuconazole or lactone residues found in almond hulls in the two sets of analyses. Comparison of the residues in the kernels was inconclusive because the residues were close to the limit of quantification of 0.01 mg/kg (Burnett, 1992c).

Products of animal origin. Samples of milk, eggs, muscle, fat, kidneys and liver fortified at unspecified levels with fenbuconazole and the lactones and RH-7968 were stored in a freezer below -4°C for periods up to 4 months. Samples taken at intervals were analysed by the method of Filchner *et al*. (1994a). Uncorrected recoveries are shown in Table 40. The analytes all showed acceptable stability for the tested periods (Chen *et al*., 1992b).

Table 40. Stability of residues in fortified animal products stored at below -4°C.

Sample	Storage period, days	Recovery, %		
		Fenbuconazole	Lactone	RH-7968
Milk	0	83-87	83-92	-
	118	86-86	89-95	-
Eggs	0	92-96	93-97	-
	74	101-119	104-125	-
Cattle muscle	0	82-84	79-85	-
	61	80-89	80-90	-
	82	86-97	90-104	-
Chicken muscle	0	91-92	92-93	-
	49	92	89-94	-
	69	90-93	94-103	-
Cattle fat	0	69-90	66-95	-
	69	68-71	69-73	-
Chicken fat	0	76-84	76-85	110-112
	48	80-82	81-84	115-120
Cattle kidney	0	100-101	99-104	75-90
	74/76	100-106	103-111	108-113
Cattle liver	0	87-94	88-96	77
	63	73-82	72-85	85-92
Chicken liver	0	90-92	92-93	82-86
	63	73	82-84	77-110

Samples of hen and cow muscles fortified with fenbuconazole and its lactone metabolites at 0.2 mg/kg were stored at -10°C and analysed by the method of Filchner *et al.* (1992) after 2.6 years. Procedural recoveries were determined concurrently and were acceptable at 83-89%. The stored samples were found to contain 88% of the original fenbuconazole and 90-101% of the lactones (Filchner and Negro, 1994).

USE PATTERN

Fenbuconazole is commonly applied as a foliar treatment and is currently registered for use on a range of fruits, vegetables, cereals and oilseeds, as shown in Tables 41-43.

Registrations which were reported by the company as pending are shaded. Where both the application rate in kg ai/ha and the spray concentration in kg ai/hl are given, the rate considered by the company to be of prime importance is shown bold. Product labels were supplied comprehensively only for France, Portugal and the UK (Costlow, 1997a).

There is no GAP in The Netherlands (Olthof, 1997).

Table 41. Registered uses of fenbuconazole on tree nuts and fruit.

Commodity	Country	Form	F/G	Application				PHI, days	Reference Remarks
				Spray vol., l/ha	Rate, kg ai/ha	Spray conc., kg ai/hl	No.		
TREE NUTS									
Almonds	Israel	EC		≤1500	≤ 0.06	0.004	4-6	14	Orpin & Costlow 1997
	USA	WP SC		≥93.5 ≤3.38	**0.105** **0.105**		≤3	160	Orpin & Costlow 1997
Pecans	USA	SC		≥93.5 ≤3740	**0.140** **0.140**		≤6	28	Orpin & Costlow 1997
FRUIT									
Apples	France	EW	F	500-1500	≤0.0525	**0.0035**	4-10	28	Orpin & Costlow 1997; Orpin 1997b
	Greece	EC	F	≤2000	≤0.060	**0.003**	6	28	Orpin & Costlow 1997; Orpin 1997b
	Israel	EC	F	≤2000	≤0.040	**0.002**	6	14	Orpin & Costlow 1997
	Italy	EC and EW	F	≤2000	≤0.060	**0.003**	6	28	Orpin & Costlow 1997
	Portugal	EW	F	≤1000	≤0.004	**0.003-0.004**	4-6	28	Orpin & Costlow 1997; Orpin 1997b
	South Africa	EW	F	≤2000	≤0.050	**0.0025**	4-10	28	Orpin & Costlow 1997
	Turkey	EC	F	≤2000	≤0.040	**0.002**	4	14	Orpin & Costlow 1997
	UK (Company submission)	EW	F	≤2000 200 min	**0.068**		≤10	28	Orpin & Costlow 1997
	UK (Country submission)	EW	F	500-1000	0.035-0.07	0.007	-	28	UK, 1997 Overall spray Max total dose 7 kg ai/ha/year.
	USA	WP		≥93.5 ≤ 3740	**0.140** **0.140**		≤9	14	Orpin & Costlow 1997 Formulation in water sol. pouches

Commodity	Country	Form	F/G	Application				PHI, days	Reference Remarks
				Spray vol., l/ha	Rate, kg ai/ha	Spray conc., kg ai/hl	No.		
Apricots	France	EC	F	1000 1000	0.05 0.075	**0.005** **0.0075**	1 1-2	3 3	Orpin & Costlow 1997; Orpin 1997b
	Israel	EC	F	≤1500	≤0.037	**0.0025**	4-6	14	Orpin & Costlow 1997
	USA	WP		≥93.5 ≤2337.6	**0.105** **0.105**		≤8	0	Orpin & Costlow 1997 Foundation in water sol. pouches
Bananas	Columbia Costa Rica Ecuador Guatemala Honduras Mexico Panama Venezuela	OS SC		≥93.5 ≤1000	**0.105** **0.105**		≤8	0	Orpin & Costlow 1997
	Philippines USA	SC		≥93.5 ≤1000	**0.105** **0.105**		≤8	0	Orpin & Costlow 1997
Cherries	USA	WP		≥93.5 ≤2337.6	**0.105** **0.105**		6	0	Orpin & Costlow 1997
Grapefruit	USA	SC		≥93.5 ≤3740	**0.280**		≤3	0	Orpin & Costlow 1997
Grapes, Table and Wine	France Indar Duo	EC	F	150-1000[1]	**0.030**	-	4-6	21	Orpin & Costlow 1997
	France Indar 5EC	EC	F	150-1000[1]	**0.0375**	-	4-6	21	Orpin & Costlow 1997
	Greece 'Indar 5EC'	EC	F	1000-1500	≤0.045	**0.003**	5	14	Orpin & Costlow 1997; Orpin 1997b
	Greece 'Rogano' (Indar Duo)	EC	F	1000-1500	≤0.045	**0.003**	5	14	Orpin & Costlow 1997; Orpin 1997b
	Israel	EC	F	≤1000 ≤1000	≤0.075 ≤0.02	**0.0075** **0.002**	3-5	7	Orpin & Costlow 1997
	Italy	EC	F	1000-1500	≤0.045	**0.003**	5	14	Orpin & Costlow 1997
	Italy Indar 5EW	EW	F	1000-1500	≤0.045	**0.003**	5	14	Orpin & Costlow 1997
	Italy Indar 3EW	EW	F	1000-1500	≤0.045	**0.003**	5	14	Orpin & Costlow 1997
	Portugal Indar 5EC	EC	F	500	**0.040**	-	3	28	Orpin & Costlow 1997
	Portugal Karamat	EC	F	500	**0.040**	-	3	28	Orpin & Costlow 1997
	Spain	EC	F	600	**0.040**	-	2-4	21	Orpin & Costlow 1997
	Turkey	EC	F	≤1500	≤0.030	**0.002**	5	14	Orpin & Costlow 1997
Loquats	Israel	EC	F	≤2000	≤0.03	**0.0015**	4-6	14	Orpin & Costlow 1997
Mangoes	Israel	EC		≤ 2000	<0.040	0.002	4-6	14	Orpin & Costlow 1997
Nectarines	France	EC	F	1000	0.05	**0.005**	1-2	60	Orpin & Costlow 1997; Orpin 1997b

Commodity	Country	Form	F/G	Application				PHI, days	Reference Remarks
				Spray vol., l/ha	Rate, kg ai/ha	Spray conc., kg ai/hl	No.		
	Israel	EC	F	≤1500	≤0.030	**0.002**	4-6	14	Orpin & Costlow 1997
	South Africa	EW	F	≤1500	≤0.0375	**0.0025**	4-6	7	Orpin & Costlow 1997; Orpin 1997b
	USA	WP		≥93.5 ≤2337.6	**0.105** **0.105**		≤8	0	Orpin & Costlow 1997 formulation in water sol. pouches
Oranges	USA	SC		≥93.5 ≤3740	**0.280** **0.280**		Up to 3	0	Orpin & Costlow 1997
Peaches	France	EC	F	1000	0.05	**0.005**	1-2	3[2]	Orpin & Costlow 1997; Orpin 1997b
	Israel	EC	F	≤1500	≤0.030	**0.002**	4-6	14	Orpin & Costlow 1997
	South Africa	EW	F	≤1500	≤0.0375	**0.0025**	4-6	7	Orpin & Costlow 1997; Orpin 1997b
	USA	WP		≥93.5 ≤2337.6	**0.105** **0.105**		≤8	0	Orpin & Costlow 1997 formulation in water sol. pouches
Pears	France	EW	F	500-1500	≤0.0525	**0.0035**	4-10	28	Orpin & Costlow 1997; Orpin 1997b
	Greece	EC	F	≤2000	≤0.060	**0.003**	6	28	Orpin & Costlow 1997
	Israel	EC	F	≤2000	≤0.040	**0.002**	6	14	Orpin & Costlow 1997
	Italy	EC & EW	F	≤2000	≤0.050	**0.0025**	6	28	Orpin & Costlow 1997
	Portugal	EW	F	≤1000	≤0.040	**0.003-0.004**	4-6	28	Orpin & Costlow 1997; Orpin 1997b
	South Africa	EW	F	≤2000	≤0.050	**0.0025**	6	28	Orpin & Costlow 1997
	UK (Company submission)	EC	F	≤2000 200 min	**0.068**		≤10	28	Orpin & Costlow 1997
	UK (Country submission)	EW	F	500-1000	**0.035-0.07**		-	28	U.K., 1997 Overall spray Max total dose 0.7 kg ai/ha/year.
Plums/ Prunes, fresh	France	EC	F	1000 1000 1000	0.05 0.05 0.075	**0.005** **0.005** **0.0075**	1-2 1 1-2	90 3 3	Orpin & Costlow 1997; Orpin 1997b
	Israel	EC	F	≤1500	≤0.030	**0.002**	4-6	14	Orpin & Costlow 1997
	USA	WP		≥93.5 ≤2337.6	**0.105** **0.105**		≤8	0	Orpin & Costlow 1997
Strawberries	Israel	EC	F/G	1000	0.075	0.0075	4-6	14	Orpin & Costlow 1997

[1]Originally reported by company as 50-400l/ha
[2]Originally reported by company as 60 days

Table 42. Registered uses of fenbuconazole on vegetables.

Commodity	Country	Form	F/G	Application				PHI, Days	Reference Remarks
				Spray vol., l/ha	Rate, kg ai/ha	Spray conc., kg ai/hl	No.		
Cucumber	France	EC 'Indar 5EC'	F/G	≤1000	**0.1**		4	3	Orpin & Costlow 1997; Orpin 1997b
	France	EC 'Indar Duo'	F/G	≤1000	**0.06**		4	3	Orpin & Costlow 1997; Orpin 1997b
	Israel		F	500	**0.025-0.0375**	-	3-5	7	Orpin & Costlow 1997
	Israel		G	≤1000	-	**0.0075**	3-5	7	Orpin & Costlow 1997
	Morocco		F/G	1000-2000	0.1-0.2	**0.010**	1-4	3	Orpin & Costlow 1997; Orpin 1997b
	Spain		F/G	≤1000	≤0.060	**0.006**	1-3	7	Orpin & Costlow 1997
	Turkey		F/G	≤1000	≤0.050	**0.005**	2-4	3	Orpin & Costlow 1997
Egg plant	Morocco			1000-2000		0.10	6	3	Ross 1997
Gherkin	France Indar 5EC	EC	F/G	<1000	**0.1**		4	3	Orpin & Costlow 1997; Orpin 1997b
	France Indar Duo	EC	F/G	≤1000	**0.06**		4	3	Orpin & Costlow 1997; Orpin 1997b
	Israel		F	500	**0.025-0.0375**	-	3-5	7	Orpin & Costlow 1997
	Israel		G	≤1000	-	**0.0075**	3-5	7	Orpin & Costlow 1997
	Morocco		F/G	1000-2000	0.1-0.2	**0.010**	1-4	3	Orpin & Costlow 1997
	Spain		F/G	≤1000	≤0.060	**0.006**	1-3	7	Orpin & Costlow 1997
	Turkey		F/G	≤1000	≤0.050	**0.005**	2-4	3	Orpin & Costlow 1997
Melons	France Indar 5EC	EC	F/G	≤1000	**0.1**		4	7[1]	Orpin & Costlow 1997; Orpin 1997b
	France Indar Duo	EC	F/G	≤1000	**0.06**		4	7[1]	Orpin & Costlow 1997; Orpin 1997b
	Israel	EC	F/G	500	**0.025-0.0375**		3-5	7	Orpin & Costlow 1997
	Italy	EC	F/G	≤1000	0.050	**0.005**	2-4	7	Orpin & Costlow 1997
	Italy Indar 5EW	EW	F/G	≤1000	0.050	**0.005**	2-4	7	Orpin & Costlow 1997
	Italy Indar 3EW	EW	F/G	≤1000	0.050	**0.005**	2-4	7	Orpin & Costlow 1997
	Morocco	EC	F/G	1000-2000	0.1-0.2	**0.010**	1 - 6	3	Orpin & Costlow 1997; Orpin 1997b
	Portugal	EC	F/G	≤1000	0.050-0.060	**0.005-0.006**	1-4	7	Orpin & Costlow 1997

Commodity	Country	Form	F/G	Application: Spray vol., l/ha	Application: Rate, kg ai/ha	Application: Spray conc., kg ai/hl	Application: No.	PHI, Days	Reference Remarks
	Spain	EC	F/G	≤1000	0.060	**0.006**	1-3	7	Orpin & Costlow 1997; Orpin 1997b
	Turkey	EC	F/G	≤1000	≤0.050	**0.005**	2-4	3	Orpin & Costlow 1997
Peppers	Israel	EC	F	1000 min	0.075 min	**0.0075**	3-5	7	Orpin & Costlow 1997
	Morocco	EC	G/F	1000-2000	0.1-0.2	**0.01**	3-6	3	Orpin & Costlow 1997; Orpin 1997b
Sugar beet	USA	WP		≥47 ≤187	**0.14** **0.14**		≤ 8	14	Orpin & Costlow 1997
	Italy	EC	F	500	**0.100**		2	14	Orpin & Costlow 1997
Summer squash (zucchini, courgette)	France	EC	F/G	≤1000	**0.06**		4	3	Orpin & Costlow 1997 Orpin 1997c
	Israel		F	500	**0.025-0.0375**	-	3-5	7	Orpin & Costlow 1997
	Israel		G	≤1000	-	**0.0075**	3-5	7	Orpin & Costlow 1997
	Morocco		F/G	1000-2000	0.1-0.2	**0.010**	1-4	3	Orpin & Costlow 1997 Orpin 1997c
	Spain		F/G	≤1000	≤0.060	**0.006**	1-3	7	Orpin & Costlow 1997
	Turkey		F/G	≤1000	≤0.050	**0.005**	2-4	3	Orpin & Costlow 1997
Tomatoes	Israel	EC	G F	1000 min 300-500	0.075 min **0.050**	**0.0075** -	3-5 3-5	7 7	Orpin & Costlow 1997
	Morocco	EC	G/F	1000-2000	0.1-0.2	**0.01**	3-6	3	Orpin & Costlow 1997; Orpin 1997b
Watermelons	Israel	EC	F/G	500	**0.025-0.0375**	-	3-5	7	Orpin & Costlow 1997
	Italy	EC	F/G	≤1000	0.050	**0.005**	2-4	7	Orpin & Costlow 1997
	Italy Indar 5EW	EW	F/G	≤1000	0.050	**0.005**	2-4	7	Orpin & Costlow 1997
	Italy Indar 3EW	EW	F/G	≤1000	0.050	**0.005**	2-4	7	Orpin & Costlow 1997
	Morocco	EC	F/G	1000-2000	0.1-0.2	**0.010**	1 - 6	3	Orpin & Costlow 1997; Orpin 1997b
	Portugal	EC	F/G	≤1000	0.050-0.060	**0.005-0.006**	1-4	7	Orpin & Costlow 1997
	Spain	EC	F/G	≤1000	0.060	**0.006**	1-3	7	Orpin & Costlow 1997
	Turkey	EC	F/G	≤1000	≤0.050	**0.005**	2-4	3	Orpin & Costlow 1997

[1]PHI reported as 3 days in original company submission

Table 43. Registered uses of fenbuconazole on cereals and oilseeds.

Commodity	Country	Form	F/G	Application				PHI, days	Reference Remarks
				Spray vol., l/ha	Rate, kg ai/ha	Spray conc., kg ai/hl	No		
CEREALS									
Barley	France	SE	F	200	**0.072**	-	2	45	Orpin & Costlow 1997
	Germany (company submission)	EC	F	200-400	**0.075**	-	2	35	Orpin & Costlow 1997
	Germany (country submission)	EC	F	200-400	**0.075**		2	35	Germany, 1996
	South Africa	EC	F	200-400	**0.125**	-	1	35	Orpin & Costlow 1997 Amendment of registration to a two-spray programme pending.
	UK (Company submission)	EC	F	200	**0.075**	-	2	-	Orpin & Costlow 1997
	UK (Country submission)	EC	F	200	**0.075**		2	-	U.K., 1997 Overall spray Before beginning of flowering GS 59
	UK (Country submission)	EC	F	200-400	**0.075**		2	-	U.K., 1997 Overall spray Before beginning of flowering GS 59
Maize	France	SE	F	200-400	0.075		2	45	Orpin & Costlow 1997
Rye	Germany (company submission)	EC	F	200-400	0.075		2	35	Orpin & Costlow 1997
	Germany (country submission)	EC	F	200-400	**0.075**		2	35	Germany, 1996
Wheat	Belgium	EC/SC		200	**0.075**		2	42	Orpin & Costlow 1997
	France	SE		200	**0.080**		2	45	Orpin & Costlow 1997
	France	EC		200	**0.075**	-	2	45	Orpin & Costlow 1997
	Germany (company submission)	EC	F	200-400	**0.075**		2	35	Orpin & Costlow 1997
	Germany (country submission)	EC	F	200-400	**0.075**		2	35	Germany, 1996
	Israel	EC		40-200	**0.075**	-	2	90	Orpin & Costlow 1997
	Morocco	SE		200-400	**0.075**	-	1	N/S	Orpin & Costlow 1997
	Portugal	EC		500	**0.075**	-	2	42	Orpin & Costlow 1997
	South Africa	EC		500	**0.125**	-	1	42	Orpin & Costlow 1997 Amendment of registration to a two-spray programme pending.

Commodity	Country	Form	F/G	Application				PHI, days	Reference Remarks
				Spray vol., l/ha	Rate, kg ai/ha	Spray conc., kg ai/hl	No		
	UK (Company submission)	EC		200	**0.075**	-	2	-	Orpin & Costlow 1997
	UK (Country submission)	EC	F	200	**0.075**		2	-	U.K., 1997 Overall spray to winter sown wheat only. Before beginning of flowering GS 59
	UK (Country submission)	EC	F	200-400	**0.075**		2	-	U.K., 1997 Overall spray to winter sown wheat only. Before beginning of flowering GS 59
	USA	WP		≥47 ≤187	**0.070** **0.070**		≤ 3	35	Orpin & Costlow 1997 Seed treatment also pending
OILSEEDS									
Groundnut (Peanut)	Israel	EC		200-500	**0.05**		2 -3	14	Orpin & Costlow 1997
Oilseed rape	France Indar Mega/Troika	SE	F	200	**0.060**		1-2	30	Orpin & Costlow 1997
	France Polka	SE	F	200	**0.075**		1	45	Orpin & Costlow 1997
Sunflower	France Indar Mega/Troika	SE	F	200-400	**0.060**		2	80	Orpin & Costlow 1997; Orpin 1997b
	France Polka	SE	F	200-400	**0.075**		2	80	Orpin & Costlow 1997; Orpin 1997b

N/S: Not stated

RESIDUES RESULTING FROM SUPERVISED TRIALS

The results of the residue trials are shown in Tables 44-72. Unless otherwise indicated trials were carried out under field conditions, were reported in sufficient detail, and acceptable analytical information was supplied. Residues from trials before 1993 were generally corrected for analytical recoveries, but those from trials during and after 1993 generally not. Recoveries in most trials were acceptable, but where they were outside the range 70-120% and/or where samples were stored for longer than 6 months this is indicated. In a few trials details of the method of analysis were not given. Trials which the Meeting considered unsatisfactory have been identified by shading in the Tables. Trials have been regarded as according to GAP when the maximum conditions of GAP (minimum PHI, maximum dose rate and maximum number of treatments) have been applied.

The manufacturers explained their interpretation of the trials on tree fruit as follows.

“For USA tree crops application has not historically been to runoff, but rather to *adequate coverage*. Since there is no firmly fixed end point for application, and since water volume varies according to the size and leaf coverage of the trees, the amount of pesticide delivered to each tree may vary from one trial to another, but the amount of pesticide per hectare will be held constant. For U.S. residue trials, the kg ai/ha value is the controlling factor. In contrast, outside of the USA the concentration of the spray solution (kg ai/hl) is usually fixed for tree fruit and the spray volume is fixed to an endpoint such as runoff (assuming a constant tree size). In this case, the amount of pesticide per hectare will vary depending on the density of planting and size of the trees and for these residue trials the kg ai/hl value is the controlling factor."

Grapefruit, oranges. Only pending GAP in the USA was reported. The maximum application rate is 0.28 kg ai/ha with a PHI of 0 days.

The residues in trials considered to comply with the pending US GAP are underlined in Tables 44 and 45. In all the trials the samples were stored for more than 6 months before analysis.

Table 44. Supervised residue trials on grapefruit, USA.

State, Year	Application				PHI, days	Sample	Residue, mg/kg				Ref.
	Form.	No.	kg ai/ha	kg ai/hl			Fenbuc-onazole	RH-9129	RH-9130	RH-6467	
FL, 1993	SC	3	**0.28**	0.0323	0	whole fruit	0.487	≤0.01	≤0.01	≤0.01	Batra, 1993a
					15	whole fruit	0.318	<0.01	<0.01	<0.01	
					26	whole fruit	0.319	<0.01	<0.01	<0.01	
					59	whole fruit	0.126	<0.01	<0.01	<0.01	
	-	-		control	-	whole fruit	0.03	<0.01	<0.01	<0.01	
CA, 1993	SC	3	**0.28**	0.0291	0	whole fruit	0.342	≤0.01	≤0.01	≤0.01	Batra, 1993a
TX, 1993	SC	3	**0.28**	0.0118	0	whole fruit	0.19	≤0.01	≤0.01	≤0.01	Batra, 1993a
TX, 1993	SC	3	**0.28**	0.029	0	whole fruit	0.162	≤0.01	≤0.01	---	Batra, 1996d
						pulp	≤0.01	≤0.01	≤0.01	---	
TX, 1993	SC	3	**0.28**	0.03	0	whole fruit	0.123	≤0.01	≤0.01	---	Batra, 1996d
						pulp	≤0.01	≤0.01	≤0.01	---	
CA, 1994	SC	3	**0.28**	0.015	0	whole fruit	0.134	≤0.01	≤0.01	---	Batra, 1996d
CA, 1994	SC	3	**0.28**	0.015	0	pulp	0.0199	≤0.01	≤0.01	---	
FL, 1994	SC	3	**0.28**	0.013	0	whole fruit	0.0976	≤0.01	≤0.01	---	Batra, 1996d
						pulp	≤0.01	≤0.01	≤0.01	---	
FL, 1994	SC	3	**0.28**	0.0236	0	whole fruit	0.155	≤0.01	≤0.01	---	Batra, 1996d
						pulp	0.464	≤0.01	≤0.01	---	
FL, 1994	SC	3	**0.28**	0.0275	0	whole fruit	0.157	≤0.01	≤0.01	---	Batra, 1996d
						pulp	≤0.01	≤0.01	≤0.01	---	

Table 45. Supervised residue trials on oranges, USA.

Country, year	Application				PHI, days	Sample	Residue, mg/kg			P	Ref.
	Form.	No.	kg ai/ha	kg ai/hl			Fenbuconazole	RH-9129	RH-9130	RH-6467	
CA, 1993	SC	3	**0.28**	0.0286	0	whole fruit	0.339	≤0.01	≤0.01	≤0.01	Batra, 1993a
FL, 1993	SC	3	**0.28**	0.0323	0	whole fruit	0.442	≤0.01	≤0.01	≤0.01	Batra, 1993a
					15	whole fruit	0.259	<0.01	<0.01	<0.01	
					26	whole fruit	0.348	<0.01	<0.01	<0.01	
					59	whole fruit	0.184	<0.01	<0.01	<0.01	
TX, 1993	SC	3	**0.28**	0.012	0	whole fruit	0.518	0.0101	≤0.01	≤0.01	Batra, 1993a
					15	whole fruit	0.303	0.0105	<0.01	<0.01	
					30	whole fruit	0.45	0.012	<0.01	<0.01	
					60	whole fruit	0.272	0.0101	<0.01	<0.01	
TX, 1993	SC	3	**0.28**	0.029	0	whole fruit	0.178	≤0.01	≤0.01	---	Batra, 1996d
						pulp	≤0.01	≤0.01	≤0.01	---	
TX, 1993	SC	3	**0.28**	0.03	0	whole fruit	0.176	≤0.01	≤0.01	---	Batra, 1996d
						pulp	≤0.01	≤0.01	≤0.01	---	
CA, 1994	SC	3	**0.28**	0.015	0	whole fruit	0.304	0.0374	0.0682	---	Batra, 1996d
						pulp	≤0.01	≤0.01	≤0.01	---	
FL, 1994	SC	3	**0.28**	0.0196	0	whole fruit	0.19	≤0.01	≤0.01	---	Batra, 1996d
						pulp	≤0.01	≤0.01	≤0.01	---	
FL, 1994	SC	3	**0.28**	0.0275	0	whole fruit	0.279	≤0.01	≤0.01	---	Batra, 1996d
						pulp	0.0104	≤0.01	≤0.01	---	
FL, 1994	SC	3	**0.28**	0.0172	0	whole fruit	0.187	≤0.01	≤0.01	---	Batra, 1996d

Apples. GAP was reported for France, Israel, Italy, Portugal, South Africa, Turkey and the UK, and pending GAP for the USA and Greece. The maximum applications are defined by concentrations of 002-0004kg ai/hl except in the UK and USA where the application rates were reported as 0.068 and 0.140 kg ai/ha respectively. The PHIs are either 14 or 28 days.

The residues in trials considered to comply with the pending US GAP, UK GAP and Southern European GAP (France, Greece, Italy and Portugal) are underlined, double underlined and set in bold italics, respectively, in Table 46.

Table 46. Supervised residue trials on apples.

Location	Application				PHI	Sample	Residue, mg/kg			Reference/
Year, (notes)	Form.	No.	kg ai/ha	kg ai/hl	days		Fenbuconazole	RH-9129	RH-9130	Comment
MI/USA 1990[1]	SC	10	**0.14**	0.0106	0	fruit	0.244	0.010	<0.01	Burnett, 1992f
					7	fruit	0.126	0.011	<0.01	
					13	fruit	0.059	≤0.01	≤0.01	
MI/USA 1990[1]	SC	10	**0.28**	0.0211	13	fruit	0.142	0.011	<0.01	Burnett, 1992f

Location	Application				PHI	Sample	Residue, mg/kg			Reference/
Year, (notes)	Form.	No.	kg ai/ha	kg ai/hl	days		Fenbuc-onazole	RH-9129	RH-9130	Comment
NC/USA 1990[1]	SC	8	**0.14**	0.016	0	fruit	0.018	<0.01	<0.01	Burnett, 1992f
					7	fruit	0.043	<0.01	<0.01	
					14	fruit	<u>0.123</u>	<u>≤0.01</u>	<u>≤0.01</u>	
PA/USA 1990[1]	SC	9	**0.14**	0.0032	0	fruit	0.165	<0.01	<0.01	Burnett, 1992f
					7	fruit	0.076	<0.01	<0.01	
					14	fruit	<u>0.052</u>	<u>≤0.01</u>	<u>≤0.01</u>	
VA/USA 1990[1]	SC	8	**0.14**	0.015	0	fruit	0.154	<0.01	<0.01	Burnett, 1992f
					7	fruit	0.085	<0.01	<0.01	
					14	fruit	<u>0.080</u>	<u>≤0.01</u>	<u>≤0.01</u>	
VA/USA 1990[1]	SC	8	**0.28**	0.03	14	fruit	0.159	0.013	<0.01	Burnett, 1992f
WA/USA 1990[1]	SC	8	**0.14**	0.0035	0	fruit	0.166	<0.01	<0.01	Burnett, 1992f
					7	fruit	0.140	<0.01	<0.01	
					14	fruit	<u>0.119</u>	<u>≤0.01</u>	<u>≤0.01</u>	
					0	fruit	0.142	<0.01	<0.01	
					7	fruit	0.065	<0.01	<0.01	
					14	fruit	<u>0.038</u>	<u>≤0.01</u>	<u>≤0.01</u>	
WA/USA 1990[1]	SC	8	**0.28**	0.0067	14	fruit	0.093	0.012	<0.01	Burnett, 1992f
WV/USA 1990[1]	SC	8	**0.14**	0.0143	0	fruit	0.164	<0.01	<0.01	Burnett, 1992f
					7	fruit	0.128	<0.01	<0.01	
					14	fruit	<u>0.129</u>	<u>≤0.01</u>	<u>≤0.01</u>	
PA/USA 1993	SC	11	**0.14**	0.015	14	fruit	<u>0.116</u>	<u>0.020</u>	<u>≤0.01</u>	Batra, 1994b
	WP	11	**0.14**	0.015	14	fruit	<u>0.145</u>	<u>0.017</u>	<u>≤0.01</u>	
WA/USA 1993	SC	10	**0.14**	0.015	14	fruit	<u>0.200</u>	<u>0.028</u>	<u>≤0.01</u>	Batra, 1994b
	WP	10	**0.14**	0.015	14	fruit	<u>0.161</u>	<u>0.024</u>	<u>≤0.01</u>	
OH/USA 1995	SC	8	**0.14**	0.0347	14	fruit	<u>0.012</u>	<u>≤0.01</u>	<u>≤0.01</u>	Batra, 1996f (conc. spray)
	SC	8	**0.14**	0.0141	14	fruit	<u>0.016</u>	<u>≤0.01</u>	<u>≤0.01</u>	Batra, 1996f (dilute spray)
NY/USA 1995	SC	10	**0.14**	0.0375	14	fruit	<u>0.074</u>	<u>≤0.01</u>	<u>≤0.01</u>	Batra, 1996f (conc. spray) (RH-7905=0)
	SC	10	**0.14**	0.01	14	fruit	<u>0.053</u>	<u>0.010</u>	<u>≤0.01</u>	Batra, 1996f (dilute spray) (RH-7905=0)
WA/USA 1995	SC	8	**0.14**	0.0375	14	fruit	<u>0.197</u>	<u>≤0.01</u>	<u>≤0.01</u>	Batra, 1996f (conc. spray)
	SC	8	**0.14**	0.01	14	fruit	<u>0.129</u>	<u>≤0.01</u>	<u>≤0.01</u>	Batra, 1996f (dilute spray)
CA/USA 1995	SC	8	**0.14**	0.0333	14	fruit	<u>0.273</u>	<u>0.016</u>	<u>≤0.01</u>	Batra, 1996f (conc. spray) (RH-7905=0)

Location	Application				PHI	Sample	Residue, mg/kg			Reference/
Year, (notes)	Form.	No.	kg ai/ha	kg ai/hl	days		Fenbuc-onazole	RH-9129	RH-9130	Comment
	SC	8	**0.14**	0.01	14	fruit	0.283	0.027	≤0.01	Batra, 1996f (dilute spray) (RH-7905=0)
MI/USA 1995	SC	9	**0.14**	0.0319	14	fruit	0.159	0.010	≤0.01	Batra, 1996f (conc. spray)
	SC	9	**0.14**	0.0139	14	fruit	0.179	0.011	≤0.01	Batra, 1996f (dilute spray)
OR/USA 1995	SC	10	**0.14**	0.0371	14	fruit	0.122	≤0.01	≤0.01	Batra, 1996f (conc. spray) (RH-7905=0)
	SC	10	**0.14**	0.0099	14	fruit	0.171	0.009	≤0.01	Batra, 1996f (dilute spray) (RH-7905=0)
CO/USA 1995	SC	9	**0.14**	0.0333	14	fruit	0.089	0.008	≤0.01	Batra, 1996f (conc. spray)
	SC	9	**0.14**	0.0075	14	fruit	0.086	0.015	≤0.01	Batra, 1996f (dilute spray)
PA/USA 1994	SC	9	**0.14**	0.0347	14	fruit unwashed	0.0610	0.0038	≤0.01	Batra, 1996k
	SC	9	**0.14**	0.0347	14	fruit washed	0.0656	0.0044	≤0.01	Batra, 1996k
	SC	9	**0.14**	0.0347	14	wet pomace	0.152	0.0074	<0.01	Batra, 1996k conc. factor 2.46
	SC	9	**0.14**	0.0347	14	unpasteur- ized juice	0.0038	ND	<0.01	Batra, 1996k conc. factor 0.059
	SC	9	**0.14**	0.0347	14	pasteuriz- ed juice	<0.01	<0.01	<0.01	Batra, 1996k conc. factor zero
UK 1990[2] Cambs	EW	10	0.068	0.0034	0 3 5 14	fruit	0.15 0.07 0.09 0.07	0.02 0.01 0.01 0.02	<0.01 <0.01 <0.01 <0.01	Murray, 1991
UK 1991[2] Cambs	EW	9	0.068	0.0034	0 28	fruit	0.13 0.03	<0.01 0.01	<0.01 ≤0.01	Murray, 1992
UK 1991[2] Cambs	EW	9	0.068	0.0034	0 28	fruit	0.12 0.02	<0.01 ≤0.01	<0.01 ≤0.01	Murray, 1992
UK 1991[2] Norfolk	EW	9	0.068	0.0034	0 28	fruit	0.10 0.04	0.01 0.01	<0.01 ≤0.01	Murray, 1992
UK 1991[2] Cambs	EW	9	0.068	0.0034	0 28	fruit	0.18 0.04	<0.01 ≤0.01	<0.01 ≤0.01	Murray, 1992
UK 1991[2] Gloucs	EW	10 10	0.068 0.136	0.0034 0.0068	0 7 14 21 28 28	fruit fruit	0.27 0.15 0.18 0.07 0.06 0.12	0.03 0.02 0.03 0.02 0.01 0.03	<0.01 <0.01 <0.01 <0.01 ≤0.01 <0.01	Murray, 1992
UK 1991[2] Cambs	EW	11	0.068	0.0034	0 7 14 21 28	fruit	0.09 0.09 0.06 0.05 0.02	0.01 0.02 0.02 0.01 0.01	<0.01 <0.01 <0.01 <0.01 0.01	Murray, 1992
UK 1992 Essex	EW	10	0.068	0.0034	0 28	fruit	0.12 0.05	0.01 0.01	<0.01 ≤0.01	Murray1994b
UK 1992 Warks	EW	10	0.068	0.0034	0 28	fruit	0.13 0.03	0.02 0.01	<0.01 ≤0.01	Murray, 1994b

Location Year, (notes)	Application				PHI days	Sample	Residue, mg/kg			Reference/ Comment
	Form.	No.	kg ai/ha	kg ai/hl			Fenbuc-onazole	RH-9129	RH-9130	
UK 1992 Norfolk	EW	11	0.068 0.132	0.0034 0.0068	0 28 0 28	fruit Whole Fruit	0.09 0.02 0.14 0.04	<0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01	Murray, 1994b
UK 1992 Kent	EW	10	0.068 0.132	0.0034 0.0068	0 28 0 28	fruit fruit	0.05 0.03 0.06 0.01	<0.01 <0.01 <0.01 <0.01	<0.01 0.01 <0.01 <0.01	Murray, 1994b
N. France 1992 Charentilly	EW	4	0.04	-	0 7 14 29 41	fruit	0.09 0.03 0.04 <0.02 0.02	<0.02 <0.02 <0.02 <0.02 <0.02	<0.02 <0.02 <0.02 <0.02 <0.02	 Anadiag., 1993
S.France 1993 Dame Marie Les Bois Loiret Cher	EW	2 2	0.035 0.05	- -	0 21 0 21	fruit fruit	0.20 0.17 0.26 0.28	0.02 0.03 0.03 0.04	<0.02 <0.02 <0.02 <0.02	Roussel, 1994a
Israel[3,4] 1990	EC	4	-	0.002	35	fruit	0.35			Jewin/Joffe, 1991
Italy 1990[5] Ferraro (8)	EC	12 12	(0.08) (0.16)	0.003 0.006	0 4 7 14 21 28 30 21 30	fruit fruit	0.06 0.05 0.02 0.03 <0.005 ***<0.005*** <0.005 <0.005 <0.005			Pessina, 1991h
Italy 1991[5] Padora	EW	11 11	0.06 0.12	0.003 0.006	21 28 43 21 28 43	fruit fruit	0.05 ***0.03*** 0.01 0.03 0.05 0.02			Pessina, 1992s
S France 1992 Durance	EW	9	0.04	-	33	fruit	0.03	<0.02	<0.02	Rhone-Poulenc, 1993a
S France 1992 Sauveterre	EW	9	0.03	-	20	fruit	0.04	<0.02	<0.02	Rhone-Poulenc, 1993b
S France 1989[5] Villemade	EW	8	0.05 0.07	-	27 27	fruit fruit	<0.02 <0.02	<0.02 <0.02	<0.02 <0.02	Journet, 1990
S France 1993 Gironde	EW	6	0.05		0 21	fruit	0.07 0.03	<0.02 <0.02	<0.02 <0.02	Herrise, 1994a
S France 1993 Montaban	EW	4	0.05		28	fruit	0.03	<0.02	<0.02	Herrise, 1994b
S France 1993 Tourouzelle	EW	6	0.05		0 21	fruit	0.09 0.05	<0.02 <0.02	<0.02 <0.02	 Herrise, 1994c
S France 1993 Les Cheres	EW	10	0.05		0 21	fruit	0.07 0.04	<0.02 <0.02	<0.02 <0.02	 Herrise, 1994d
S France 1993 Caumont	EW	4	0.05		0 21	fruit	0.03 0.03	<0.02 <0.02	<0.02 <0.02	 Herrise, 1994e
S France 1993 Regnenas	EW	4	0.05		0 21	fruit	0.05 0.02	<0.02 <0.02	<0.02 <0.02	 Herrise, 1994f

Location Year, (notes)	Application				PHI days	Sample	Residue, mg/kg			Reference/ Comment
	Form.	No.	kg ai/ha	kg ai/hl			Fenbuc-onazole	RH-9129	RH-9130	
Greece 1993 Larissa	EW	4	0.03	0.003	0 7 14 21 28 35	fruit	0.04 0.02 <0.01 0.03 ***<0.01*** <0.01	<0.01 <0.01 <0.01 <0.01 ***<0.01*** <0.01	<0.01 <0.01 <0.01 <0.01 ***<0.01*** <0.01	Huntingdon, 1994a
Italy 1994 Ferrara	EW	6 6	0.049 0.095	0.003 0.006	0 28 0 28	fruit fruit	0.07 ***0.02*** 0.14 0.02	0.01 ***0.01*** 0.01 0.01	0.01 ***0.01*** 0.01 0.01	Pessina, 1995a
Italy 1995 Ferrara	EW	6 6	0.046 0.093	0.003 0.006	0 28 0 28	fruit fruit	0.07 ***0.01*** 0.14 0.03	0.01 ***0.01*** 0.01 0.01	0.01 ***0.01*** 0.01 0.01	Pessina, 1995a
South Africa 1994[4,6]	EW	1	-	0.004	0 14 28		0.15 0.09 0.08			Elgin, 1994

[1]Results corrected for recoveries
[2]Samples stored for more than 6 months before analysis
[3]No recovery data with trial but acceptable recoveries (70-120%) from this commodity reported
[4]Duration of sample storage unspecified
[5]Report was not in English
[6]No detailed study report submitted

Pears. GAP was reported for Greece, Israel, Italy, Portugal, South Africa and the UK, and pending GAP for France. The maximum application concentrations are 0.002-0.004 kg ai/hl, except in the UK where the application rate is defined as 0.068 kg ai/ha. PHIs are either 14 or 28 days.

The residues in trials considered to comply with Southern European GAP (France, Greece, Italy and Portugal) are underlined in Table 47.

Table 47. Supervised residue trials on pears. Fruit analysed.

Country Year, (notes)	Application				PHI, days	Residue, mg/kg			Ref.
	Form.	No.	kg ai/ha	kg ai/hl		Fenbuc-onazole	RH-9129	RH-9130	
Italy 1990[1] Ravenna	EC	6 6	0.10 0.20	0.003 0.006	14 21 14 21	0.02 0.02 0.05 0.02			Pessina 1991e
Italy 1991[1] Ferrara	EW	6 5	0.06 0.12	0.003 0.006	28 42 28 42	0.01 0.02 0.03 0.03			Pessina 1992i
Spain 1992 Montserrat	SE	4 4	0.037 0.056	- -	36 36	0.034 0.057	<0.02 <0.02	<0.02 <0.02	Anadiag. 1993d
South Africa 1994[2,3]	EW	1	-	0.004	0 14 28	0.13 0.08 0.04			Applefarth 1995

[1]Report was not in English [2]Duration of sample storage unspecified [3]No detailed study report submitted

Cherries. GAP was reported only for the USA. The maximum application is 0.105 kg ai/ha with a PHI of 0 days.

The residues in trials considered to comply with US GAP are underlined in Table 48.

Table 48. Supervised residue trials on cherries, USA. Whole fruit analysed.

State Year (notes)	Application				PHI days	Residue, mg/kg				Reference
	Form.	No.	kg ai/ha	kg ai/hl		Fenbuc-onazole	RH-9129	RH-9130	RH-6467	
Results corrected for recoveries. RH-6467 not determined										
CA 1990	SC	5	**0.11**	0.012	0	0.202	≤0.01	≤0.01		Burnett, 1991c
					4	0.192	<0.01	<0.01		
					7	0.125	<0.01	<0.01		
CA 1990	SC	5	**0.22**	0.024	0	0.355	<0.01	<0.01		Burnett, 1991c
					4	0.191	<0.01	<0.01		
					7	0.168	<0.01	<0.01		
WA 1990	SC	5	**0.11**	0.0021	0	0.212	≤0.01	≤0.01		Burnett, 1991c
					3	0.286	<0.01	<0.01		
					7	0.128	<0.01	<0.01		
WA 1990	SC	5	**0.22**	0.0043	0	0.258	<0.01	<0.01		Burnett, 1991c
					3	0.381	<0.01	<0.01		
					7	0.349	<0.01	<0.01		
MI 1990	SC	6	**0.11**	0.0082	0	0.333	0.0269	≤0.01		Burnett, 1991c
					3	0.303	0.0256	<0.01		
					7	0.167	0.0616	0.0167		
MI 1990	SC	6	**0.22**	0.016	0	0.539	0.0327	<0.01		Burnett, 1991c
					3	0.295	0.0564	0.0167		
					7	0.429	0.0867	0.0212		
PA 1987[1,2]	SC	4	**0.11**	0.0039	14	0.144	0.015	<0.01		Burnett, 1991d Martin, 1988a
PA 1987[1,2]	SC	4	**0.22**	0.0079	14	0.273	0.0161	<0.01		Burnett, 1991d Martin, 1988a
CA 1987[3]	SC	5	**0.11**	0.0023	0	0.359	≤0.01	≤0.01		Burnett, 1992e
CA 1987[1,2]	SC	5	**0.11**	0.0026	7	0.252	<0.01	<0.01		Burnett, 1991d Martin, 1988a
					15	0.0285	<0.01	<0.01		
CA 1987[3]	SC	5	**0.11**	0.0023	0	0.511	0.0126	≤0.01		Burnett, 1992e
CA 1987[1,2]	SC	5	**0.11**	0.0026	7	0.216	<0.01	<0.01		Burnett, 1991d Martin, 1988a
					15	0.409	0.0249	0.022		
OR 1987[3]	SC	5	**0.11**	0.012	0	0.307	≤0.01	≤0.01		Burnett, 1992e
0R 1987[1,2]	SC	5	**0.11**	0.012	7	<0.01	<0.01	<0.01		Burnett, 1991d Martin, 1988a

State Year (notes)	Application				PHI days	Residue, mg/kg				Reference
	Form.	No.	kg ai/ha	kg ai/hl		Fenbuc-onazole	RH-9129	RH-9130	RH-6467	
					14	<0.01	<0.01	<0.01		
MI 1987[3]	SC	6	**0.11**	0.0589	0	0.422	0.0656	≤0.01		Burnett, 1992e
MI 1987[1,2]	SC	6	**0.11**	0.059	7	0.471	0.0774	0.016		Burnett, 1991d Martin, 1988a
					14	0.209	0.105	0.0117		
MI 1987[3]	SC	6	**0.11**	0.0589	0	0.358	0.0553	≤0.01		Burnett, 1992e
MI 1987[1,2]	SC	6	**0.11**	0.0589	7	0.427	0.0674	0.0156		Burnett, 1991d Martin, 1988a
					14	0.274	0.0866	0.0177		
WI 1987[3]	SC	6	**0.11**	0.0059	0	0.341	0.0518	≤0.01		Burnett, 1992e
WI 1987[1,2]	SC	6	**0.11**	0.0059	7	0.117	0.0702	0.0138		Burnett, 1991d Martin, 1988a
					14	0.0887	0.0796	<0.01		
Results not corrected for recoveries										
WA 1993	SC	6	**0.14**	0.091	0	0.525	0.039	<0.01	<0.01	Batra, 1993b
USA 1993	WP	6	**0.14**	0.091	0	0.553	0.0292	<0.01	<0.01	
PA 1993	SC	6	**0.14**	0.0248	0	0.468	0.164	<0.01	<0.01	Batra, 1993b
USA 1993	WP	6	**0.14**	0.0248	0	0.434	0.103	<0.01	<0.01	

[1]Metabolites determined about 3.5 years after sampling
[2]Samples stored for more than 6 months before analysis
[3]Samples stored for 3.5-4 years before analysis

Apricots. GAP was reported for Israel and the USA, and pending GAP for France. The maximum applications are 0.0025 kg ai/hl in Israel, 0.0075 kg ai/hl in France, and 0.105 kg ai/ha in the USA, with PHIs of 0-14 days.

The residues in trials considered to comply with US GAP and the pending French GAP with the highest application rate (0.0075kg ai/hl) are underlined and doubly underlined respectively in Table 49.

Table 49.-Supervised residue trials on apricots.

Location, Year (notes)	Application				PHI, days	Sample	Residue, mg/kg				Reference/ Comments
	Form.	No.	kg ai/ha	kg ai/hl			Fenbuc-onazole	RH-9129	RH-9130	RH-6467	
CA/USA 1993	SC	6	**0.140**	0.091	0	whole fruit	0.157	0.0114	≤0.01	≤0.01	Batra, 1993b
	WP	6	**0.140**	0.091	0	whole fruit	0.214	0.012	≤0.01	≤0.01	
WA/USA 1993	SC	6	**0.140**	0.015	0	whole fruit	0.268	0.0114	≤0.01	≤0.01	Batra, 1993b
	WP	6	**0.140**	0.015	0	whole fruit	0.254	0.0135	≤0.01	≤0.01	

Location, Year (notes)	Application				PHI, days	Sample	Residue, mg/kg				Reference/ Comments
	Form.	No.	kg ai/ha	kg ai/hl			Fenbuc-onazole	RH-9129	RH-9130	RH-6467	
Italy 1991[1] Bologna	EC	2	0.120	**0.005**	7	fruit	0.05				Pessina 1991b
					14		0.03				
		2	0.240	0.010	7	fruit	0.09				
					14		0.07				
Italy 1996 Malborgh	EC	5	0.074	0.005	0	fruit without stone	0.13	0.02	<0.01		Pessina 1996b
					3		0.05	0.01	<0.01		
					7		0.08	0.03	<0.01		
			0.012	0.0075	0	fruit without stone	0.14	0.03	<0.01		
					3		0.06	0.02	<0.01		
					7		0.08	<0.03	<0.01		
France[2] 1996 Beaucaire	EC	5	0.049	0.005	0	fruit without stone	0.16	0.03	<0.01		Promovert 1996b
					3		0.09	0.02	<0.01		
					7		0.10	0.02	<0.01		
		5	0.075	0.0075	0	fruit without stone	0.33	0.04	<0.01		
					3		0.26	0.04	<0.01		
					7		0.15	0.04	<0.01		
France[2] 1996 Beaucaire	EC	5	0.052	0.0050	0	fruit without stone	0.16	0.04	<0.01		Promovert 1996b
					3		0.14	0.03	<0.01		
					7		0.19	0.04	<0.01		
		5	0.085	0.0075	0	fruit without stone	0.23	0.03	<0.01		
					3		0.21	0.05	<0.01		
					7		0.14	0.05	<0.01		
France, South 1995[3] Tupin-Semons	`	5	0.047	0.0067	0	fruit (4 cm)	0.13	0.01	<0.01		Ross & Howie 1996a Only day-7 samples corrected for stone weight
			0.050	0.0068							
			0.050	0.0068	3		0.17	0.02	<0.01		
			0.050	0.0068							
			0.050	0.0068	7		0.10	0.02	<0.01		
France, South 1995[3] Tupin-Semons			0.074	0.0103	0	fruit (4 cm)	0.25	0.03	<0.01		Ross & Howie 1996a Only day-7 samples corrected for stone weight
			0.075	0.0103							
			0.075	0.0103	3		0.33	0.03	<0.01		
			0.075	0.0103							
			0.075	0.0103	7		0.23	0.04	<0.01		

[1]Report was not in English
[2]No recovery data with trial but acceptable recoveries (70-120%) from this commodity reported
[3]Samples stored for more than 6 months before analysis

Peaches. GAP was reported for Israel and the USA, and pending GAP for France and South Africa. The maximum application rates are 0.002-0.005 kg ai/hl or 0.105 kg ai/ha, with PHIs of 0 to 14 days.

The residues in trials considered to comply with US GAP are underlined in Table 50. The pending French GAP was originally reported by the company as having a 60-day PHI but the Meeting was informed that the PHI should be 3 days. The results considered to comply with this amended GAP are double underlined in the Table. They were 0.07 (3), 0.09, 0.10 (2), 0.11, 0.13 and 0.21 mg/kg.

Table 50. Supervised residue trials on peaches.

Country, Year (notes)	Application				PHI, days	Fruit sample	Residue, mg/kg			Reference/ Comment
	Form.	No.	kg ai/ha	kg ai/hl			Fenbuc-onazole	RH-9129	RH-9130	
CA/USA 1990[1]	SC	7	**0.11**	0.012	0	W/S	0.367	≤0.01	≤0.01	Burnett, 1991a
					3	W/S	0.341	<0.01	<0.01	
					7	W/S	0.362	0.0082	<0.01	
CA/USA 1990[1]	SC	7	**0.22**	0.024	0	W/S	1.29	0.0054	<0.01	Burnett, 1991a
					3	W/S	1.42	0.0102	<0.01	
					7	W/S	1.23	0.0182	0.0034	
WA/USA 1990[1]	SC	7	**0.11**	0.0045	0	W/S	0.505	0.0046	≤0.01	Burnett, 1991a
					3	W/S	0.213	<0.01	<0.01	
					7	W/S	0.0989	0.0082	<0.01	
WA/USA 1990[1]	SC	7	**0.22**	0.009	0	W/S	0.276	0.0056	<0.01	Burnett, 1991a
					3	W/S	0.406	0.0045	<0.01	
					7	W/S	0.429	0.0103	<0.01	
PA/USA 1990[1]	SC	9	**0.11**	0.0337	0	W/S	0.189	0.0174	≤0.01	Burnett, 1991a
					3	W/S	0.126	0.0192	<0.01	
					7	W/S	0.147	0.233	<0.01	
PA/USA 1990[1]	SC	9	**0.22**	0.067	0	W/S	0.318	0.0183	<0.01	Burnett, 1991a
					3	W/S	0.179	0.015	<0.01	
					7	W/S	0.278	0.0269	<0.01	
GA/USA 1990[1]	SC	7	**0.11**	0.024	0	W/S	0.248	0.0125	≤0.01	Burnett, 1991a
					4	W/S	0.184	0.0115	<0.01	
					7	W/S	0.152	0.0134	<0.01	
GA/USA 1990[1]	SC	7	**0.22**	0.047	0	W/S	0.278	0.0092	<0.01	Burnett, 1991a
					4	W/S	0.218	0.0113	<0.01	
					7	W/S	0.117	0.0087	<0.01	
GA/USA 1990[1]	SC	8	**0.11**	0.024	0	W/S	0.252	0.0157	≤0.01	Burnett, 1991a
					4	W/S	0.0789	0.0065	<0.01	
					7	W/S	0.011	0.0082	<0.01	
GA/USA 1990[1]	SC	8	**0.22**	0.047	0	W/S	0.324	0.0158	<0.01	Burnett, 1991a
					4	W/S	0.153	0.01	<0.01	
					7	W/S	0.117	0.0056	<0.01	
AR/USA 1987[1,2]	SC	9	**0.22**	0.024	0	W/S	0.585	0.0112	<0.01	Burnett, 1992e
AR/USA 1987[1,3,4]	SC	9	**0.22**	0.024	7	W/S	0.219	0.0111	<0.01	Burnett, 1991b
					14	W/S	0.062	0.0855	<0.01	
CA/USA 1987[1,3,4]	SC	7	**0.11**	0.003	0	W/S	0.278	0.0098	0.0083	Burnett, 1991b
					14	W/S	0.196	0.007	<0.01	

Country, Year (notes)	Application				PHI, days	Fruit sample	Residue, mg/kg			Reference/ Comment
	Form.	No.	kg ai/ha	kg ai/hl			Fenbuconazole	RH-9129	RH-9130	
					21	W/S	0.136	0.0066	<0.01	
CA/USA 1987[1,3,4]	SC	7	**0.22**	0.006	0	W/S	1.18	0.0075	<0.01	Burnett, 1991b
					14	W/S	0.257	0.015	0.01	
					21	W/S	0.237	0.0086	0.00315	
NC/USA 1987[1,3,4]	SC	10	**0.11**	0.0059	14	W/S	0.457	0.0266	<0.01	Burnett, 1991b
NC/USA 1987[1,2]	SC	10	**0.22**	0.012	0	W/S	1.55	0.0342	<0.01	Burnett, 1992e
NC/USA 1987[1,3,4]	SC	10	**0.22**	0.012	14	W/S	1.4	0.05	0.0035	Burnett, 1991b
PA/USA 1987[1,3,4]	SC	8	**0.11**	0.0039	0	W/S	0.459	0.0158	<0.01	Burnett, 1991b
					7	W/S	0.408	0.0243	<0.01	
					14	W/S	0.308	0.023	<0.01	
					21	W/S	0.238	0.0149	<0.01	
PA/USA 1987[1,3,4]	SC	8	**0.22**	0.0079	0	W/S	1.35	0.03	0.0066	Burnett, 1991b
					7	W/S	0.778	0.033	0.0054	
					14	W/S	0.842	0.0353	<0.01	
					21	W/S	0.396	0.0233	0.0017	
AR/USA 1987	SC	9	**0.11**	0.0118	0	whole	0.116	≤0.01	≤0.01	Burnett, 1991e
AR/USA 1987	SC	9	**0.22**	0.024	0	whole	0.585	0.0112	<0.01	Burnett, 1991e
AR/USA 1987	SC	10	**0.22**	0.0118	0	whole	1.55	0.0342	<0.01	Burnett, 1991e
GA/USA 1993	SC	10	**0.14**	0.029	0	whole	0.376	0.0105	<0.01	Batra, 1993b (RH-6467=0)
	WP	10	**0.14**	0.029	0	whole	0.477	0.0152	<0.01	Batra, 1993b (RH-6467=0)
VA/USA 1995	WP	4	**0.101**	0.0108	8	whole	0.0668	0.0030	<0.01	Batra, 1996l unwashed
VA/USA 1995	WP	4	**0.101**	0.0108	8	whole	0.0414	0.0037	<0.01	Batra, 1996l washed
PA/USA 1995	WP	2	**0.101**	0.0216	7	whole	0.121	<0.01	<0.01	Batra, 1996l unwashed
PA/USA 1995	WP	2	**0.101**	0.0216	7	whole	0.0572	<0.01	<0.01	Batra, 1996l washed
NJ/USA 1995	WP	2	**0.101**	0.011	8	whole	0.0448	<0.01	<0.01	Batra, 1996l unwashed
NJ/USA 1995	WP	2	**0.101**	0.011	8	whole	0.0496	<0.01	<0.01	Batra, 1996l washed
GA/USA 1995	WP	2	**0.101**	0.0216	7	whole	0.0254	<0.01	<0.01	Batra, 1996l unwashed
GA/USA 1995	WP	2	**0.101**	0.0216	7	whole	0.0157	<0.01	<0.01	Batra, 1996l washed

Country, Year (notes)	Application				PHI, days	Fruit sample	Residue, mg/kg			Reference/ Comment
	Form.	No.	kg ai/ha	kg ai/hl			Fenbuc-onazole	RH-9129	RH-9130	
SC/USA 1995	WP	1	**0.105**	0.0112	17	whole (RAC)	0.0588	<0.01	<0.01	Batra, 1996k
SC/USA 1995	WP	1	**0.105**	0.0112	17	whole (line start)	0.0549	<0.01	<0.01	Batra, 1996k
SC/USA 1995	WP	1	**0.105**	0.0112	17	peeled (inspection belt)	0.0044	<0.01	<0.01	Batra, 1996k
SC/USA 1995	WP	1	**0.105**	0.0112	17	purée (baby food)	0.0044	<0.01	<0.01	Batra, 1996k
Israel, 1991[5,6]	EC	3-5?	≤0.03	0.002	10	without stone	0.17			Jewnin
					13		0.07			Joffe, 1991
					18		0.06			
		3-5?	≤0.06	0.004	10	without stone	0.19			
					13		0.13			
					18		0.19			
Italy , 1991[7]	EC	2	0.13	0.005	0	whole	0.17			Pessina, 1991i
Ravenna					3-4		0.13			
					7		0.06			
					14		0.07			
		2	0.26	0.010	7	whole	0.12			
					14		0.07			
Italy , 1991[7]	EW	5	0.075	0.005	3-4	whole	0.07			Pessina, 1992h
Ravenna					7		0.06			
					14		0.05			
		5	0.15	0.010	3-4	whole	0.13			
					7		0.13			
					14		0.07			
Italy , 1991[7]	EC	3	0.05	0.005	20	whole	0.02			Pessina, 1992m
Livorno		3	0.10	0.010	20	whole	0.01			
Italy , 1991[7] Copparo	SC	2	0.07	0.005	14	whole	0.01			Pessina, 1992c
Italy , 1992[7] Copparo	SC	2	0.07	0.005	14	whole	0.02			Pessina, 1992c
Italy , 1995	EW	4	0.078	0.005	0	without stone	0.34	0.01	<0.01	R80.2
Ferrara					3		0.21	0.01	<0.01	F Pessina, 1995b
					7		0.10	<0.01	<0.01	
Italy, 1995	EW	4	0.076	0.005	0	without stone	0.09	<0.01	<0.01	R80.2
Ferrara					3		0.11	0.01	<0.01	F Pessina, 1995b
					7		0.06	0.01	<0.01	
Italy, 1995	EC	4	0.075	0.005	0	without stone	0.16	0.01	<0.01	R80.3
Ferrara					3		0.10	0.01	<0.01	F Pessina, 1995b
					7		0.08	0.02	<0.01	
Italy, 1995	EW	4	0.074	0.005	0	without stone	0.17	0.02	<0.01	R80.4
Ferrara					3		0.10	0.02	<0.01	F Pessina, 1995c
					7		0.12	0.02	<0.01	

Country, Year (notes)	Application				PHI, days	Fruit sample	Residue, mg/kg			Reference/ Comment
	Form.	No.	kg ai/ha	kg ai/hl			Fenbuc-onazole	RH-9129	RH-9130	
S. France, 1996 Fronton	EC	5	0.053	0.005	0	without stone	0.11	0.01	<0.01	R81.10 Promovert, 1996a
					3		0.07	0.01	<0.01	
					7		0.04	<0.01	<0.01	
		5	0.082	0.0075	0	without stone	0.09	0.01	<0.01	
					3		0.11	0.02	<0.01	
					7		0.08	0.02	0.01	
S. France, 1996 St Gilles	EC	4	0.050	0.0061	1	without stone	0.13	0.01	<0.01	R81.10 Promovert, 1996a
					3		0.07	<0.01	<0.01	
					7		0.06	<0.01	<0.01	
		4	0.078	0.0090	1		0.26	0.01	<0.01	
					3	without stone	0.16	0.01	<0.01	
					7		0.09	0.01	<0.01	
S. France Saint Bazeille, 1995[3]	EC	5	0.050	0.031	2 hrs	whole	0.07	<0.01	<0.01	Ross & Howie 1996b Only day 7 samples corrected for stone weight
					3		0.05	0.01	<0.01	
			0.075	0.047	7		0.03	<0.01	<0.01	
					2 hrs		0.10	<0.01	<0.01	
					3		0.09	<0.01	<0.01	
					7		0.05	<0.01	<0.01	
S. France Meynes, 1995[3]	EC	5	0.050	0.021	2 hrs	whole	0.07	<0.01	<0.01	Ross & Howie 1996b Only day 7 samples corrected for stone weight
					3		0.07	0.01	<0.01	
			0.075	0.032	7		0.06	0.02	<0.01	
					2 hrs		0.11	0.02	<0.01	
					3		0.15	0.02	<0.01	
					7		0.07	0.02	<0.01	

W/S: without stone

[1]Results corrected for recoveries
[2]Samples stored for 3.5-4 years before analysis
[3]Samples stored for more than 6 months before analysis
[4]Metabolites determined about 3.5 years after sampling
[5]Duration of sample storage unspecified
[6]No recovery data with trial but acceptable recoveries (70-120%) from this commodity reported
[7]Report was not in English

Plums and prunes. GAP was reported for Israel and pending GAP for the USA and France. The maximum application rates are 0.002-0.0075 kg ai/hl in France and Israel and 0.105 kg ai/ha in the USA. PHIs are 0-14 days.

The residues in trials considered to comply with the pending US and French GAP with the highest application rate (0.0075kg ai/hl) are underlined and double underlined respectively in Table 51.

Table 51. Supervised residue trials on plums and prunes.

Location, Year	Application				PHI, days	Fruit sample	Residue, mg/kg			Reference & Comment
	Form.	No.	kg ai/ha	kg ai/hl			Fenbuc-onazole	RH-9129	RH-9130	
CA/USA 1990[1]	SC	6	**0.11**	0.0087	0	WS	0.011	≤0.01	≤0.01	Burnett, 1991e/ plums

Location, Year	Application				PHI, days	Fruit sample	Residue, mg/kg			Reference & Comment
	Form.	No.	kg ai/ha	kg ai/hl			Fenbuc-onazole	RH-9129	RH-9130	
					3	WS	0.012	<0.01	<0.01	
					7	WS	0.023	<0.01	<0.01	
CA/USA 1990[1]	SC	6	**0.22**	0.0173	0	WS	0.023	<0.01	<0.01	Burnett, 1991e/ plums
					3	WS	0.019	<0.01	<0.01	
					7	WS	0.027	<0.01	<0.01	
WA/USA 1990[1]	SC	6	**0.11**	0.0031	0	WS	0.071	≤0.01	≤0.01	Burnett, 1991e/ plums
					3	WS	0.020	<0.01	<0.01	
					7	WS	0.018	<0.01	<0.01	
WA/USA 1990[1]	SC	6	**0.22**	0.0061	0	WS	0.077	<0.01	<0.01	Burnett, 1991e/ plums
					3	WS	0.043	<0.01	<0.01	
					7	WS	0.029	<0.01	<0.01	
MI/USA 1990[1]	SC	9	**0.11**	0.0087	0	WS	0.056	≤0.01	≤0.01	Burnett, 1991e/ plums
					2	WS	0.074	<0.01	<0.01	
					7	WS	0.037	<0.01	<0.01	
MI/USA 1990[1]	SC	9	**0.22**	0.0173	0	WS	0.143	<0.01	<0.01	Burnett, 1991e/ plums
					2	WS	0.111	<0.01	<0.01	
					7	WS	0.088	<0.01	<0.01	
CA/USA 1987[1,2]	SC	7	**0.11**	0.0038	0	whole	0.030	≤0.01	≤0.01	Burnett, 1992e/ plums
CA/USA 1987[1,2]					7	WS	0.047	<0.01	<0.01	Burnett, 1991f/ plums
					14	WS	0.071	<0.01	<0.01	
CA/USA 1987[1,2]	SC	8	**0.11**	0.0038	0	whole	0.028	≤0.01	≤0.01	Burnett, 1992e/ plums
CA/USA 1987[1,2]					6	WS	0.072	<0.01	<0.01	Burnett, 1991f/ plums
					14	WS	0.015	<0.01	0.0029	
WA/USA 1987[1,2]	SC	8	**0.11**	0.0073	0	whole	0.040	≤0.01	≤0.01	Burnett, 1992e/ plums
WA/USA 1987[1,2]	SC	8	**0.11**	0.0073	7	WS	<0.01	<0.01	<0.01	Burnett, 1991f/ plums
MI/USA 1987[1,2]	SC	6	**0.11**	0.0589	0	whole	≤0.01	≤0.01	≤0.01	Burnett, 1992e/ plums
MI/USA 1987[1,2]					7	WS	0.096	<0.01	<0.01	Burnett, 1991f/ plums
					14	WS	0.071	<0.01	<0.01	
ID/USA 1988[1,2]	SC	5	**0.11**	0.0015	15	WS	0.032	<0.01	<0.01	Burnett, 1991f/ plums
WA/USA 1990[1]	SC	6	**0.11**	0.0031	0	WS	0.139	<0.01	<0.01	Burnett, 1991g/ prunes (dried)
					3	WS	0.082	<0.01	<0.01	
					7	WS	0.079	<0.01	<0.01	
WA/USA 1990[1]	SC	6	**0.22**	0.0061	0	WS	0.259	<0.01	<0.01	Burnett, 1991g/ prunes (dried)
					3	WS	0.102	<0.01	<0.01	

Location,	Application				PHI,	Fruit	Residue, mg/kg			Reference & Comment
Year	Form.	No.	kg ai/ha	kg ai/hl	days	sample	Fenbuc-onazole	RH-9129	RH-9130	
					7	WS	0.102	<0.01	<0.01	
CA/USA 1990[1]	SC	6	**0.11**	0.0118	0	WS	0.037	≤0.01	≤0.01	Burnett, 1991g/ prunes (fresh)
					7	WS	0.029	<0.01	<0.01	
					14	WS	0.030	<0.01	<0.01	
CA/USA 1990[1]	SC	6	**0.11**	0.0118	0	WS	0.156	0.0124	<0.01	Burnett, 1991g/ prunes (dried)
					7	WS	0.097	0.006	<0.01	
					14	WS	0.084	<0.01	<0.01	
CA/USA 1990[1]	SC	6	**0.11**	0.0118	0	WS	0.024	≤0.01	≤0.01	Burnett, 1991g/ prunes (fresh)
					7	WS	0.019	<0.01	<0.01	
					14	WS	0.018	<0.01	<0.01	
CA/USA 1990[1]	SC	6	**0.11**	0.0118	0	WS	0.084	<0.01	<0.01	Burnett, 1991g/ prunes (dried)
					7	WS	0.067	<0.01	<0.01	
					14	WS	0.056	<0.01	<0.01	
S. France 1992 Bourran	EC	4	0.03	-	54 54	whole prunes	0.04 0.02	0.02 <0.02	0.04 <0.02	Anadiag,1993 prunes
S. France 1993 Bourran	EC	2	0.05	-	20	whole prunes	0.48	<0.02	<0.02	Lecigne, 1994 prunes
Italy 1994 Ferrara	EW	4	0.068	0.005	0 3 7	without stone	0.01 0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	Pessina 1995d
Italy 1994 Ferrara	EW	4	0.075	0.005	0 3 7	without stone	0.06 0.04 0.03	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	Pessina 1995d
Italy 1994 Ferrara	EC	4	0.067	0.005	0 3 7	without stone	0.01 0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	Pessina 1995d plums
Italy 1995 Bologna	EC	5	0.077	0.005	0 3 7	without stone	0.04 0.04 0.02	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	Pessina 1996a
			0.09	0.0075	0 3 7	without stone	0.07 0.05 0.05	<0.01 ≤0.01 <0.01	<0.01 ≤0.01 <0.01	
Italy 1995 Leno	EC	5	0.116	0.0075	0 3 7	without stone	0.07 0.06 0.04	<0.01 ≤0.01 <0.01	<0.01 ≤0.01 <0.01	Pessina 1996a
Italy 1996 Malborgh	EC	5	0.051	0.005	0 3 7	without stone	0.03 0.03 0.02	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	Pessina 1996a
			0.074	0.0075	0 3 7	without stone	0.07 0.07 0.03	<0.01 0.01 0.01	<0.01 ≤0.01 <0.01	
Italy 1996 Bologna	EC	5	0.060	0.005	0 3 7	without stone	0.04 0.04 0.03	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	Pessina 1996a
Italy 1996 Ferrara	EC	5	0.057	0.005	0 3 7	without stone	0.14 0.13 0.07	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	Pessina 1996a

Location, Year	Application Form.	No.	kg ai/ha	kg ai/hl	PHI, days	Fruit sample	Residue, mg/kg Fenbuc-onazole	RH-9129	RH-9130	Reference & Comment
			0.086	0.0075	0		0.31	<0.01	<0.01	
					3	without	0.23	≤0.01	≤0.01	
					7	stone	0.17	<0.01	<0.01	
S. France 1995 Moissac	EC	5	0.053	0.0054	0	plums	0.12	<0.01	<0.01	Promovert 1996c
					3	w'out stone	0.09	<0.01	<0.01	
					7	prunes	0.04	<0.01	<0.01	
					3	w'out stone	0.17	0.01	<0.01	
					0	without	0.26	<0.01	<0.01	
		5	0.073	0.0075	3	stone	0.16	≤0.01	≤0.01	
					7	prunes	0.13	<0.01	<0.01	
					3	w'out stone	0.36	0.02	≤0.01	
S. France 1996 St Amans	EC	5	0.051	0.0055	0	without	0.17	<0.01	<0.01	Promovert 1996c
					3	stone	0.13	<0.01	<0.01	
					7		0.12	<0.01	<0.01	
					3	prunes w'out stone	0.26	0.02	0.01	
			0.073	0.0083	0	without	0.21	0.01	<0.01	
					3	stone	0.27	0.01	≤0.01	
					7		0.24	0.01	<0.01	
					3	prunes w'out stone	0.38	0.02	≤0.01	
S. France 1996 Moissac	EC	5	0.053	0.0050	0		0.18	0.01	<0.01	Promovert 1996c
					3		0.16	0.02	<0.01	
					7		0.13	0.01	<0.01	
		5	0.075	0.0075	0		0.49	0.03	<0.01	
					3		0.30	0.02	≤0.01	
					7		0.24	0.02	<0.01	
S. France 1996 Sorgues	EC	5	0.051	0.0050	0		0.13	<0.01	<0.01	Promovert 1996c
					3	without	0.13	<0.01	0.01	
					7	stone	0.07	<0.01	<0.01	
			0.078	0.0075	0		0.32	<0.01	<0.01	
					3	without	0.20	≤0.01	≤0.01	
					7	stone	0.16	0.02	<0.01	

WS: without stone

[1]Results corrected for recoveries
[2]Samples stored for 3.5-4 years before analysis

Grapes. GAP was reported for France, Israel, Italy, Portugal, Spain and Turkey, and pending GAP for Greece. The maximum applications are 0.002–0.0075 kg ai/hl or 0.03–0.04 kg ai/ha, with PHIs of 7-28 days.

The residues in trials considered to comply with French and Spanish GAP are underlined and those complying with Italian and pending Greek GAP double underlined in Table 52.

Table 52. Supervised residue trials on grapes.

Location year	Application Form.	No.	Kg ai/ha	kg ai/hl	PHI, days	Sample	Residue, mg/kg Fenbuc-onazole	RH-9129	RH-9130	Reference & Comment
Germany, 1996 Kesten	EC	8	0.075	0.00375	0	fruit	0.96	<0.01	<0.01	Gilbert, 1996c
					7		0.92	0.01	<0.01	
					14		0.70	<0.01	<0.01	
					21		0.61	0.02	<0.01	
					28		0.48	0.01	<0.01	

 CLICK HERE to continue

Location year	Application				PHI, days	Sample	Residue, mg/kg			Reference & Comment
	Form.	No.	Kg ai/ha	kg ai/hl			Fenbuc-onazole	RH-9129	RH-9130	
Germany, 1996 Niderkirchen	EC	8	0.037- 0.060	0.00375	0 7 14 21 28	fruit	0.61 0.75 0.50 0.46 0.48	<0.01 <0.01 <0.01 <0.01 0.01	<0.01 <0.01 <0.01 <0.01 <0.01	Gilbert, 1996c
Germany, 1996 Niderkirchen	EC	8	0.060	0.00375	0 7 14 21 28	fruit	0.58 0.69 0.52 0.48 0.54	0.01 0.01 0.01 0.02 0.02	<0.01 <0.01 <0.01 <0.01 <0.01	Gilbert, 1996c
Germany, 1996 Niderkirchen	EC	8	0.037- 0.056	0.00375	0 7 14 21 28	fruit	0.41 0.43 0.41 0.36 0.32	<0.01 0.01 0.01 0.01 0.01	<0.01 <0.01 nd <0.01 <0.01	Gilbert, 1996c
S. France[1], 1987 Vayres	EC	8	0.03 0.0375		33 33	fruit fruit	0.2 0.3			Herisse, 1987
S. France[1], 1987 Coursan	EC	5	0.03		0 7 14 21 30 45	fruit	0.07 0.07 0.04 0.02 0.01 <0.01			Faugeron, 1987a
S. France [1], 1988 Montbazin	EC	7	0.0375		0 14 28 42	fruit	0.6 0.5 0.2 0.25			Faugeron, 1988. Interpolated residue= 0.35 at 21 days
S. France [1], 1988 Moulon	EC	7	0.0375		0 14 28 42	fruit	0.5 0.4 0.3 0.35	0.02 0.02 0.01 0.02		Herisse, 1988 Interpolated residue= 0.35 at 21 days
S. France [1], 1989 Moulon	EC	6	0.0375		32	fruit	0.02	0.01		Herisse, 1989a
S. France [1], 1989 Moulon	EC	6	0.0375		32	fruit	0.02	<0.01		Herisse, 1989b
S. France [1], 1989 Moulon	EC	6	0.0375		32	fruit	0.05	<0.01		Herisse, 1989c
S. France [1], 1989 Vias	EC	1 1 1 1	0.0375 0.0375 0.0375 0.0375		0 14 28 42	fruit fruit fruit fruit	0.35 0.35 0.12 0.10	0.05 0.04 0.01 0.01		Faugeron, 1989a Interpolated residue= 0.235 & 0.025 at 21 days
S. France [1], 1989 Nizas	EC	7 8	0.03 0.03		37 37	fruit fruit	0.25 0.30	0.03 0.05		Faugeron, 1989b
S. France [1], 1989 Mizas	EC	7 8	0.03 0.03		40 30	fruit fruit	0.25 0.40	0.05 0.05		Faugeron, 1989c
S. France [1], 1989 Mizas	EC	1	0.0375		0 14 28 42 57	fruit fruit fruit fruit fruit	0.35 0.40 0.30 0.20 0.18	<0.01 [0.01 [0.01 <0.01 <0.01		Faugeron, 1989d Interpolated residue= 0.35 & <0.01 at 21 days
S. France [1], 1989 Vias	EC	7 8	0.03 0.03		37 37	fruit fruit	0.25 0.10	<0.01 <0.01		Faugeron, 1989e
S. France [1] 1989 Nizas	EC	1 1 1 1 1	0.0375 0.0375 0.0375 0.0375 0.0375		0 14 28 42 57	fruit fruit fruit fruit fruit	0.35 0.30 0.10 0.04 0.06	<0.01 [0.01 [0.01 <0.01 <0.02		Faugeron, 1989f Interpolated residue= 0.2 & [0.01 at 21 days
Israel[2,3], 1991	EC	3	0.075	0.0075	0 3 5 7 21	fruit	0.83 0.54 1.01 0.86 0.58			Jewnin/ Joffe, 1991
France [1], 1990 Moulon	EC	6	0.0375		11	fruit	<0.02	<0.02	<0.02	Herisse, 1990a
France [1], 1990 Coursan	EC	6	0.0375 0.060 0.060		50 50 26	fruit fruit fruit	<0.02 0.05 0.11	<0.02 <0.02 <0.02	<0.02 <0.02 <0.02	Faugeron, 1990
France, 1991 Vayres	EC	6	0.0375		32	fruit	<0.02	<0.02	<0.02	Herisse, 1991aa

Location year	Application				PHI, days	Sample	Residue, mg/kg			Reference & Comment
	Form.	No.	Kg ai/ha	kg ai/hl			Fenbuc-onazole	RH-9129	RH-9130	
France, 1991 Vayres	EC	6	0.0375		32	fruit	<0.02	<0.02	<0.02	Herisse, 1991a
France, 1991 Vayres	EC	6	0.0375		32	fruit	0.077	<0.02	<0.02	Herisse, 1991b
France [1], 1991 Moulon	EC	6	0.0375		32 32	fruit wine	1.89 0.09	<0.02 <0.02	<0.02 <0.02	Herisse, 1991d
Italy [1], 1991 Retorbido	EC	5 5	0.03 0.06	0.003 0.006	0 1 3 7 14 21 40 14 14 21 14	fruit wine fruit wine	0.12 0.10 0.11 0.11 0.04 0.05 0.05 0.008 0.23 0.21 0.034			Pessina, 1991k
Italy [1], 1992 Gaiole	EC	5	0.045 0.090	0.003 0.006	20 20	fruit fruit	0.04 0.12			Pessina, 1992n
Italy [1], 1992 Mango	EC	5	0.06 0.12	0.003 0.006	22 22	fruit fruit	0.03 0.05			Pessina, 1992r
Italy [1], 1992 Gaiole	SC	5	0.045 0.090 0.045 0.090 0.045 0.090	0.003 0.006 0.003 0.006 0.003 0.006	14 14 21 21 35 35	fruit fruit fruit fruit fruit fruit	0.17 0.21 0.16 0.30 0.08 0.13			Pessina, 1992a
Italy [1], 1992 Battaglia	SC	1 1	0.045 0.090	0.0025 0.0025	16 23	whole fruit fruit	0.04 0.01			Pessina, 1992b (0.04 result is also comparable with the French GAP)
Italy [1], 1992 Battaglia	SC	5 5 3 3	0.027 0.027 0.027 0.027	0.0015 0.0015 0.0015 0.0015	35 50 35 50	fruit fruit fruit fruit	0.02 0.02 0.01 0.02			Pessina, 1992d
Portugal, 1993 Salvaterra	EC	3	0.0075	0.0075	12	fruit	0.05	<0.020	<0.02	Abela, 1993
S. France, 1993 Cabara	EC	5	0.0375		27 27 27	fruit must wine	0.09 <0.02 <0.02	[0.02 <0.02 <0.02	[0.02 <0.02 <0.02	Herisse/ Anadiag, 1993b
S. France, 1993 Cabara	EC	5	0.03		27	fruit must wine	0.04 <0.02 <0.02	[0.02 <0.02 <0.02	[0.02 <0.02 <0.02	Herisse/ Anadiag, 1993c
Greece[2], 1993 Attica	EC	3	0.063	0.006	0 7 14 21	fruit	0.58 0.63 0.32 0.35	<0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01	Huntingdon, 1994d
Italy, 1994 Ferrara	EW	6	0.04	0.0038	0 14 14 14 14 14	fruit fruit flower must must wine dregs wine	0.16 0.05 0.02 0.02 0.18 <0.01	<0.01 <0.01 <0.01 <0.01 0.02 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	Pessina, 1995g
Italy, 1994 Ferrara	EC	6	0.03	0.003	0 14 14 14 14 14	fruit fruit flower must must wine dregs wine	0.17 0.05 0.03 <0.01 0.17 <0.01	<0.01 <0.01 <0.01 <0.01 0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	Pessina, 1995g

[1]Report was not in English
[2]Duration of sample storage unspecified
[3]No recovery data with trial but acceptable recoveries (70-120%) from this commodity reported

Strawberries. GAP was reported only for Israel. The maximum application is 0.075kg ai/ha with a PHI of 14 days.

The residues in trials considered to comply with the Israeli GAP are underlined in Table 53.

Table 53. Supervised residue trials on strawberries.

Location year	F/G	Application				PHI, days	Sample	Residue, mg/kg			Reference & Comment
		Form.	No.	kg ai/ha	kg ai/hl			Fenbuconazole	RH-9129	RH-9130	
Spain[1], 1990 Valencia	F	EC	1	0.08	0.05	0	Fruit	0.45	Sum <0.01		Jousseaume 1990a
						3		0.40	sum <0.01		
						7		0.20	sum 0.01		
						14		0.20	sum 0.01		
				0.12	0.0075	0	Fruit	0.90	sum 0.02		
						3		0.60	sum 0.01		
						7		0.30	sum 0.02		
						14		0.20	sum 0.01		
Italy[1], 1991 Forli	F	EC	2	0.07	0.005	0	fruit	0.04			Pessina 1991j
						4		0.10			
						7		0.11			
						11		0.13			
						14		0.07			
		EC	2	0.14	0.010	0	fruit	0.04			
						4		0.29			
						7		0.14			
						11		0.18			
						14		0.14			
Spain, 1991 Valencia	F	SE	1	0.05	0.005	0	fruit	0.13			Jousseaume 1991f
						3		0.09			
						7		0.06			
						14		0.04			
						21		<0.02			
				0.075	0.0075	0	fruit	0.13			
						3		0.08			
						7		0.05			
						14		0.03			
						21		<0.02			
Italy[1], 1992 Verona	F	EW	3	0.05	0.005	3	fruit	0.01			Pessina 1992k
						7		0.017			
				0.10	0.0075	3	fruit	0.02			
						7		0.02			
Israel[2,3, 19], 1993 Tira	G	EC	?	0.03	0.005	1	fruit	0.65			Min of Agro. 1993
						3		0.48			
						5		0.20			
						10		0.07			
				0.06	0.0075	1	fruit	0.90			
						3		0.55			
						5		0.30			
						10		<u>0.17</u>			

Location year	F/G	Application				PHI, days	Sample	Residue, mg/kg			Reference & Comment
		Form.	No.	kg ai/ha	kg ai/hl			Fenbuc-onazole	RH-9129	RH-9130	
Spain [4], 1993 Heuelva	G	SE	1	0.075	0.0075	0	fruit	0.29	<0.02	<0.02	Anadiag. 1993c
						5		0.20	<0.02	<0.02	
						10		0.13	<0.02	<0.02	
			2	0.075	0.0075	0	fruit	0.38	<0.02	<0.02	
						5		0.19	<0.02	<0.02	
						10		0.15	<0.02	<0.02	
			3	0.075	0.0075	0	fruit	0.31	<0.02	<0.02	
						5		0.16	<0.02	<0.02	
						10		0.13	<0.02	<0.02	
Spain [4], 1993 Valencia	G	SE	9	0.075	-	7	fruit	0.12	<0.02	<0.02	Anadiag. 1993d
Spain, 1993 Bonares	F	SE	2	0.13	0.0075	0	fruit	0.27	<0.02	<0.02	Anadiag. 1993f
						3		0.18	<0.02	<0.02	
						7		0.08	<0.02	<0.02	
						14		0.06	<0.02	<0.02	
Spain, 1993 Bornas	G	SE	2	0.07	0.0075	0	fruit	0.10	<0.02	<0.02	Anadiag. 1993g
						3		0.12	<0.02	<0.02	
						7		0.08	<0.02	<0.02	
						14		0.03	<0.02	<0.02	
Spain [4], 1993 Moguer	F	SE	1	0.075	0.0075	0	fruit	0.28	<0.02	<0.02	Anadiag. 1993h
						5		0.18	<0.02	<0.02	
						10		0.09	<0.02	<0.02	
			2	0.075	0.0075	0		0.20	<0.02	<0.02	
						5		0.17	<0.02	<0.02	
						10		0.05	<0.02	<0.02	
			3	0.075	0.0075	0		0.30	<0.02	<0.02	
						5		0.17	<0.02	<0.02	
						10		0.08	<0.02	<0.02	
Spain [4], 1993 Sevilla	F	SE	5	0.075	0.0075	5	fruit	0.17	<0.02	<0.02	Jousseaume 1993

[1]Report was not in English
[2]Samples stored for more than 6 months before analysis

Bananas**.** GAP was reported for Columbia, Costa Rica, Ecuador, Guatemala, Honduras, Mexico, Panama, Venezuela, the Philippines and the USA. GAP in all of these countries is the same, with an application rate of 0.105 kg ai/ha and a PHI of 0 days.

The residues in trials considered to comply with GAP are underlined in Table 54.

Table 54. Supervised residue trials on bananas.

Location year	Application				PHI, days	Sample	Residue, mg/kg			Reference & Comment
	Form.	No.	kg ai/ha	kg ai/hl			Fenbuc-onazole	RH-9129	RH-9130	
HI/USA 1991[1]	SC	8	**0.099**	0.0352	0	pulp	≤0.01	≤0.01	≤0.01	Stavinski, 1992/ bagged
						peel	≤0.01	≤0.01	≤0.01	

Location year	Application				PHI, days	Sample	Residue, mg/kg			Reference & Comment
	Form.	No.	kg ai/ha	kg ai/hl			Fenbuc-onazole	RH-9129	RH-9130	
HI/USA 1991[1]	SC	8	**0.197**	0.07	0	pulp	<0.01	<0.01	<0.01	Stavinski, 1992/ bagged
						peel	0.0133	<0.01	<0.01	
FL/USA 1991[1]	SC	7	**0.099**	0.0352	0	pulp	0.0123	≤0.01	≤0.01	Stavinski, 1992/ bagged
						peel	0.03	0.0126	≤0.01	
FL/USA 1991[1]	SC	8	**0.197**	0.07	0	pulp	0.0571	0.0233	<0.01	Stavinski, 1992/ bagged
						peel	0.142	0.0469	<0.01	
HI/USA 1991[1]	SC	8	**0.099**	0.0391	0	pulp	≤0.01	≤0.01	≤0.01	Stavinski, 1992/ bagged
						peel	≤0.01	≤0.01	≤0.01	
HI/USA 1991[1]	SC	8	**0.197**	0.078	0	pulp	<0.01	<0.01	<0.01	Stavinski, 1992/ bagged
						peel	0.0106	<0.01	<0.01	
HI/USA 1991[1]	SC	8	**0.099**	0.0391	0	pulp	≤0.01	≤0.01	≤0.01	Stavinski, 1992/ bagged
					7	pulp	<0.01	<0.01	<0.01	Stavinski, 1992/ bagged
					0	peel	≤0.01	≤0.01	≤0.01	Stavinski, 1992/ bagged
					7	peel	<0.01	<0.01	<0.01	Stavinski, 1992/ bagged
HI/USA 1991[1]	SC	8	**0.197**	0.078	0	pulp	<0.01	<0.01	<0.01	Stavinski, 1992/ bagged
					7	pulp	<0.01	<0.01	<0.01	
					14	pulp	<0.01	<0.01	<0.01	
					0	peel	<0.01	<0.01	<0.01	
					7	peel	<0.01	<0.01	<0.01	
					14	peel	<0.01	<0.01	<0.01	
HI/USA 1991[1]	SC	8	**0.099**	0.0391	0	pulp	≤0.01	≤0.01	≤0.01	Stavinski, 1992/ bagged
					7	pulp	<0.01	<0.01	<0.01	
					14	pulp	<0.01	<0.01	<0.01	
					0	peel	≤0.01	≤0.01	≤0.01	
					7	peel	<0.01	<0.01	<0.01	
					14	peel	<0.01	<0.01	<0.01	
HI/USA 1991[1]	SC	8	**0.099**	0.0391	0	pulp	0.0173	≤0.01	≤0.01	Stavinski, 1992/ unbagged
					7	pulp	0.0197	<0.01	<0.01	
					14	pulp	0.0169	<0.01	<0.01	
					7	peel	0.133	0.0202	<0.01	

Location year	Application				PHI, days	Sample	Residue, mg/kg			Reference & Comment
	Form.	No.	kg ai/ha	kg ai/hl			Fenbuc-onazole	RH-9129	RH-9130	
					14	peel	0.0349	<0.01	<0.01	
					0	peel	0.0878	0.0136	≤0.01	
HI/USA 1991[1]	SC	8	**0.197**	0.078	0	pulp	<0.01	<0.01	<0.01	Stavinski, 1992/ bagged
					7	pulp	<0.01	<0.01	<0.01	
					14	pulp	<0.01	<0.01	<0.01	
					0	peel	<0.01	<0.01	<0.01	
					7	peel	<0.01	<0.01	<0.01	
					14	peel	<0.01	<0.01	<0.01	
HI/USA 1991[1]	SC	8	**0.197**	0.078	0	pulp	<0.01	<0.01	<0.01	Stavinski, 1992/ unbagged
					7	pulp	0.0522	0.0196	<0.01	
					14	pulp	0.048	0.0168	<0.01	
					0	peel	0.0643	0.0127	<0.01	
					7	peel	0.242	0.0386	<0.01	
					14	peel	0.1695	0.0332	<0.01	
Costa Rica 1994	OF	8	**0.1**	0.97	0	pulp	<0.01	<0.01	<0.01	Batra, 1994b/ bagged
					3	pulp	<0.01	<0.01	<0.01	
					7	pulp	<0.01	<0.01	<0.01	
					0	whole	≤0.01	≤0.01	≤0.01	
					3	whole	<0.01	<0.01	<0.01	
					7	whole	<0.01	<0.01	<0.01	
Costa Rica 1994	OF	8	**0.1**	0.97	0	pulp	≤0.01	≤0.01	≤0.01	Batra, 1994b/ unbagged
					3	pulp	<0.01	<0.01	<0.01	
					7	pulp	<0.01	<0.01	<0.01	
Costa Rica 1994	OF	8	**0.1**	0.97	0	whole	≤0.01	≤0.01	≤0.01	Batra, 1994b/ unbagged
					3	whole	<0.01	<0.01	<0.01	
					7	whole	<0.01	<0.01	<0.01	
Costa Rica 1994	OF	8	**0.1**	0.97	0	pulp	<0.01	<0.01	<0.01	Batra, 1994b/ bagged
					3	pulp	<0.01	<0.01	<0.01	
					7	pulp	<0.01	<0.01	<0.01	
					0	whole	≤0.01	≤0.01	≤0.01	
					3	whole	<0.01	<0.01	<0.01	
					7	whole	<0.01	<0.01	<0.01	
Costa Rica 1994	OF	8	**0.1**	0.97	0	pulp	<0.01	<0.01	<0.01	Batra, 1994b/ unbagged

Location year	Application				PHI, days	Sample	Residue, mg/kg			Reference & Comment
	Form.	No.	kg ai/ha	kg ai/hl			Fenbuconazole	RH-9129	RH-9130	
					3	pulp	<0.01	<0.01	<0.01	
					7	pulp	<0.01	<0.01	<0.01	
					0	whole	0.014	≤0.01	≤0.01	
					3	whole	0.0116	<0.01	<0.01	
					7	whole	0.0107	<0.01	<0.01	
Martinique 1994	OF	5	**0.1**	0.51	0	pulp	<0.01	<0.01	<0.01	Batra, 1994b/ bagged
	OF	5	**0.1**	0.51	4	pulp	<0.01	<0.01	<0.01	
	OF	5	**0.1**	0.51	8	pulp	<0.01	<0.01	<0.01	
	OF	5	**0.1**	0.51	0	whole	≤0.01	≤0.01	≤0.01	
	OF	5	**0.1**	0.51	4	whole	<0.01	<0.01	<0.01	
	OF	5	**0.1**	0.51	8	whole	<0.01	<0.01	<0.01	
Martinique 1994	OF	5	**0.1**	0.51	0	pulp	<0.01	<0.01	<0.01	Batra, 1994b/ unbagged
					4	pulp	<0.01	<0.01	<0.01	
					8	pulp	<0.01	<0.01	<0.01	
					0	whole	0.0188	≤0.01	≤0.01	
					4	whole	<0.01	<0.01	<0.01	
					8	whole	<0.01	<0.01	<0.01	
Mexico 1994	OF	8	**0.1**	0.043	0	pulp	<0.01	<0.01	<0.01	Batra, 1994b/ bagged
					3	pulp	<0.01	<0.01	<0.01	
					7	pulp	<0.01	<0.01	<0.01	
					0	whole	≤0.01	≤0.01	≤0.01	
					3	whole	<0.01	<0.01	<0.01	
					7	whole	<0.01	<0.01	<0.01	
Mexico 1994	OF	8	**0.1**	0.043	0	pulp	<0.01	<0.01	<0.01	Batra, 1994b/ bagged
					3	pulp	<0.01	<0.01	<0.01	
					7	pulp	<0.01	<0.01	<0.01	
					0	whole	≤0.01	≤0.01	≤0.01	
					3	whole	<0.01	<0.01	<0.01	
					7	whole	<0.01	<0.01	<0.01	

[1]Results corrected for recoveries
The trials in Costa Rica and Martinique were low-volume applications by either an aircraft-mounted sprayer or a backpack-mounted mist-blower

Melons. GAP was reported for France, Israel, Italy, Portugal, and Turkey, and pending GAP for Spain and Morocco. The defining maximum application rates are 0.0375-0.1 kg ai/ha or 0.005-0.010 kg ai/hl, with PHIs of 3 or 7 days.

The residues in trials considered to comply with the Italian and French GAP are underlined and double underlined, respectively, in Table 55.

Table 55. Supervised field trials on melons. Whole fruit analysed.

Location Year	Application				PHI, days	Residue, mg/kg			Reference & Comment
	Form.	No.	kg ai/ha	kg ai/hl		Fenbuc-onazole	RH-9129	RH-9130	
Italy 1991 Mantova [1]	EC	2 2	0.075 0.150	0.005 0.010	3 7 3 7	<0.005 ≤0.005 <0.005 <0.005			Pessina, 1991f
Italy 1992 Mantova[1]	EC	3 3	0.05 0.10	0.005 0.010	3 7 3 7	0.006 0.009 0.006 0.02			Pessina, 1992e
Italy 1992 Cremona [1]	EC	2 2	0.05 0.10	0.005 0.010	7 7	0.05 0.03	Not tested etc		Pessina, 1992p
S.France 1994 Moulon	EC	4	0.10		0 3	0.063 0.02	<0.02 <0.02	<0.02 <0.02	Herisse, 1994h
Italy 1995 Ferrara [1]	EW	3	0.05	0.005	0 3 7	0.05 0.06 0.02	<0.01 <0.01 ≤0.01	<0.01 <0.01 ≤0.01	Pessina, 1995e
Italy 1996 Ferrara	EC	6	0.1		0 3 7	0.09 0.06 0.07	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	Pessina, 1996c
Italy 1996 Ferrara	EC	6	0.1		0 3 7	0.07 0.03 0.02	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	Pessina, 1996c
S.France 1996 Loubessac [2]	EC	4	0.1		3 7	0.14 0.09			Adme Bioanal, 1996
S.France 1996 Molione[2]	EC	4	0.1		0 3 7	0.13 0.11 0.13			Adme Bioanal, 1996
S.France 1996 Villolaine[2]	EC	4	0.1		0 3 10	0.09 0.03 0.04			Adme Bioanal, 1996
S.France 1996 St Nazoine[2]	EC	4	0.1		0 3 7	0.05 <0.02 <0.02			Adme Bioanal, 1996
S.France 1996 Flourdos[2]	EC	4	0.1		3 7	0.26 0.10			Adme Bioanal, 1996

[1]Duration of sample storage unspecified
[2]Report was not in English

Watermelons. GAP was reported for Israel, Italy, Portugal, Spain and Turkey, and pending GAP for Morocco. The defining application rates are 0.025-0.0375 kg ai/ha or 0.005 -0.010 kg ai/hl with PHIs of 3 or 7 days.

The residue in the single trial according to Italian GAP is underlined in Table 56.

Table 56. Supervised residue trials on watermelons in Italy (Mantora), 1991. Pulp analysed.

Application				PHI, Days	Residue, mg/kg			Reference
Form.	No.	kg ai/ha	kg ai/hl		Fenbuconazole	RH-9129	RH-9130	
EC	2	0.075	0.005	3	<0.005			Fessina 1991g
				7	<0.005			
	2	0.150	0.010	3	<0.005			
				7	<0.005			

[1]Report was not in English

Cucumbers. GAP was reported for Israel, Spain and Turkey, and pending GAP for France and Morocco. The maximum application rates are 0.0375-0.1 kg ai/ha or 0.005-0.010 kg ai/hl with PHIs of 3 or 7 days.

The residues in trials considered to comply with the indoor Spanish GAP, the outdoor Spanish GAP and the indoor Israeli GAP are underlined, double underlined and in bold italics, respectively, in Table 57.

Table 57. Supervised residue trials on cucumbers. Whole fruit analysed.

Location year	F/ G	Application				PHI, days	Residue, mg/kg			Reference
		Form.	No.	kg ai/ha	kg ai/hl		Fenbuc-onazole	RH-9129	RH-9130	
Israel 1991[1,2]	G	EC	2	0.075	0.0075	1	0.17			Jewnin Joffe 1991
						2	0.12			
						4	0.13			
						7	***0.1***			
			2	0.150	0.015	1	0.33			
						2	0.33			
						4	0.28			
						7	0.36			
Spain 1991 Las Norias[3]	F	SE	1	0.095	0.0056	0	<0.02			Jousseaume 1991a
						3	<0.02			
						14	<0.02			
			1	0.125	0.0075	0	0.03			
						3	0.05			
						7	0.02			
						14	<0.02			
Italy 1992	F	EW	3	0.05	0.005	3	0.01			Pessina 1992j
						7	0.02			
			3	0.10	0.010	3	0.15			
						7	0.05			

Location year	F/G	Application				PHI, days	Residue, mg/kg			Reference
		Form.	No.	kg ai/ha	kg ai/hl		Fenbuc-onazole	RH-9129	RH-9130	
Italy 1992 Cremona	F	EC	3	0.05	0.005	7	0.02			Pessina 1992o
			3	0.10	0.010	7	0.015			
Spain 1993 Villadecanes	G	EC	2	0.01	0.0075	0	0.11	<0.02	<0.02	Anadiag 1993b
						3	0.07	<0.02	<0.02	
						7	0.11	≤0.02	≤0.02	
						14	0.10	<0.02	<0.02	
			1	0.01	0.0075	0	0.13	<0.02	<0.02	
						3	0.10	<0.02	<0.02	
						7	0.03	≤0.02	≤0.02	
						14	0.02	<0.02	<0.02	
Spain 1994 El Ejide	G	SE	3	0.09	0.0075	0	0.08	<0.02	<0.02	Jousseaume 1994a
						3	0.03	<0.02	<0.02	
						7	0.02	≤0.02	≤0.02	
						14	<0.02	<0.02	<0.02	
Spain 1994 Villadecanes	G	EC	3	0.11	0.093	0	0.14	<0.02	<0.02	Jousseaume 1994d
				0.11	0.060	3	0.09	<0.02	<0.02	
				0.11	0.055	7	0.03	≤0.02	≤0.02	
						14	<0.02	<0.02	<0.02	
Greece 1994 Attica	G	EC	3	0.13	0.006	0	0.03	<0.01	<0.01	Huntingdon 1994b
						3	0.02	<0.01	<0.01	
						7	≤0.01	≤0.01	≤0.01	

[1]Duration of sample storage unspecified
[2]No recoveries with trial but acceptable recoveries (70-120%) for this commodity were reported
[3]Report was not in English

Summer squash (courgettes, zucchini). GAP was reported for Israel, Spain and Turkey, and pending GAP for France and Morocco. The maximum application rates are 0.0375-0.2 kg ai/ha or 0.005-0.01 kg ai/hl with PHIs of 3 or 7 days.

In Table 58 the residues in trials considered to comply with Spanish and/or Israeli GAP are underlined, and those in the trials according to pending French GAP are double underlined. The results corresponding to pending Moroccan GAP are in bold italics. Four residues from trials in Italy and Greece which comply with Turkish GAP are marked with double asterisks.

Table 58. Supervised residue trials on summer squash. Whole fruit analysed.

Location year	F/G	Application				PHI, days	Residue, mg/kg			Reference & Comment
		Form.	No.	kg ai/ha	kg ai/hl		Fenbuc-onazole	RH-9129	RH-9130	
Italy 1991 Cesena[1]	F	EC	2	0.10	0.005	3	<0.02**			Pessina 1991d
						7	≤0.02			
				0.20	0.010	3	***<0.02***			
						7	<0.02			

Location year	F/G	Application				PHI, days	Residue, mg/kg			Reference & Comment
		Form.	No.	kg ai/ha	kg ai/hl		Fenbuconazole	RH-9129	RH-9130	
S France 1990 Moulon[2]	F	EC	4	0.075		0	<0.02	<0.02	<0.02	Herisse 1990c
						2	<0.02	<0.02	<0.02	
						3	≤0.02	≤0.02	≤0.02	
Italy 1992 Cremona[2]	F	EW	3	0.05	0.005	3	0.03**			Pessina 1992f
						7	≤0.02			
				0.10	0.010	3	***0.04***			
						7	0.04			
Italy 1992 Cremona[2]	F	EC	3	0.05	0.005	7	0.01			
				0.10	0.010	7	<0.01			
Spain 1993 Villadecanes	F	EC	2	0.075	0.0075	0	0.14	<0.02	<0.02	Anadiag 1993a
						3	0.08	≤0.02	≤0.02	
						7	≤0.02	≤0.02	≤0.02	
						14	<0.02	<0.02	<0.02	
				0.075	0.0075	0	0.11	<0.02	<0.02	
						3	***0.04***	***<0.02***	***<0.02***	
						7	<0.02	<0.02	<0.02	
			1			14	<0.02	<0.02	<0.02	
S France 1993 Coursan	F	EC	4	0.10	0.013	0	0.07	<0.02	<0.02	J Faugeron/ Anadiag. 1993
						3	***0.03***	***<0.02***	***<0.02***	
						7	0.03	<0.02	<0.02	
						14	<0.02	<0.02	<0.02	
S France 1993	F	EC		0.10	0.025	0	<0.02	<0.02	<0.02	Herisse/ Anadiag 1993a
						3	<0.02	<0.02	<0.02	
						7	<0.02	<0.02	<0.02	
						14	<0.02	<0.02	<0.02	
Spain 1994 Villadecanes	F	EC	3	0.11	0.008	0	0.21	<0.02	<0.02	Jousseaume Anadiag 1994a
						3	***0.06***	***<0.02***	***<0.02***	
						7	0.02	≤0.02	≤0.02	
						14	<0.02	<0.02	<0.02	
						7DAT2	0.04	<0.02	<0.02	
Spain 1994 Pacheco	G	SE	3	0.09	0.0075	0	0.20	<0.02	<0.02	Jousseaume Anadiag 1994b
						3	***0.08***	***<0.02***	***<0.02***	
						7	0.03	≤0.02	≤0.02	
						14	<0.02	<0.02	<0.02	
Greece 1994 Attica[3]	F	EC	4	0.126	0.006	0	0.23	<0.01	<0.01	Huntingdon 1994c
						3	0.06**	<0.01	<0.01	
						7	≤0.01	≤0.01	≤0.01	
Italy 1995 Ferara[3]	F	EW	4	0.05	0.005	0	0.03	<0.01	<0.01	Pessina 1995f
						3	0.03**	≤0.01	≤0.01	
						7	≤0.01	≤0.01	≤0.01	

[1]Samples stored for more than 6 months before analysis
[2]Report was not in English
[3]Duration of sample storage unspecified

Tomatoes. GAP was reported for Israel and pending GAP for Morocco. The application rates are 0.0075-0.01 kg ai/hl or 0.05 kg ai/ha with PHIs of 3 or 7 days.

The residues in trials considered to comply with the Israeli glasshouse GAP, the pending Moroccan GAP and the Israeli field GAP are underlined, double underlined and in bold italics, respectively, in Table 59.

Table 59. Supervised residue trials on tomatoes.

Location year	F/ G	Application				PHI, days	Sample	Residue, mg/kg			Reference
		Form.	No.	kg ai/ha	kg ai/hl			Fenbuc-onazole	RH-9129	RH-9130	
Israel	G	EC	2	0.075	0.0075	1	fruit	0.04			Jewnin Joffe
1991[1,2]						2		0.13			1991
						4		0.13			
						8		0.21			
			2	0.15	0.0150	1	fruit	0.17			
						2		0.16			
						4		0.16			
						8		0.17			
Spain	F	SE	1	0.11	0.0056	0	fruit	<0.02	<0.02	<0.02	Jousseaume
1991[3]						3		<0.02	<0.02	<0.02	1991b
La Majorca						7		<0.02	<0.02	<0.02	
						14		0.06	<0.02	<0.02	
						21		0.04	<0.02	<0.02	
			1	0.15	0.0075	0	fruit	0.04	<0.02	<0.02	
						3		0.02	<0.02	<0.02	
						7		<0.02	<0.02	<0.02	
						14		0.04	<0.02	<0.02	
						21		0.04	<0.02	<0.02	
Spain	F	SE	1	0.056	0.0056	0	fruit	0.03	<0.02	<0.02	
1991						3		0.02	<0.02	<0.02	Jousseaume
Campello						7		***0.02***	***<0.02***	***<0.02***	1991d
						15		<0.02	<0.02	<0.02	
			1	0.075	0.0075	3	fruit	0.03	<0.02	<0.02	
						7		0.03	<0.02	<0.02	
						15		<0.02	<0.02	<0.02	
Spain	F	EC	1	0.056	0.0056	0	fruit	0.03	<0.02	<0.02	
1991						7		***<0.02***	***<0.02***	***<0.02***	Jousseaume
						15		<0.02	<0.02	<0.02	1991e
Machamiel			1	0.075	0.0075	0	fruit	<0.02	<0.02	<0.02	
						3		0.03	<0.02	<0.02	
						7		<0.02	<0.02	<0.02	
						15		<0.02	<0.02	<0.02	
Italy	F	EW	3	0.05	0.005	3	fruit	0.05			Pessina
1991[3]						7		***0.02***			1992g
Parma				0.1	0.010	3	fruit	0.08			
						7		0.04			
Italy	F	EC	3	0.05	0.005	20	fruit	0.02			Pessina
1991[3]				0.1	0.010	20	fruit	0.05			1992l
Parma											
Spain	F	SE	3	0.25	0.0075	0	fruit	0.24	<0.02	<0.02	Anadiag
1992						3		0.16	<0.02	<0.02	1993e
El Ejido						7		0.12	<0.02	<0.02	
						14		0.03	<0.02	<0.02	

Location year	F/ G	Application				PHI, days	Sample	Residue, mg/kg			Reference
		Form.	No.	kg ai/ha	kg ai/hl			Fenbuc-onazole	RH-9129	RH-9130	
SFrance	G	EC	3	0.18	-	18	fruit	0.02	<0.02	<0.02	Sonito
1992[4]						washed					1993
Sorgue						18		0.02	<0.02	<0.02	
						unwashed					
						27		<0.02	<0.02	<0.02	
						washed					
						27		0.05	<0.02	<0.02	
						unwashed					
France	G	EC	4	0.15	-	0	fruit	0.05	<0.02	<0.02	Herisse
1992						washed					1993d
Moulin						0		0.07	<0.02	<0.02	
						unwashed					
						15		0.04	<0.02	<0.02	
						washed					
						15		0.02	<0.02	<0.02	
						unwashed					
						30		0.03	<0.02	<0.02	
						washed					
						30		0.06	<0.02	<0.02	
						unwashed					
Spain	G	SE	3	0.10	0.007	0	fruit	0.16	<0.02	<0.02	Jousseaume
1993						3		0.14	≤0.02	≤0.02	1994b
Balanegra						7		0.08	[0.02	[0.02	
						14		0.11	<0.02	<0.02	
Spain	G	EC	3	0.11	0.0072	0	fruit	0.24	<0.02	<0.02	Jousseaume
1994						3		0.18	≤0.02	≤0.02	1994e
Villadecanes						7		0.19	[0.02	[0.02	
						15		0.13	<0.02	<0.02	
						7DAT2		0.23	<0.02	<0.02	
Spain	G	EC	3	0.11	0.010	0DAT1	fruit	<0.02	<0.02	<0.02	Jousseaume
1994						7DAT2					1994f
Machamiel						0		<0.02	<0.02	<0.02	
						4		0.05	≤0.02	≤0.02	
						7		0.06	<0.02	<0.02	
						14		0.04	<0.02	<0.02	
								0.05	<0.02	<0.02	
Portugal	G	SE	4	0.075	0.0075	0	fruit	0.15	<0.02	<0.02	Abela/Anadiag
1993						13		0.21	<0.02	<0.02	1994
Torres Vedras											
Italy	G	EC	6	0.15	0.01	0	fruit	0.43	<0.02	0.02	Gilbert
1996						3		0.31	≤0.02	0.02	
Diana Marina						7		0.27	<0.02	0.02	
Italy	G	EC	6	0.15	0.01	0	fruit	0.42	0.02	0.02	Gilbert
1996						3		0.38	0.02	0.02	1996a
Diana Albenga						7		0.23	0.02	0.02	
S France	G	EC	6	0.14	0.01	0	fruit	0.17	<0.01	<0.01	Gilbert
1996						3		0.05	0.02	0.02	1996a
Aucamville						7		0.18	<0.01	0.02	
						3	juice	0.06	0.02	0.02	
						3	preserves	0.06	0.02	0.02	
						3	purée	0.17	0.02	0.02	

Location year	F/ G	Application				PHI, days	Sample	Residue, mg/kg			Reference
		Form.	No.	kg ai/ha	kg ai/hl			Fenbuc-onazole	RH-9129	RH-9130	
S France	G	EC	6	0.15	0.01	0	fruit	0.23	<0.02	<0.02	Gilbert
1996						3		0.10	≤0.02	≤0.02	1996a
Bresolls						7		0.17	<0.02	<0.02	
						3	juice	0.02	<0.02	<0.02	
						3	preserves	0.03	<0.02	<0.02	
						3	purée	0.11	<0.02	<0.02	

[1]Duration of sample storage unspecified
[2]No recovery data with trial but acceptable recoveries (70-120%) from this commodity reported
[3]Report was not in English
[4]Samples stored for more than 6 months before analysis

Sweet peppers. GAP was reported for Israel and pending GAP for Morocco, with application rates of 0.0075 and 0.01 kg ai/hl and corresponding PHIs of 7 and 3 days.

The residues in trials considered to comply with pending Moroccan GAP are double underlined in Table 60.

Table 60. Supervised residue trials on sweet peppers. Whole peppers analysed.

Location year	F/ G	Application				PHI, days	Residue, mg/kg			Reference
		Form.	No.	kg ai/ha	kg ai/hl		Fenbuc-onazole	RH-9129	RH-9130	
Italy	F	EC	2	0.10	0.005	3	0.01			
1990						7	0.02			F Pessina
Forli[1]				0.20	0.010	3	0.02			1991c
						7	0.04			
Spain	F	SE	1	0.08	0.0056	0	0.02	<0.02	<0.02	
1990						3	0.08	<0.02	<0.02	Jousseaume
La Monjonera[1]						14	0.08	<0.02	<0.02	1991c
						21	<0.02	<0.02	<0.02	
			1	0.10		0	0.03	<0.02	<0.02	
					0.0075	3	0.10	≤0.02	≤0.02	
						7	0.12	≤0.02	≤0.02	
						14	0.13	<0.02	<0.02	
						21	0.07	<0.02	<0.02	
Spain	G	SE	3	0.12	0.0075	0	0.43	<0.02	<0.02	
1993						3	0.38	≤0.02	≤0.02	Jousseaume
Balanegra						7	0.32	<0.02	<0.02	1994c
						14	0.24	<0.02	<0.02	
Spain	G	EC	6	0.23	0.01	0	0.19	<0.02	<0.02	R82.5
1996						3	0.20	≤0.02	≤0.02	Gilbert
Case Maria						7	0.14	<0.02	<0.02	1996b
Spain	G	EC	6	0.23	0.01	0	0.36	<0.02	<0.02	
1996						3	0.41	≤0.02	≤0.02	Gilbert
Las Torres						7	0.29	<0.02	<0.02	1996b

Location year	F/G	Application				PHI, days	Residue, mg/kg			Reference
		Form.	No.	kg ai/ha	kg ai/hl		Fenbuconazole	RH-9129	RH-9130	
Spain 1996 La Algaida	G	EC	6	0.23	0.01	0 3 7	0.14 0.18 0.08	<0.01 <0.02 <0.01	<0.01 <0.01 <0.01	Gilbert 1996b
Spain 1996 La Algaida	G	EC	6	0.23	0.01	0 3 7	0.26 0.29 0.29	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	Gilbert 1996b

[1]Report was not in English

Egg plant. GAP was reported only for Morocco, with an application rate of 0.1 kg ai/hl and a PHI of 3 days.

The residues in the one trial which accorded with Moroccan GAP are underlined in Table 61.

Table 61. Supervised residue trials on egg plant.

Location year	F/G	Application				PHI, days	Residue, mg/kg			Reference & Comment
		Form.	No.	kg ai/ha	kg ai/hl		Fenbuconazole	RH-9129	RH-9130	
Spain 1991[1] Majorca	G	SE	1	0.075	0.0056	0 3 7 14 21	0.05 0.04 0.03 <0.02 <0.02	<0.02 <0.02 <0.02 <0.02 <0.02	<0.02 <0.02 <0.02 <0.02 <0.02	Jousseaume 1991g Indoor trial
			1	0.099	0.0075	0 3 14 21	<0.02 ≤0.02 <0.02 <0.02	<0.02 ≤0.02 <0.02 <0.02	<0.02 ≤0.02 <0.02 <0.02	

[1]Report was not in English

Sugar beet. GAP was reported for Italy, and pending GAP for the USA, with application rates of 0.14 and 0.1 kg ai/ha respectively and PHIs of 14 days.

The residues in trials considered to comply with the pending US GAP and Italian GAP are underlined and double underlined, respectively, in Table 62.

Table 62. Supervised residue trials on sugar beet.

Location Year	Application				PHI, days	Sample	Residue, mg/kg			Reference
	Form.	No.	kg ai/ha	kg ai/hl			Fenbuconazole	RH-9129	RH-9130	
ID/USA 1994[1]	WP	9	0.14	0.05	0	root	0.0282	<0.01	<0.01	Batra, 1996f
					0	top[2]	7.48	0.0368	<0.01	Batra, 1996f

Location Year	Application				PHI, days	Sample	Residue, mg/kg			Reference
	Form.	No.	kg ai/ha	kg ai/hl			Fenbuconazole	RH-9129	RH-9130	
					7	root	0.0332	<0.01	<0.01	
					7	top[2]	11.4	0.0348	<0.01	
					14	root	0.0234	<0.01	<0.01	
					14	top[2]	8.85	0.044	<0.01	
ID/USA 1994[1]	WP	9	0.28	0.1	0	root	0.0596	<0.01	<0.01	Batra, 1996f
					0	top[2]	24.4	0.0712	<0.01	
					7	root	0.0502	<0.01	<0.01	
					7	top[2]	25.7	0.125	0.154	
					14	root	0.0574	<0.01	<0.01	
					14	top[2]	30.2	0.179	<0.01	
MN/USA 1994[1]	WP	8	0.14	0.075	0	root	0.0528	<0.01	<0.01	Batra, 1996f
					0	top[2]	2.34	0.0308	<0.01	
					7	root	0.0656	<0.01	<0.01	
					7	top[2]	0.828	0.0148	<0.01	
					14	root	0.0558	≤0.01	≤0.01	
					14	top[2]	0.546	0.0115	≤0.01	
MN/USA 1994[1]	WP	8	0.28	0.15	0	root	0.0834	<0.01	<0.01	Batra, 1996f
					0	top[2]	11.2	0.068	<0.01	
					7	root	0.278	<0.01	<0.01	
					7	top[2]	1.91	0.0216	<0.01	
					14	root	0.183	<0.01	<0.01	
					14	top[2]	1.69	0.0358	<0.01	
ND/USA 1994[1]	WP	8	0.14	0.075	14	root	0.0366	≤0.01	≤0.01	Batra, 1996f
						top[2]	0.796	0.0354	0.0234	
ND/USA 1994[1]	WP	8	0.28	0.15	14	root	0.0528	<0.01	<0.01	Batra, 1996f
						top[2]	0.98	0.0236	<0.01	
CA/USA 1995	WP	8	0.14	0.043	14	root	0.033	<0.01	<0.01	Batra, 1996g
						top[2]	4.17	0.0241	0.0226	
CO/USA 1995	WP	8	0.14	0.05	11	root	0.0359	<0.01	<0.01	Batra, 1996g
						top[2]	3.1	0.0175	<0.01	
CO/USA 1995	WP	8	0.14	0.05	14	root	0.0702	<0.01	<0.01	Batra, 1996g
						top[2]	1.2	0.022	<0.01	
MI/USA 1995	WP	8	0.14	0.063	14	root	0.0324	<0.01	<0.01	Batra, 1996g
						top[2]	0.513	0.0299	0.0121	
MN/USA 1995	WP	8	0.14	0.075	14	root	0.037	<0.01	<0.01	Batra, 1996g
						top[2]	0.847	0.016	<0.01	
ND/USA 1995	WP	8	0.14	0.075	14	root	0.0777	<0.01	<0.01	Batra, 1996g
						top[2]	2.58	0.0288	0.0367	

Location Year	Application				PHI, days	Sample	Residue, mg/kg			Reference
	Form.	No.	kg ai/ha	kg ai/hl			Fenbuc-onazole	RH-9129	RH-9130	
OH/USA 1995	WP	8	0.14	0.063	14	root	0.199	<0.01	<0.01	Batra, 1996g
						top[2]	1.23	0.0273	0.0258	
ID/USA 1995	WP	8	0.14	0.05	14	root	≤0.01	<0.01	<0.01	Batra, 1996g
						top[2]	4.97	0.0289	0.0279	
ID/USA 1995	SC	8	0.14	0.05	14	root	0.0266	<0.01	<0.01	Batra, 1996g
	WP	8	0.14	0.05	14	top[2]	4.45	0.036	0.0298	
TX/USA 1995	WP	8	0.14	0.06	15	root	0.0237	<0.01	<0.01	Batra, 1996g
						top[2]	1.38	0.0377	0.0171	
TX/USA 1995	WP	8	0.14	0.06	15	root	0.0197	<0.01	<0.01	Batra, 1996g
	SC	8	0.14	0.06	15	top[2]	2.55	0.0547	0.0257	
ND/USA 1995	WP	8	0.14	0.075	14	root	0.0738	<0.01	<0.01	Batra, 1996g
						top[2]	0.947	0.016	<0.01	
ND/USA 1995	SC	8	0.14	0.075	14	root	0.0434	<0.01	<0.01	Batra, 1996g
					14	top[2]	0.999	0.0157	<0.01	
ND/USA 1994	WP	8	0.14	0.075	14	root	0.089	<0.01	<0.01	Batra, 1996j
						wet pulp	0.038	<0.01	<0.01	
						dry pulp	0.481	<0.01	<0.01	
						molasses	0.162	<0.01	<0.01	
						refined sugar	≤0.01	<0.01	<0.01	
Italy 1991[3] Ferrara	EC	3	0.100 0.200	-	14 20 14 20	root root	0.03 0.08 0.08 0.05			Pessina 1991a
Italy 1991[3] Voghera	EC	3	0.100 0.200	-	14 21 21 14 21 21	root root leaves root root leaves	0.02 0.01 0.15 0.02 0.06 0.34			Pessina 1991l

[1]Samples stored for more than 6 months before analysis
[2]Top including crown
[3]Report was not in English

Wheat. GAP was reported for Belgium, France, Germany, Israel, Morocco, Portugal, South Africa and the UK, and pending GAP for the USA. The maximum application rates are 0.070–0.125 kg ai/ha with PHIs of 35–90 days or expressed as “before beginning of flowering growth stage 59”.

The residues in trials considered to comply with German, Portuguese and/or the pending US GAP are underlined in Table 63.

Table 63. Supervised residue trials on wheat in the USA.

Location Year	Application				PHI days	Sample	Residue, mg/kg				Reference
	Form.	No.	kg ai/ha	kg ai/hl			Fenbuc-onazole	RH-9129	RH-9130	RH-6467	
AR/USA 1987[1,2]	SC	2	0.0673	0.036	36	grain	<0.01	<0.01	<0.01	<0.01	Burnett, 1992f
						straw	4.37	0.314	0.0783	<0.01	
MS/USA 1987[1,2]	SC	2	0.067	0.0359	33	grain	<0.01	<0.01	<0.01	<0.01	Burnett, 1992f
						straw	0.554	0.142	0.0899	<0.05	
GA/USA 1987[1,2]	SC	3	0.0673	0.036	42	grain	<0.01	<0.01	<0.01	<0.01	Burnett, 1992f
						straw	1.053	0.239	0.0798	<0.05	
TX/USA 1988[1,2]	SC	3	0.0673	0.036	42	grain	<0.01	<0.01	<0.01	<0.01	Burnett, 1992f
TN/USA 1988[1,2]	SC	3	0.0673	0.036	37	grain	0.0111	<0.01	<0.01	<0.01	Burnett, 1992f
						straw	0.27	<0.05	<0.05	<0.05	
TN/USA 1988[1,2]	SC	3	0.14	0.075	37	grain	0.0388	<0.01	<0.01	<0.01	Burnett, 1992f
							0.791	<0.05	<0.05	<0.05	
PA/USA 1988[1,2]	SC	3	0.0673	0.024	41	grain	<0.01	<0.01	<0.01	<0.01	Burnett, 1992f
						straw	2	0.0545	<0.05	0.0263	
CA/USA 1989[1]	SC	3	0.071	0.038	37	grain	0.0154	≤0.01	≤0.01	≤0.01	Burnett, 1992f
						straw	1.94	0.0729	≤0.05	≤0.05	
CA/USA 1989[1]	SC	3	0.14	0.075	37	grain	<0.01	<0.01	<0.01	<0.01	Burnett, 1992f
						straw	4.38	0.154	0.06	<0.05	
ARUSA 1990[1,3]	SC	3	0.0706	0.0378	37	grain	≤0.01	≤0.01	≤0.01	≤0.01	Burnett, 1992f
						straw	0.7	0.122	0.0578	0.0205	
AR/USA 1990[1,3]	SC	3	0.14	0.075	37	grain	<0.01	<0.01	<0.01	<0.01	Burnett, 1992f
						straw	0.944	0.223	0.143	0.106	
FL/USA 1990[1,3]	SC	3	0.0706	0.0378	35	grain	≤0.01	≤0.01	≤0.01	≤0.01	Burnett, 1992f
							2.43	0.407	0.268	0.145	
FL/USA 1990[1,3]	SC	3	0.14	0.075	35	grain	<0.01	<0.01	<0.01	<0.01	Burnett, 1992f
							2.53	0.375	0.257	<0.05	
CA/USA 1990[1,3]	SC	3	0.0706	0.0378	32	grain	0.0105	≤0.01	≤0.01	≤0.01	Burnett, 1992f
							≤0.05	0.353	0.161	0.148	
MS/USA 1990[1,3]	SC	3	0.0706	0.0378	34	grain	≤0.01	≤0.01	≤0.01	≤0.01	Burnett, 1992f
						straw	0.774	0.141	0.106	≤0.05	
PA/USA 1990[1,3]	SC	3	0.0706	0.0315	30	grain	≤0.01	≤0.01	≤0.01	≤0.01	Burnett, 1992f
							≤0.05	0.104	0.0913	0.082	
OH/USA 1990[1,3]	SC	3	0.0706	0.0229	34	grain	≤0.01	≤0.01	≤0.01	≤0.01	Burnett, 1992f

Location Year	Application				PHI days	Sample	Residue, mg/kg				Reference
	Form.	No.	kg ai/ha	kg ai/hl			Fenbuc-onazole	RH-9129	RH-9130	RH-6467	
						straw	0.795	0.172	0.09	≤0.05	
OR/USA 1990[1,3]	SC	3	0.0695	0.0372	32	grain	≤0.01	≤0.01	≤0.01	≤0.01	Burnett, 1992f
						straw	1.44	0.122	≤0.05	≤0.05	
WA/USA 1990[1,3]	SC	3	0.0695	0.0372	32	grain	0.0118	≤0.01	≤0.01	≤0.01	Burnett, 1992f
							1.41	0.115	≤0.05	≤0.05	
VA/USA 1990[1,3]	SC	3	0.0706	0.028	30	grain	0.0164	≤0.01	≤0.01	≤0.01	Burnett, 1992f
						straw	0.76	0.107	≤0.05	≤0.05	
NC/USA 1990[1,3]	SC	3	0.0706	0.028	33	grain	≤0.01	≤0.01	≤0.01	≤0.01	Burnett, 1992f
						straw	1.55	0.18	0.0904	≤0.05	
TX/USA 1991[1,3]	SC	2	0.0706	0.0378	35	grain	0.0161	≤0.01	≤0.01	≤0.01	Burnett, 1992f
						straw	0.282	0.109	≤0.05	≤0.05	
TX/USA 1991[1,3]	SC	2	0.14	0.075	35	grain	0.0137	<0.01	<0.01	<0.01	Burnett, 1992f
						straw	0.788	0.24	0.108	<0.05	
TX/USA 1991[1,3]	SC	2	0.0706	0.0378	28	grain	0.0114	≤0.01	≤0.01	≤0.01	Burnett, 1992f
						straw	0.405	0.152	0.0593	≤0.05	
TX/USA 1991[1,3]	SC	2	0.14	0.075	28	grain	0.0296	0.0113	<0.01	<0.01	Burnett, 1992f
						straw	0.584	0.163	0.0689	<0.05	
OK/USA 1991[1,3]	SC	2	0.0706	0.0378	44	grain	≤0.01	≤0.01	≤0.01	≤0.01	Burnett, 1992f
						straw	0.569	0.0795	≤0.05	≤0.05	
OK/USA 1991[1,3]	SC	2	0.14	0.075	44	grain	0.0101	<0.01	<0.01	<0.01	Burnett, 1992f
						straw	1.15	0.128	0.052	<0.05	
NE/USA 1991[3]	SC	2	0.0706	0.0378	35	grain	≤0.01	≤0.01	≤0.01	≤0.01	Burnett, 1994d
						straw	0.115	0.06	≤0.05	≤0.05	
KS/USA 1991[3]	SC	2	0.0706	0.0338	37	grain	≤0.01	≤0.01	≤0.01	≤0.01	Burnett, 1994d
						straw	0.0825	≤0.05	≤0.05	≤0.05	
KS/USA 1991[3]	SC	2	0.0706	0.0344	32	grain	≤0.01	≤0.01	≤0.01	≤0.01	Burnett, 1994d
						straw	0.584	0.202	0.0965	≤0.05	
ND/USA 1991[3]	SC	2	0.0706	0.0504	49	grain	<0.01	<0.01	<0.01	<0.01	Burnett, 1994d
						straw	0.096	<0.05	<0.05	<0.05	
TX/USA 1993[3]	SC	2	0.0706	0.0378	40	grain	≤0.01	≤0.01	≤0.01	≤0.01	Batra, 1994d
						straw	0.225	0.063	≤0.05	≤0.05	
TX/USA 1993[3]	WP	2	0.0706	0.0378	40	grain	≤0.01	≤0.01	≤0.01	≤0.01	Batra, 1994d

Location Year	Application				PHI days	Sample	Residue, mg/kg				Reference
	Form.	No.	kg ai/ha	kg ai/hl			Fenbuconazole	RH-9129	RH-9130	RH-6467	
						straw	0.268	0.0715	≤0.05	≤0.05	
GA/USA 1994	SC	2	0.0706	0.0382	41	grain	≤0.01	≤0.01	≤0.01	≤0.01	Batra, 1994d
						straw	2.97	0.363	0.319	0.132	
GA/USA 1994	WP	2	0.0706	0.0382	41	grain	≤0.01	≤0.01	≤0.01	≤0.01	Batra, 1994d
						straw	4.45	0.493	0.378	0.189	
TX/USA 1995[3]	SC	3	0.0706	0.03	40	grain	≤0.01	<0.01[3]	≤0.01	RH-7905 =≤0.01	Batra, 1996h (foliar)
						straw	0.0975	<0.05[3]	≤0.05	--	
	SC	3	0.0706	0.03	40	grain	≤0.01	<0.01[3]	≤0.01	--	(seed treatment+ foliar)
						straw	0.11	0.36[3]	0.02	--	
GA/USA 1995[3]	SC	3	0.0706	0.038	40	grain	0.0067	≤0.01	≤0.01	RH-7905 ≤0.01	Batra, 1996h (foliar)
						straw	0.75	0.0615	0.051	--	
	SC	3	0.0706	0.038	40	grain	0.0054	≤0.01	0.0038	RH-7905 =<0.01	(seed treament+ foliar)
						straw	0.45	0.041	0.032	--	

[1]Results corrected for recoveries
[2]Samples stored for 3.5-4 years before analysis
[3]Samples stored for more than 6 months before analysis

The residues in trials considered to comply with German or Portuguese GAP are double underlined in Table 64, and those from trials according to UK GAP are underlined. Residues from trials complying with both German and UK GAP are in bold italics. Treatment at growth stages 55-63 was considered to conform to UK GAP.

Table 64. Supervised residue trials on wheat in Europe. All EC formulations.

Location, Year	Application			PHI, days	GS[1]	Sample	Residues, mg/kg			Ref./ Comment
	No.	kg ai/ha	kg ai/hl				Fenbuconazole	RH-9129	RH-9130	
UK[2], 1991 Dorset	2	0.075 0.094		54 54	51 51	grain grain	<0.02 <0.02	<0.02 <0.02	<0.02 <0.02	Orpin, 1991a
UK[2], 1991 Devon	2	0.075 0.094		84 84	51 51	grain grain	<0.02 <0.02	<0.02 <0.02	<0.02 <0.02	Orpin, 1991a
UK[2], 1991 Essex	2	0.075 0.094		60 60	51 51	grain grain	<0.02 <0.02	<0.02 <0.02	<0.02 <0.02	Orpin, 1991a
UK[2], 1991 Berks	2	0.075 0.094		77 77	50-60 50-60	grain grain	<0.02 <0.02	<0.02 <0.02	<0.02 <0.02	Orpin, 1991a
UK, 1991 Suffolk	2	0.075		56	58	grain	≤0.02	≤0.02	≤0.02	Orpin, 1991b
UK, 1991 Dorset	2	0.075		53	55	grain	≤0.02	≤0.02	≤0.02	Orpin, 1991b

Location, Year	Application			PHI, days	GS[1]	Sample	Residues, mg/kg			Ref./ Comment
	No.	kg ai/ha	kg ai/hl				Fenbuc-onazole	RH-9129	RH-9130	
UK, 1991	2	0.075		48	55-61	grain	≤0.02	≤0.02	≤0.02	Orpin, 1991b
Cambs	2	0.15		48		grain	<0.02	<0.02	<0.02	
UK[3] 1994	2	0.075		73	61	grain	≤0.02	≤0.02	≤0.02	Murray, 1994a
Tyneside				73		straw	0.85	0.28	0.18	
UK[3] 1994 Fife	2	0.075		62	66	grain	<0.02	<0.02	<0.02	Murray, 1994a
				62		straw	0.95	0.30	0.18	
UK[3] 1994 Warks	2	0.075		53	59-61	grain	≤0.02	≤0.02	≤0.02	Murray, 1994a
				53		straw	0.79	0.23	0.12	
Germany, 1993	2	0.075		35	61	grain	≤0.02	≤0.02	≤0.02	Agroplan
Goch-Niorswolde[4,5]				42		straw	1.01	0.20	0.06	Specht, 1993a
				42		grain	<0.02	<0.02	<0.02	52 day metabolite residues> 42 day residues.
				52		straw	0.75	0.26	0.09	
				52		grain	≤0.02	≤0.02	≤0.02	
Germany, 1993	2	0.075		35	61	grain	≤0.02	≤0.02	≤0.02	Agroplan
Celle[4,5]				42		straw	1.06	0.25	0.08	Specht, 1993c
				42		grain	<0.02	<0.02	<0.02	
				52		straw	1.26	0.34	0.10	
				52		grain	≤0.02	≤0.02	≤0.02	
Germany, 1992	2	0.075		42	61	straw	0.14	≤0.05	≤0.05	Specht, 1992c
Kron-prinzen-Koog[6]				42		grain	<0.02	<0.02	<0.02	
				49		straw	0.11	≤0.05	≤0.05	
				49		grain	≤0.02	≤0.02	≤0.02	
Germany, 1992	2	0.075		42	61	straw	0.51	0.40	0.15	Specht, 1992d
Marmstoft[6]				42		grain	<0.02	<0.02	<0.02	
				49		straw	0.39	0.30	0.11	
				49		grain	≤0.02	≤0.02	≤0.02	
Germany, 1992	2	0.075		42	61	straw	0.17	0.06	<0.05	Specht, 1992e
Borsum[6]				42		grain	0.06	0.02	<0.02	
Germany, 1992	2	0.075		42	58	straw	0.61	0.09	≤0.05	Specht, 1992g
Gruenstadt				55		straw	0.95	0.19	≤0.05	
				55		grain	≤0.02	≤0.02	≤0.02	
Germany, 1992	2	0.075		35	61/69	straw	2.46	0.24	0.07	Specht, 1992h
Soedenhof[6]				35		grain	<0.02	<0.02	<0.02	
				42		straw	2.35	0.21	0.07	
				42		grain	<0.02	<0.02	<0.02	
Germany, 1992	2	0.075		35	61	straw	0.84	0.20	0.08	Specht, 1992j
Christinenthal[6]				35		grain	<0.02	<0.02	<0.02	
				42		straw	1.05	0.28	0.09	
				42		grain	≤0.02	≤0.02	≤0.02	
Germany, 1992	2	0.075		35	61	straw	0.27	≤0.05	≤0.05	Specht, 1992k
Doersueben[6]				42		grain	<0.02	<0.02	<0.02	
				42		straw	0.89	0.15	0.12	
N France, 1993	2	0.075		49	-	grain	<0.02	<0.02	<0.02	Hede-Hauy, 1993c
Royer	2	0.075		49		grain	<0.02	<0.02	<0.02	
N France, 1993	2	0.075		66	50	grain	<0.02	<0.02	<0.02	Hede-Hauy, 1993d
Verneuil	2	0.075		66		grain	<0.02	<0.02	<0.02	

Location, Year	Application			PHI, days	GS[1]	Sample	Residues, mg/kg			Ref./ Comment
	No.	kg ai/ha	kg ai/hl				Fenbuc-onazole	RH-9129	RH-9130	
N France[5], 1993 Limours	2	0.075		55	50	grain	<0.02	<0.02	<0.02	
	2	0.075		55		grain	<0.02	<0.02	<0.02	Hede-Hauy, 1993e
N France, 1993[2]	2	0.075		64	50	grain	<0.02	<0.02	<0.02	
Fadainville	2	0.075		64		grain	<0.02	<0.02	<0.02	Hede-Hauy, 1993f
N. France[2], 1993	2	0.075		55	50	grain	<0.02	<0.02	<0.02	
Vovelles		0.075		55		grain	<0.02	<0.02	<0.02	Hede-Hauy, 1993g
N. France[2], 1993	2	0.075		68	50	grain	<0.02	<0.02	<0.02	
Oacques	2	0.075		68		grain	<0.02	<0.02	<0.02	Hede-Hauy, 1993h
Germany, 1993	2	0.075		35	61-65	straw	0.91	≤0.05	≤0.05	Agroplan
Klere[3,5]				35		grain	<0.02	<0.02	<0.02	Specht, 1993e
				42		straw	0.20	<0.05	<0.05	
				42		grain	<0.02	<0.02	<0.02	
Germany, 1993	2	0.075		35	61-65	straw	0.38	<0.05	≤0.05	Agroplan
Gosh-Nierswalde[3,5]				35		grain	≤0.02	≤0.02	<0.02	Specht, 1993k
				42		straw	0.41	0.07	<0.05	42 day residues> 35 day residues
				42		grain	<0.02	<0.02	<0.02	
S. France, 1989 Fontvielle	1	0.075	-	91	-	grain	<0.01	<0.01	<0.01	Vergne, 1989
S. France, 1989 Gimont	1	0.075	-	49	-	grain	≤0.01	≤0.01	≤0.01	Dumont, 1989
S. France, 1989 Estillac	1	0.075	-	47	-	grain	≤0.01	≤0.01	≤0.01	Niort, 1989
Israel[3,5], 1990	1	0.075		52	-	grain	≤0.01			Jewnin Joffe, 1991
Saad	1	0.125		52		grain	<0.01			
	2	0.075		52 +72		grain	<0.01			
	2	0.125		52 +72		grain	<0.01			
	1	0.075		72		grain	<0.01			
	1	0.125		72		grain	<0.01			
Portugal[2,6], 1990	1	0.075	-	84	-	grain	<0.02	<0.02	<0.02	Abela, 1990
Elvas	2	0.075		72		grain	<0.02	<0.02	<0.02	
Spain[2], 1990 Utera	1	0.075	-	49	-	grain	≤0.02	≤0.02	≤0.02	Jousseaume, 1990b
Spain[6] , 1990 Utera	1	0.075	-	49	-	grain	≤0.02	≤0.02	≤0.02	Jousseaume, 1990b
Spain, 1991	1	0.075	-	91	-	grain	<0.02	<0.02	<0.02	Jousseaume, 1991g
Sevilla	2	0.075		76		grain	<0.02	<0.02	<0.02	
Portugal[2], 1991 Beja	1	0.060	-	49	-	grain	≤0.02	≤0.02	≤0.02	Abela, 1991a
Portugal[2], 1991	1	0.075	-	98	-	grain	<0.02	<0.02	<0.02	
Beja				98		straw	0.38	0.06	<0.02	Abela, 1991b
	2	0.075	-	71		grain	<0.02	<0.02	<0.02	
				71		straw	2.89	0.28	0.09	
Portugal[2], 1991	1	0.075	-	85	-	grain	<0.02	<0.02	<0.02	
Vidigueria				85		straw	0.80	0.12	0.11	Abela, 1991c
	2	0.075	-	71		grain	<0.02	<0.02	<0.02	
				71		straw	1.59	0.12	0.16	

Location, Year	Application			PHI, days	GS[1]	Sample	Residues, mg/kg			Ref./ Comment
	No.	kg ai/ha	kg ai/hl				Fenbuc-onazole	RH-9129	RH-9130	
Portugal[2], 1991 Avis	1	0.075	-	80	-	grain	<0.02	<0.02	<0.02	Abela, 1991d
				80		straw	0.29	<0.02	<0.02	
	2	0.075	-	64		grain	<0.02	<0.02	<0.02	
				64		straw	0.54	0.07	0.03	
Spain[2], 1991 Brenes	1	0.075	-	74	-	grain	<0.02	<0.02	<0.02	Anadiag, 1993i
				74		straw	0.055	<0.02	<0.02	
	2	0.075	-	60		grain	<0.02	<0.02	<0.02	
				60		straw	0.052	<0.02	<0.02	

[1]Zadok growth stage at last treatment
[2]Samples stored for more than 6 months before analysis
[3]No recovery data with trial but acceptable recoveries (70-120%) from this commodity reported
[4]Method of analysis unspecified
[5]Duration of sample storage unspecified
[6]Report was not in English

Barley. GAP was reported for France, Germany, South Africa and the UK. Application rates are 0.072–0.125 kg ai/ha with PHIs of 35–45 days or, in the UK, "before beginning of flowering, growth stage 59".

The residues in trials considered to comply with German and UK GAP are underlined and double underlined respectively in Table 65, and those complying with both German and UK GAP are in bold italics. Treatment at growth stages 55-63 was considered to conform to UK GAP.

Table 65. Supervised residue trials on barley.

Location Year	Application			PHI days	GS[1]	Sample	Residue, mg/kg			Reference & Comment
	Form.	No.	kg ai/ha				Fenbuc-Onazole	RH-9129	RH-9130	
UK[2], 1991 Dorset	EC	2	0.075	62	-	straw	1.7	0.14	0.12	Orpin, 1991a
				62		grain	0.09	<0.02	<0.02	
UK[2], 1991 Devon	EC	2	0.094	62	-	straw	4.5	0.29	0.28	Orpin, 1991a
				62		grain	0.12	<0.02	<0.02	
UK[2], 1991 Essex	EC	3	0.075	36	-	straw	1.7	0.14	0.10	Orpin, 1991a
				36		grain	0.14	<0.02	<0.02	
UK[2], 1991 Berks	EC	2	0.075	55	-	straw	1.1	0.10	0.07	Orpin, 1991a
				55		grain	0.06	<0.02	<0.02	
			0.094	55		straw	0.86	0.08	<0.05	
				55		grain	0.04	<0.02	<0.02	
UK, 1991 Essex	EC	3	0.075	58	55	straw	0.67	0.05	<0.05	Orpin, 1991b
				58		grain	<0.02	<0.02	<0.02	
UK, 1991 Berks	EC	2	0.075	52	55	straw	2.4	0.19	0.13	Orpin, 1991b
				52		grain	0.04	<0.02	<0.02	
UK, 1991 Berks	EC	2	0.075	52	55	straw	2.2	0.14	0.11	Orpin, 1991b
				52		grain	<0.02	<0.02	<0.02	
UK, 1991 Berks	EC	2	0.075	52	55	straw	1.8	0.15	0.10	Orpin, 1991b
				52		grain	0.03	<0.02	<0.02	

Location Year	Application			PHI days	GS[1]	Sample	Residue, mg/kg			Reference & Comment
	Form.	No.	kg ai/ha				Fenbuc-Onazole	RH-9129	RH-9130	
UK, 1991	EC	2	0.150	52	55	straw	4.3	0.24	0.19	Orpin, 1991b
Berks				52		grain	0.05	<0.02	<0.02	
Germany[3], 1992	EC	2	0.075	35	61	grain	0.09	≤0.02	0.02	Agroplan
				42		straw	2.1	<0.05	<0.05	Specht, 1993b
Obernburg				42		grain	0.04	≤0.02	≤0.02	
Germany[3], 1992		2	0.075	42	61	straw	***2.07***	***<0.05***	***<0.05***	Agroplan
				35		grain	0.08	≤0.02	≤0.02	Specht, 1993d
Wuerzburg				42		grain	0.04	≤0.02	≤0.02	
Germany, 1990	EC	2	0.075	35	65	straw	0.25	≤0.05	≤0.05	Specht, 1992
				42			0.18	<0.05	<0.05	
Seedorf				35		grain	0.03	<0.02	<0.02	
				42			0.03	<0.02	<0.02	
Germany, 1990	EC	2	0.075	35	61	straw	0.21	≤0.05	≤0.05	Specht, 1992a
				42			0.17	≤0.05	≤0.05	
Wessenstedt				35		grain	0.03	<0.02	<0.02	
				42			0.02	≤0.02	≤0.02	
Germany, 1990	EC	2	0.075	35	61	straw	0.28	0.05	0.05	Specht, 1992b
				42			0.27	0.05	0.05	42 day parent residue >35 days residue
Borsum				35		grain	0.02	<0.02	<0.02	
				42			***0.03***	≤0.02	≤0.02	
Germany, 1990	EC	2	0.075	42	61	straw	0.68	≤0.05	≤0.05	Specht, 1992f
				65			1.13	0.13	≤0.05	
Kleinkarlbach				65		grain	0.04	≤0.02	≤0.02	
Germany, 1991	EC	2	0.075	35	61	straw	0.54	<0.05	≤0.05	Specht, 1992i
				42			***0.55***	***0.13***	≤0.05	Some day 42 residues > day 35 residues
Wettenbostel				35		grain	0.04	≤0.02	<0.02	
				42			≤0.02	≤0.02	≤0.02	
Germany, 1991 Fuerfeld	EC	2	0.075	34	61	straw	0.56	≤0.05	≤0.05	Specht, 1992l
				41			0.44	≤0.05	≤0.05	
				41		grain	***0.03***	***<0.02***	***<0.02***	
N. France, 1992	EC	2	0.072	58	earing	grain	<0.02	<0.02	<0.02	Schering, 1993h
				58		straw	0.36	0.03	0.03	
Bucy Le Roi				64		grain	<0.02	<0.02	<0.02	
				64		straw	0.07	<0.02	<0.02	
N. France, 1992	EC	2	0.072	58	49	grain	0.02	<0.02	<0.02	Schering, 1993f
Barberousse				58		straw	0.56	0.03	0.04	
N. France, 1992	EC	2	0.072	58	47	grain	<0.02	<0.02	<0.02	Schering, 1993g
Coulemelles				58		straw	0.45	<0.02	<0.02	
N. France, 1992 St Andre	EC	2	0.072	63	50	grain	<0.02	<0.02	<0.02	Hede-Hauy, 1993a
N. France, 1992 Bailleu	EC	2	0.072	63	50	grain	<0.02	<0.02	<0.02	Hede-Hauy, 1993b

Location Year	Application			PHI days	GS[1]	Sample	Residue, mg/kg			Reference & Comment
	Form.	No.	kg ai/ha				Fenbuc-Onazole	RH-9129	RH-9130	
N. France, 1992 Bucy Le Roi	SE	2	0.072	64 64	ear-ing	grain straw	<0.02 0.62	<0.02 0.06	<0.02 0.06	Schering, 1993a
N. France, 1992 Prey	SE	2	0.072	58 58	49	grain straw	0.03 1.43	<0.02 0.07	<0.02 0.07	Schering, 1993b
N. France, 1992 Coulemelles	SE	2	0.072	58 58	47	grain straw	<0.02 0.44	<0.02 <0.02	<0.02 <0.02	Schering, 1993c
N. France, 1992 St. Andre	SE	2	0.072	63	50	grain	<0.02	<0.02	<0.02	Hede-Hauy, 1993i
N. France, 1992 Bailleu	SE	2	0.072	63	50	grain	<0.02	<0.02	<0.02	Hede-Hauy, 1993j
N. France, 1992 St Just	EC	2	0.072	58 58	47	grain straw	<0.02 0.86	<0.02 0.07	<0.02 0.14	Schering, 1993d
N. France, 1992 St Just	SE	2	0.072	58 58	47	grain straw	<0.02 1.22	<0.02 0.10	<0.02 0.14	Schering, 1993e
UK[4], 1994 Fife	EC	2	0.075	57 57	57	straw grain	1.2 0.03	0.23 <0.02	0.14 0.02	Murray, 1994a
UK[4], 1994 Warks.	EC	2	0.075	46 46	59-65	straw grain	0.65 0.04	0.08 <0.02	0.07 <0.02	Murray, 1994a
UK, 1994 Fife	EC	2	0.075	55 55	57	straw grain	0.55 0.03	0.06 <0.02	≤0.05 <0.02	Murray, 1994a
Germany, 1993	EC	2	0.075	35 42 35 42	61-65	straw grain	1.7 1.9 0.05 0.04	≤0.05 <0.05 <0.02 <0.02	≤0.05 <0.05 <0.02 <0.02	Agroplan/ Specht, 1993f Parent day 42 straw residue >day 35
Germany[3], 1993 Kleve	EC	2	0.075	35 42 35 42	61-65	straw grain	0.35 0.22 <0.02 <0.02	≤0.05 <0.05 <0.02 <0.02	≤0.05 <0.05 <0.02 <0.02	Agroplan/ Specht, 1993g
Germany[3], 1993 Lusewitz	EC	2	0.075	42 42	61-65	straw grain	1.2 0.03	≤0.05 <0.02	≤0.05 <0.02	Agroplan/ Specht, 1993h

[1]Zadok growth stage at last treatment
[2]Samples stored for more than 6 months before analysis
[3]Duration of sample storage unspecified
[4]No recovery data with trial but acceptable recoveries (70-120%) from this commodity reported

Maize. Only pending GAP for France was reported, with an application rate of 0.075 kg ai/ha and a PHI of 45 days.

The residues in trials approximating pending French GAP are underlined in Table 66.

Table 66. Supervised residue trials on maize.

Location Year	Application			PHI days	Sample	Residue, mg/kg			Reference & Comment
	Form.	No.	kg ai/ha			Fenbuconazole	RH-9129	RH-9130	
S. France[1] 1990 Missanges	SE	2	0.060	27	Ears	0.02	<0.02	<0.02	Dapilly/Schering, 1990a
S. France[1] 1990 Liposthey	SE	2	0.060	27	Ears	0.02	<0.02	<0.02	Dapilly/Schering, 1990b
S. France[1] 1991 Moulon	SE	2	0.075	80	Ears	<0.02	<0.02	<0.02	Herisse, 1991e
S. France[1] 1991 Liposthey	SE	2	0.060	48	Whole plant Ears	0.26 <0.02	≤0.02 <0.02	≤0.02 <0.02	De Luis, 1991a
S. France[1] 1991 Liposthey	SE	2	0.075	48	Whole plant Ears	0.27 <0.02	≤0.02 <0.02	≤0.02 <0.02	De Luis, 1991a
S. France[1] 1991 Marenne	SE	2	0.075	43	Whole plant	0.12	≤0.02	0.03	De Luis, 199b
S. France[1] 1991 Marenne	SE	2	0.060	43	Whole plant	0.10	≤0.02	0.02	De Luis, 1991b
S. France[1] 1991 Labarie	SE	2	0.075	47	Whole plant Ears	0.26 <0.02	≤0.02 <0.02	0.06 <0.02	R71.30 De Luis, 1991c
S. France[1] 1991 Labarie	SE	2	0.060	47	Whole plant Ears	0.21 <0.02	≤0.02 <0.02	0.05 <0.02	R71.31 De Luis, 1991c
S. France 1993 Moulon	SE	2	0.075	76	Ears	<0.02	<0.02	<0.02	Herisse, 1993a
S. France 1994 Tosse	SE	2	0.075	33 33	Whole plant Ears	0.15 <0.02	≤0.02 <0.02	≤0.02 <0.02	Maigrot, 1994
S. France 1994 Moulon	SE	2	0.075	71 71	Whole plant Ears	0.22 <0.02	0.03 <0.02	0.02 <0.02	Herisse, 1994g

[1]Samples stored for more than 6 months before analysis

Rye. Only German GAP was reported, with an application rate of 0.075kg ai/ha and a PHI of 35 days.

The residues in trials considered to comply with German GAP are underlined in Table 67.

Table 67. Supervised residue trials on rye.

Location Year	Application				PHI days	Sample	Residue, mg/kg			Reference & Comment
	Form.	No.	kg ai/ha	kg ai/hl			Fenbuconazole	RH-9129	RH-9130	
Germany 1993 Eschau	EC	2	0.075		35 35 42 42	Straw Grain Straw Grain	1.1 ≤0.02 1.4 <0.02	0.12 ≤0.02 0.20 <0.02	<0.05 ≤0.02 0.05 <0.02	Agroplan Specht, 1993i Day 42 straw residues > day 35.
Germany 1993 Grob Lusewitz	EC	2	0.075		42 42	Straw Grain	0.49 0.03	0.09 <0.02	≤0.05 <0.02	Agroplan Specht,1993j

Triticale. No GAP was reported, but two trials were carried out in Germany (1993).

Table 68 Supervised residue trials on triticale, Germany, 1993.

Application			PHI days	Sample	Residue, mg/kg			Reference & Comment
No.	kg ai/ha	Kg ai/hl			Fenbuc-onazole	RH-9129	RH-9130	
2	0.075	0.025	35	Grain	<0.02	<0.02	<0.02	Agroplan Specht, 1993l
			35	Straw	0.14	<0.05	<0.05	
			42	Grain	<0.02	<0.02	<0.02	
			42	Straw	0.14	<0.05	<0.05	
2	0.075	0.025	42	Grain	<0.02	<0.02	<0.02	Agroplan Specht, 1993m
			42	Straw	0.27	<0.05	<0.05	

Almonds. GAP was reported for Israel and pending GAP for the USA with respective application rates of 0.004 kg ai/hl and 0.105 kg ai/ha and PHIs of 14 and 160 days.

Trials in the USA were reported. Residues from those according to the pending GAP are underlined in Table 69.

Table 69. Supervised residue trials on almonds, California, USA, 1987.

Application				PHI, days	Sample	Residue, mg/kg			Reference
Form.	No.	kg ai/ha	kg ai/hl			Fenbuc-onazole	RH-9129	RH-9130	
SC	3	0.11	0.0023	152	kernels	≤0.01	≤0.01	≤0.01	Burnett, 1989
					hulls	0.505	0.019	≤0.01	
SC	3	0.22	0.0045	152	kernels	<0.01	<0.01	<0.01	Burnett, 1989
					hulls	0.525	0.01	<0.01	
SC	3	0.11	0.0031	154	kernels	≤0.01	≤0.01	≤0.01	Burnett, 1989
					hulls	0.77	0.011	≤0.01	
SC	3	0.22	0.0062	154	kernels	<0.01	<0.01	<0.01	Burnett, 1989
					hulls	1.03	0.01	<0.01	
SC	3	0.11	0.0065	161	kernels	≤0.01	≤0.01	≤0.01	Burnett, 1989
					hulls	0.133	0.041	0.021	
SC	3	0.22	0.0129	161	kernels	<0.01	<0.01	<0.01	Burnett, 1989
					hulls	0.0644	0.151	0.011	
SC	3	0.11	0.0068	161	kernels	≤0.01	≤0.01	≤0.01	Burnett, 1989
					hulls	0.454	0.026	≤0.01	
SC	3	0.11	0.0039	200	Kernels	≤0.01	≤0.01	≤0.01	Burnett, 1989

The samples in all the trials were stored for more than 6 months before analysis CLICK HERE to continue

Pecans. GAP was reported only for the USA, at an application rate of 0.14 kg ai/ha with a PHI of 28 days.

The residues in trials considered to comply with US GAP are underlined in Table 70.

Table 70. Supervised residue trials on pecans, USA. Kernels analysed.

Location Year	Application				PHI, days	Residue, mg/kg				Reference & Comment
	Form.	No.	kg ai/ha	kg ai/hl		Fenbuconazole	RH-9129	RH-9130	RH-6467	
GA, 1990	SC	10	0.14	0.015	0	<0.01	<0.01	<0.01	<0.01	Burnett, 1991h
					7	<0.01	<0.01	<0.01	<0.01	
					14	<0.01	<0.01	<0.01	<0.01	
					28	≤0.01	≤0.01	≤0.01	≤0.01	
GA, 1990	SC	10	0.28	0.03	0	<0.01	<0.01	<0.01	<0.01	Burnett, 1991h
					7	<0.01	<0.01	<0.01	<0.01	
					14	<0.01	<0.01	<0.01	<0.01	
					28	<0.01	<0.01	<0.01	<0.01	
NM, 1990	SC	8	0.14	0.008	0	<0.01	<0.01	<0.01	<0.01	Burnett, 1991h
					7	<0.01	<0.01	<0.01	<0.01	
					14	<0.01	<0.01	<0.01	<0.01	
					28	≤0.01	≤0.01	≤0.01	≤0.01	
NM, 1990	SC	8	0.28	0.016	0	<0.01	<0.01	<0.01	<0.01	Burnett, 1991h
					7	<0.01	<0.01	<0.01	<0.01	
					14	<0.01	<0.01	<0.01	<0.01	
					28	<0.01	<0.01	<0.01	<0.01	
SC, 1990	SC	10	0.14	0.015	0	<0.01	<0.01	<0.01	<0.01	Burnett, 1991h
					7	<0.01	<0.01	<0.01	<0.01	
					14	<0.01	<0.01	<0.01	<0.01	
					28	≤0.01	≤0.01	≤0.01	≤0.01	
SC, 1990	SC	10	0.28	0.03	0	<0.01	<0.01	<0.01	<0.01	Burnett, 1991h
					7	<0.01	<0.01	<0.01	<0.01	
					14	<0.01	<0.01	<0.01	<0.01	
					28	<0.01	<0.01	<0.01	<0.01	
TX, 1990	SC	10	0.14	0.015	0	<0.01	<0.01	<0.01	<0.01	Burnett, 1991h
					7	<0.01	<0.01	<0.01	<0.01	
					14	<0.01	<0.01	<0.01	<0.01	
					28	≤0.01	≤0.01	≤0.01	≤0.01	
TX, 1990	SC	10	0.28	0.03	0	<0.01	<0.01	<0.01	<0.01	Burnett, 1991h
					7	<0.01	<0.01	<0.01	<0.01	
					14	<0.01	<0.01	<0.01	<0.01	
					28	<0.01	<0.01	<0.01	<0.01	
GA, 1994[1]	SC	9	0.14	0.0163	39	≤0.01	≤0.01	≤0.01	≤0.01	Batra, 1996i

Location Year	Application				PHI, days	Residue, mg/kg				Reference & Comment
	Form.	No.	kg ai/ha	kg ai/hl		Fenbuc-onazole	RH-9129	RH-9130	RH-6467	
	WP	9	0.14	0.0163	39	≤0.01	≤0.01	≤0.01	≤0.01	
LA, 1994	SC	9	0.146	0.0166	47	<0.01	<0.01	<0.01	<0.01	Batra, 1996i
	WP	9	0.146	0.0166	47	<0.01	<0.01	<0.01	<0.01	
NM, 1994	SC	11	0.146	0.0104	52	<0.01	<0.01	<0.01	<0.01	Batra, 1996i
	WP	11	0.0146	0.0104	52	<0.01	<0.01	<0.01	<0.01	
OK, 1994	SC	8	0.146	0.0124	37	≤0.01	≤0.01	≤0.01	≤0.01	Batra, 1996i
	WP	8	0.146	0.0124	37	≤0.01	≤0.01	≤0.01	≤0.01	
TX, 1994[1]	SC	10	0.146	0.0159	40	≤0.01	≤0.01	≤0.01	≤0.01	Batra, 1996i
	WP	10	0.146	0.0159	40	≤0.01	≤0.01	≤0.01	≤0.01	

[1]Samples stored for more than 6 months before analysis

A report of a further trial on pecans in the form of a "laboratory memo" had insufficient detail for evaluation (Stavinski, 1994b).

Oilseed rape. GAP was reported only for France, where there are alternative treatments of an application of 0.060 kg ai/ha with a PHI of 30 days and an application of 0.075kg ai/ha with a PHI of 45 days.

The residues in trials according to French GAP are underlined in Table 71.

Table 71. Supervised residue trials on oilseed rape, France, 1990. Seed analysed.

Location	Application				PHI days	Fenbuc-onazole	Reference
	Form.	No.	kg ai/ha	kg ai/hl			
St Mayo	SE	2		0.075	46	<0.05	Dapilly, Schering, 1990c
Chuelles	SE	1		0.075	98	<0.05	Dapilly, Schering, 1990d
Spoy	SE	1		0.075	78	<0.05	Dapilly, Schering, 1990e
Richarville	SE	2		0.075	59	<0.05	Dapilly, Schering, 1990f

Sunflower. Again only French GAP was reported. The application rate is either 0.060 or 0.075 kg ai/ha, depending on the product, with PHI of 80 days in both cases.

The residues in trials considered to comply with French GAP are underlined in Table 72. Residues in the two trials with a shorter PHI (34 days) were <0.02 mg/kg.

Table 72. Supervised residue trials on sunflowers in Southern France. Seed analysed.

Location Year	Application				PHI, days	Residue, mg/kg			Reference
	Form.	No.	kg ai/ha	kg ai/hl		Fenbuc-onazole	RH-9129	RH-9130	
Moulon, 1989	EC	2	0.075	-	86	≤0.01	≤0.01	≤0.01	Herisse, 1989d
1990	SE	2	0.075	-	34	<0.02	<0.02	<0.02	Herisse, 1990d

Location Year	Application				PHI, days	Residue, mg/kg			Reference
	Form.	No.	kg ai/ha	kg ai/hl		Fenbuc-onazole	RH-9129	RH-9130	
Moulon, 1990	EC	2	0.075	-	34	<0.02	<0.02	<0.02	Herisse, 1990d
Moulon, 1991	EC	2	0.056	-	60	≤0.02	≤0.02	≤0.02	Herisse, 1991c
1991	SE	2	0.075	-	93	≤0.02	≤0.02	≤0.02	De Luis, 1991d
1991, Bois	SE	2	0.06 0.08	-	93 93	≤0.02 <0.02	≤0.02 <0.02	≤0.02 <0.02	De Luis, 1991d
Sainte Sixte, 1991	SE	2	0.06	-	74	≤0.02	≤0.02	≤0.02	De Luis, 1991e
Caude-coste, 1991	SE	2	0.06	-	84	≤0.02	≤0.02	≤0.02	De Luis, 1991f

Livestock feeding trials

Nine lactating dairy cows each received 28 daily doses of fenbuconazole at rates equivalent to 6.5, 19.5 or 65 ppm in the feed, with three controls. On day 28, all the cows were slaughtered except four which were allowed a three-day recovery period and slaughtered on day 31. Recoveries were determined concurrently with the sample analyses and were satisfactory for milk, muscle, fat, liver and kidneys. Samples were stored frozen for up to 4 months before analysis by the methods of Filchner *et al*. (1992) and Filchner and Stavinksi (1991). The residues in all the samples from two untreated cows were below the limit of determination (0.01 mg/kg for milk and muscle and 0.05 mg/kg for liver, fat and kidneys).

Milk and muscle were analysed for residues of fenbuconazole, the lactones and iminolactones, and kidneys, liver, and fat also for RH-7968. No residues were found in the kidneys at any dose level. Only one sample of milk during the dosing period contained a measurable residue of 0.019 mg/kg after four days, from a cow dosed at the equivalent of 19.5 ppm in the feed. Residues in muscle were not determinable except one at 0.01 and one at 0.02 mg/kg. The only residue in the fat was 0.06 mg/kg in one sample from the 65 ppm group. Residues in the liver samples were up to 0.09 mg/kg in the 6.5 ppm group, 0.20 mg/kg at 19.5 ppm and 0.68 mg/kg at 65 ppm. The residues found in the milk and tissues are shown in Tables 73 and 74 (Chen *et al*., 1992b).

Table 73. Residues in the milk of cows dosed with fenbuconazole.

Dose group	Cow No.	Residue[1], mg/kg, at day									
		-1	1	4	7	10	14	21	24	28	31
Control	1	NQR[2]	NQR	NQR	NQR	NQR	NQR	NQR	NQR	NQR	
	6	NQR	NQR	NQR	NQR	NQR	NQR	NQR	NQR	NQR	
	8	NQR	NQR	NQR	NQR	NQR	NQR	NQR	NQR	NQR	NQR
6.5 ppm	9	NQR	NQR	NQR	NQR	NQR	NQR	NQR	NQR	NQR	
	11	NQR	NQR	NQR	NQR	NQR	NQR	NQR	NQR	NQR	
	12	NQR	NQR	NQR	NQR	NQR	NQR	NQR	NQR	NQR	NQR
19.5 ppm	2	NQR	NQR	0.019	NQR	NQR	NQR	NQR	NQR	NQR	
	5	0.010	NQR	NQR	NQR	NQR	NQR	NQR	NQR	NQR	
	7	NQR	NQR	NQR	NQR	NQR	NQR	NQR	NQR	NQR	NQR
65 ppm	4	NQR	NQR	NQR	NQR	NQR	NQR	NQR	NQR	NQR	

Dose group	Cow No.	Residue[1], mg/kg, at day									
		-1	1	4	7	10	14	21	24	28	31
	10	NQR	NQR	NQR	NQR	NQR	NQR	NQR	NQR	NQR	
	3	NQR	NQR	NQR	NQR	NQR	NQR	NQR	NQR	NQR	NQR

[1]Sum of fenbuconazole, RH-9130, RH-9129, and RH-6468
[2]NQR = no quantifiable residue at the limit of determination (0.010 ppm)

Table 73. Residues in the tissues of cows dosed with fenbuconazole.

Dose group	Cow No.	Residue[1], mg.kg in			
		Muscle	Fat	Kidneys	Liver
Control	1	NQR[3]	NQR	NQR	NQR
	6	NQR	NQR	NQR	NQR
	8[2]	NQR	NQR	NQR	NQR
6.5 ppm	9	0.01	NQR	NQR	0.09
	11	NQR	NQR	NQR	0.06
	12[2]	NQR	NQR	NQR	0.04
19.5 ppm	2	NQR	NQR	NQR	0.15
	5	NQR	NQR	NQR	0.20
	7[2]	NQR	NQR	NQR	0.10
65 ppm	4	NQR	NQR	NQR	0.55
	10	0.02	0.06	NQR	0.68
	3[2]	NQR	NQR	NQR	0.14

[1]Sum of fenbuconazole, RH-9130, RH-9129, and RH-6468 in muscle, sum of fenbuconazole, RH-7592, RH-9130, RH-9129, RH-6468, and RH-7968 in fat, kidneys and liver
[2]Slaughtered on day 31
[3]No quantifiable residue at the limit of determination (0.010 mg/kg for RH-7592, RH-9130, RH-9129, RH-6468, 0.05 mg/kg for RH-7968)

Hens. Four groups of 10 laying hens were dosed orally once daily by capsule with fenbuconazole equivalent to 0, 0.12, 0.34 and 1.13 ppm in the feed for 28 consecutive days after a quarantine period in 1991. Eggs were collected from each group twice daily. After slaughter, samples of liver, thigh and breast muscle, and abdominal and subcutaneous fat were bulked from three, three, and four birds of each group. Eggs, muscle, and fat were analysed for fenbuconazole, the lactones, and the iminolactones. Liver was also analysed for RH-7968. Recoveries determined concurrently with the sample analyses were satisfactory. Samples were stored frozen up to 3.5 months before analysis. The residues in all samples from the untreated and treated hens were below the limits of determination of 0.01 mg/kg in eggs and muscle and 0.05 mg/kg in liver and fat (Chen *et al*., 1992a.).

FATE OF RESIDUES IN STORAGE AND PROCESSING

In storage

No data.

In processing

Limited data on processing were included with the residue trials on apples, grapes, tomatoes and sugar beet.

In addition the following processing data were submitted.

Wheat. A processing study was carried out in 1990 in the USA. In order to obtain significant residues in the grain wheat was treated four times, at rates of 0.071 and 0.14 kg ai/ha, with the last treatment 7 days before harvest. Only fenbuconazole was present above the limit of determination, at 0.104 mg/kg from the lower application rate and 0.22 mg/kg from the higher. The results are shown in Table 75.

Table 75. Residues in wheat and its processed fractions.

Sample	Fenbuconazole, mg/kg		Mean processing factors
	0.071 kg ai/ha	0.14 kg ai/ha	
Cleaned grain	0.104	0.221	
Bread	0.039	0.122	0.46
Patent bread	0.024	0.116	0.38
Flour	0.017	0.073	0.25
Red dog	0.048	0.139	0.55
Bran	0.262	0.596	2.6
Shorts/germ	0.185	0.30	1.57
Middlings	0.042	0.12	0.47

In another study in the USA wheat was treated twice at an application rate of 0.71 kg ai/ha. The total residue in the grain at harvest was 0.016 mg/kg. No residues were detected in the bran, shorts, middlings, low-grade flour, patent flour or bread. The limit of determination was 0.01 mg/kg (Burnett, 1992g; Burnett and Martin, 1992; Burnett and Wu, 1994).

Grapes. Field-treated grapes were vinified on a pilot-plant scale in a research faculty. Samples were analysed by the method of Martin (1990). Residues of RH-9129 and RH-9130 were below the 0.004 mg/kg limit of detection (the validated limit of quantification was 0.01 mg/kg). Acceptable chromatograms were submitted and procedural recoveries were satisfactory (Gilbert, 1997).

Table 76. Summary of grape processing data.

Country, year	Form.	No.	kg ai/ha	kg ai/hl	PHI days	Sample	Fenbuc-onazole	RH-9129	RH-9130	Reference & comment
Germany 1996 Niederkirchen	EC	8	0.037-0.06	0.01125	14	Whole fruit	0.50	<0.01	<0.01	Gilbert, 1997
						Young wine	<0.01	<0.01	<0.01	
						Mature wine	<0.01	<0.01	<0.01	
						Juice	0.05	<0.01	<0.01	
Germany 1996 Niederkirchen	EC	8	0.015-0.06	0.00375	14	Whole fruit	0.52	<0.01	<0.01	Gilbert, 1997
						Young wine	<0.01	<0.01	<0.01	
						Mature wine	<0.01	<0.01	<0.01	
						Juice	0.05	<0.01	<0.01	

Peaches. Processed samples were derived from field-treated peaches processed with commercial equipment. The inspection-belt fruit sample was taken after washing the peaches in a bath of 1-3% NaOH at 88-93°C, peeling, and passing through an abrasive wash reel. Samples were analysed by the method of Martin (1990). The residues of RH-9129 and RH-9130 were below the limit of determination of 0.01 mg/kg. Acceptable chromatograms were submitted, and procedural recoveries were satisfactory (Batra, 1996k).

Table 77. Summary of peach processing data.

Country, year	Form.	No.	kg ai/ha	kg ai/hl	PHI days	Sample	Fenbuconazole	RH-9129	RH-9130	Reference
USA 1995 S. Carolina	WP	1	0.105	0.0112	17	Whole fruit Inspection belt fruit Purée	0.056 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	Batra, 1996k

Table 78. Summary of processing factors from the processing studies.

Commodity	Residue levels of fenbuconazole, mg/kg									Mean processing factors
Apple	0.066									
Wet pomace	0.152									2.3
Unpasteurised juice	0.0038									0.06
Pasteurised juice	<0.01									<0.15
Grape	0.04	0.23	1.89	0.09	0.04	0.05	0.05	0.5	0.52	
Must				<0.02	<0.02	0.02	<0.01			- [a]
Wine	0.008	0.034	0.09	<0.02	<0.02	<0.01	<0.01	<0.01	<0.01	0.06 [a]
Juice								0.05	0.05	0.10
Tomato	0.05	0.10								
Tomato juice	0.06	0.02								- [b]
Tomato preserves	0.06	0.03								- [b]
Tomato purée	0.17	0.11								- [b]
Sugar beet-root	0.089									
Wet pulp	0.039									0.43
Dry pulp	0.481									5.4
Molasses	0.162									1.8
Refined sugar	<0.01									0.11
Peach	0.056									
Purée	<0.01									0.18
Wheat										
Cleaned grain		0.104	0.221							
Bread		0.039	0.122							0.46
Flour		0.017	0.073							0.25
Bran		0.262	0.596							2.6

[a] Only initial grape residues of >0.1mg/kg have been used in calculating the processing factor
[b] In this single laboratory study the initial residues were low and the results are difficult to interpret

Residues in the edible portion of food commodities

The residues in the pulp of apples, bananas, grapefruit and oranges are shown in the Tables of residues found in supervised trials. Residues in apple juice and wine are shown in Tables.76 and 78.

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

No data were submitted.

NATIONAL MAXIMUM RESIDUE LIMITS

There are no harmonised European Union MRLs for fenbuconazole. MRLs in individual member states are shown in the Table below:

National MRLs for fenbuconazole in member states of the European Union (Orpin, 1997b,e).

All commodities	Netherlands	"Zero tolerance" using analytical detection limit between 0.02 and 0.05mg/kg
Apples	Italy France	0.2 0.1
Banana	Germany	0.05
Barley	France Germany	0.02 0.1
Cucumber	Spain	0.05
Grapes	France Spain Italy	0.3 0.5 0.2
Melons, except watermelon	Spain Italy France	0.02 0.2 0.3
Pears	Italy France	0.2 0.1
Pepper	France	0.1
Rape seed	France	0.02
Rye	Germany	0.1
Squash, Summer	Spain	0.05
Sugar beet	Italy	0.2
Sunflower seed	France	0.02
Tomatoes	France	0.3
Triticale	Germany	0.1
Watermelon	Spain	0.02
Wheat	Germany France Belgium	0.1 0.02 0.02

The only other reported MRLs were for the USA as shown below.

MRLs for fenbuconazole in the USA (Orpin, 1997e).

Commodity	MRL, mg/kg
Grapefruit	1 (P)
Orange	1 (P)
Apples	0.4 (P)
Apricots	2
Cherries	2
Nectarine	2
Peach	2
Plum/prune (fresh)	2
Banana, whole fruit	3

pulp	0.05
Sugar beet	0.2 (P)
Wheat	0.05 (P)
Almond, kernels hulls	0.05 (P) 3 (P)
Pecan	0.1

P: provisional

APPRAISAL

Fenbuconazole is a white solid with low solubility in water and low vapour pressure. It has a log P_{OW} of 3.22 and is reasonably soluble in fat. It is a triazole fungicide formulated mainly as an EC or an EW.

Fenbuconazole was rapidly absorbed and eliminated, mainly in the faeces with significant biliary excretion, in rats; there was no evidence of any retention in the tissues. The compound was also extensively metabolized with oxidation and hydroxylation at a number of sites in the molecule, followed by conjugation to form sulfates and glucuronides, mainly the latter.

Metabolism and distribution were investigated in lactating goats and chickens. In goats dosed at levels equivalent to 1, 10 and 100 ppm in the feed, less than 0.5% of the TRR remained in the milk and less than 1.6% in the tissues. Two major metabolites were identified in the milk as 1,2,4-triazole (0.24 mg/kg fenbuconazole equivalents at the highest dose) and triazolylalanine (TA, 0.15 mg/kg at the highest dose), with very low levels of the parent compound. At the highest dose, the levels of the TRR in the liver, kidney, muscle and fat were 12.4, 0.97, 0.22 and 0.11 mg/kg respectively. In the liver five major components were identified: the parent compound (0.95 mg/kg), 4-(4-chlorophenyl)-2-hydroxymethyl-2-(phenyl)butanenitrile (RH-7968, 0.95 mg/kg), the glucuronide of the 4-hydroxy derivative (1.23 mg/kg), the triazole (1.79 mg/kg) and TA (4.95 mg/kg). A further phenol metabolite was present in the kidney.

When chickens were dosed with the equivalent of 100 ppm in the feed, less than 0.7% of the TRR was found in the eggs and less than 0.8% in the tissues (0.04% in the lean meat, 0.01% in the fat, 0.02% in the liver and 0.02% in the kidneys). Three major components were identified in the eggs: the parent (0.89 mg/kg), two isomeric lactones (0.29 mg/kg) and the triazole (0.54 mg/kg). In the liver the glucuronide (3.69 mg/kg) and the triazole (1.25 mg/kg) were the major metabolites. In the fat the parent was the major component (0.43 mg/kg) with several low-level metabolites identified.

Plant metabolism was studied in peaches, wheat and peanuts. Metabolism occurs by oxidation at the benzylic carbon adjacent to the chlorophenyl ring.

In the wheat study, phenyl- and triazole-labelled [^{14}C]fenbuconazole were foliar-applied at a rate of 2 x 0.4 kg ai/ha. At harvest the total ^{14}C residues (expressed as fenbuconazole equivalent) were 0.04-0.44 mg/kg in the grain and 9.8-10.6 mg/kg in the straw. The predominant components in the grain were TA and triazolylacetic acid (TAA) which were present at levels of 0.25 and 0.11 mg/kg respectively. The parent compound was present in the grain, but at less than 0.01 mg/kg. In the straw fenbuconazole was the main component at levels of 8.8-11.8 mg/kg. Three other components were identified in the straw as the lactones found in chickens (1.1-1.4 mg/kg), a ketone (the 4-oxo derivative, 0.59-0.62 mg/kg) and a glucoside conjugate of the 4-(4-chloro-3-hydroxyphenyl) analogue (0.43 mg/kg).

In the peach study, phenyl- and triazole-labelled [^{14}C]fenbuconazole were foliar-applied five times, at a rate of 0.2 kg ai/ha. At harvest the total ^{14}C residues in the peaches expressed as fenbuconazole equivalent were 0.08-0.12 mg/kg. TA and fenbuconazole predominated, at levels of 0.06 and 0.02-0.04 mg/kg respectively. The lactones and TAA were also identified, plus 5 unknowns which were not individually above 0.01 mg/kg.

In the third study, peanuts were treated four times with phenyl- and triazole-labelled [^{14}C]fenbuconazole at a rate of 0.57 kg ai/ha. The major component in both the vine and the shells was fenbuconazole (48 and 34% respectively), with TA, the glucoside conjugate, and the ketone the main remaining components. In the nut no fenbuconazole was present and 92% of the residue was TA.

In a study of metabolism and distribution in rotational crops, wheat, turnips and collards were planted in soil which had been treated bare with either phenyl- or triazole-labelled fenbuconazole at the exaggerated rate of 8.96 kg ai/ha. At harvest TA and TAA were by far the predominant components of the TRR in the crops. In a further study, phenyl-labelled fenbuconazole was applied to soil at 3 x 0.07 or 4 x 0.28 kg ai/ha and lettuce, radishes, sorghum, carrots or barley were planted 35-260 days after treatment. The total residues in all crop samples at harvest were ≤0.04 mg/kg.

The lactones and ketone formed in plants were identified in the rat metabolism studies; TA and TAA were not identified in rats but are common metabolites of all triazoles. In several of the residue trials the levels of the lactones and ketone were determined but were generally at low concentrations compared with fenbuconazole.

Fenbuconazole was found to be persistent in soil, although degradation varied greatly, with half-lives of 38-367 days in the laboratory and 28-425 days (DT90 >1 year) in the field. The compound was immobile in column leaching studies and showed K_{oc} values of 2185-9042. Neither photolysis nor hydrolysis of fenbuconazole occurred in aqueous media, and in sediment/water systems it partitioned rapidly into the sediment where it persisted.

Several methods of analysis were reported for various commodities, most of them by GLC with NP detection. Most of the methods also allow determination of metabolites (e.g. the lactones) and have limits of determination of 0.01-0.05 mg/kg with recoveries of about 80%. The Meeting agreed that 0.05 mg/kg was an appropriate practical limit of determination of parent fenbuconazole in most commodities for routine monitoring and the enforcement of MRLs.

Residues of fenbuconazole and the lactones, the ketone in plant products and the 2-hydroxymethyl derivative in animal products were stable in apples, wheat grain and straw stored at -10° and products of animal origin stored at -4°Cfor at least 18 months and 2-4 months respectively. Residues of fenbuconazole and the lactones (and the ketone in some commodities) were also stable when stored at -10°C for 54 months in peaches and pecans, 12 months in almonds, and about 31 months in hen and cow muscle. There was some decrease in total fenbuconazole residues in some cereal fractions that had been stored for about 56 months.

The Meeting agreed that the residue should be defined as fenbuconazole both for compliance with MRLs and the estimation of dietary intake.

The Meeting concluded that although the solubility of the residue in fat was intermediate the enforcement of MRLs could best be carried out on a whole-product basis; this conclusion is supported by the data on animal metabolism. Accordingly the Meeting agreed not to describe the residue as fat-soluble.

Supervised trials

In listing the residue results, values at or above 0.1 mg/kg have been given to two significant figures and those below 0.1 mg/kg to one significant figure.

Grapefruit and oranges. The only reported GAP was pending in the USA where the maximum application rate for both commodities is 0.28 kg ai/ha with a PHI of 0 days. Residues of fenbuconazole from trials on grapefruit complying with this GAP were 0.02, 0.10, 0.12, 0.13, 0.16, 0.16, 0.19, 0.34 and 0.49 mg/kg in the whole fruit and <0.01 (5) and 0.02 mg/kg in the pulp.

The residues in oranges were 0.18 (2), 0.19 (2), 0.28, 0.30, 0.34, 0.44 and 0.52 mg/kg in the whole fruit and <0.01 (4) and 0.01 mg/kg in the pulp. Because GAP was only pending the Meeting was unable to estimate a maximum residue level for either fruit.

Pome fruit. GAP for apples was reported for France, Israel, Italy, Portugal, South Africa, Turkey and the UK and for pears for Greece, Israel, Italy, Portugal, South Africa and the UK, and was reported to be pending for apples in the USA and Greece and for pears in France. The maximum application concentrations are 0.002-0.004 kg ai/hl except in the UK and the USA where the application rates were reported as 0.068 and 0.14 kg ai/ha respectively. PHIs were either 14 or 28 days.

Residues of fenbuconazole in apples from trials complying with the pending US GAP were 0.01, 0.02, 0.04, 0.05 (2), 0.06 (2), 0.07 (2), 0.08, 0.09 (2), 0.12 (4), 0.13 (2), 0.15, 0.16 (2), 0.17, 0.18, 0.20 (2), 0.27 and 0.28 mg/kg. Many of the results from these trials had been corrected for the average recovery. Because the GAP was pending the Meeting could not use these results to estimate a maximum residue level.

Residues of fenbuconazole in apples from trials according to UK GAP were <0.02, 0.02 (3), 0.03 (3), 0.04 (2), 0.05 and 0.06 mg/kg, those from trials complying with Southern European GAP (France, Greece, Italy and Portugal) were <0.005, <0.01, 0.01, 0.02 and 0.03 mg/kg, and the residues in pears from trials complying with Southern European GAP were 0.01 (2), 0.02 (2), 0.05 and 0.06 mg/kg.

The residues from the trials on apples and pears according to GAP appear to be from similar data populations. The combined residues were <0.005, <0.01, <0.02, 0.01 (3), 0.02 (6), 0.03 (4), 0.04 (2), 0.05 (2) and 0.06 (2) mg/kg. The Meeting estimated a maximum residue level of 0.1 mg/kg and an STMR of 0.025 mg/kg for fenbuconazole in pome fruit.

Cherries. GAP was reported only for the USA. The maximum application is 0.105 kg ai/ha with a PHI of 0 days.

The residues in the trials complying with the maximum US GAP were 0.20, 0.21, 0.31, 0.33, 0.34, 0.36 (2), 0.42 and 0.51 mg/kg. Four other trials with an exaggerated application rate (33% higher than GAP) showed residues of 0.43, 0.47, 0.53 and 0.55 mg/kg at day 0. In addition, trials at the maximum US application rate with residues of <0.01, 0.12, 0.22, 0.25, 0.43 and 0.47 mg/kg at a PHI of 7 days were considered to be within the range of GAP. All of the above residues at day 0 had been corrected for average recoveries. Some of the trial samples were stored frozen for 3.5 to 4 years before analysis. Since the data on the storage stability of fenbuconazole residues in peaches indicated that they were stable up to 54 months the Meeting agreed to use these results, but emphasized that the storage of trial samples for long periods before analysis was undesirable.

The Meeting agreed that an STMR should be estimated from the residues on day 0, including those from the exaggerated rate: 0.20, 0.21, 0.31, 0.33, 0.34, 0.36 (2), 0.42, 0.43, 0.47, 0.51, 0.53 and 0.55 mg/kg. The Meeting estimated a maximum residue level of 1 mg/kg and an STMR of 0.36 mg/kg for fenbuconazole in cherries.

Apricots and peaches. GAP for apricots was reported for Israel and the USA, and pending GAP for France. The maximum application rates are 0.0025 kg ai/hl in Israel, 0.0075 kg ai/hl in France, and 0.105 kg ai/ha in the USA, with PHIs of 0-14 days.

The residues in the trials on apricots considered to comply with US GAP were 0.12, 0.16, 0.21, 0.25 and 0.27 mg/kg. All of these results had been corrected for average recoveries and referred to the residue in the fruit without stone. The trials complying with the pending French GAP with the highest application rate (0.0075 kg ai/hl) gave residues of 0.06, 0.17, 0.26, 0.21 and 0.33 mg/kg. Some of these were in the fruit without stone.

GAP for peaches was reported for Israel and the USA, and pending GAP for France and South Africa. The maximum application rates are 0.002-0.005 kg ai/hl or 0.105 kg ai/ha, with PHIs ranging from 0 to 14 or 60 days: the pending French GAP was originally reported by the company as having a 60-day PHI, but the Meeting was informed that the PHI would be 3 days. Based on a 3-day PHI the residues in trials complying with the pending French GAP were 0.07 (3), 0.09, 0.10 (2), 0.11, 0.13 and 0.21 mg/kg. Some of these results were for the fruit without stone. The residues in the trials considered to comply with US GAP were 0.19, 0.25 (2), 0.28, 0.37, 0.46 and 0.51 mg/kg, corrected for average recoveries, in the fruit without stone.

The Meeting agreed that the US residues in apricots and peaches were mutually supportive and could be combined, giving residues in rank order of 0.12, 0.16, 0.19, 0.21, 0.25 (3), 0.27, 0.28, 0.37, 0.46 and 0.51 mg/kg. The Meeting noted that these results had been corrected for recovery and that the residues were in the fruit without stone, and estimated maximum residue levels of 0.5 mg/kg and STMRs of 0.25 mg/kg for fenbuconazole in peaches and apricots.

Plums (including prunes). GAP was originally reported for Israel and the USA, with pending GAP for France, but the Meeting was informed that the US GAP was actually pending. The maximum application rates are 0.002-0.0075 kg ai/hl in Israel and France and 0.105 kg ai/ha in the USA. PHIs are 0-14 days.

The residues in trials considered to comply with the pending French GAP were 0.05, 0.06, 0.07, 0.16, 0.20, 0.23, 0.27, 0.30, 0.36 and 0.38 mg/kg, and those in the trials complying with the pending US GAP were <0.01, 0.01, 0.02, 0.03 (2), 0.04 (2), 0.06 and 0.07 mg/kg. Three further residues were reported in dried prunes: 0.08, 0.14 and 0.16 mg/kg. All of the US results had been corrected for average recoveries and the residue was in the fruit without stone. As both the French and US GAP is pending, the Meeting could not estimate a maximum residue level.

Grapes. GAP was reported for France, Israel, Italy, Portugal, Spain and Turkey, and pending GAP for Greece. The maximum application rates are 0.002-0.0075 kg ai/hl or 0.03-0.04 kg ai/ha, with PHIs of 7-28 days.

The residues in the trials considered to comply with Italian and pending Greek GAP were 0.04 (2), 0.05 (2) and 0.17 mg/kg and those in the trials complying with French and Spanish GAP were 0.02, 0.05, 0.10, 0.12, 0.16, 0.2, 0.3 (3), 0.35, 0.4 (2) and 0.5 mg/kg. Since the French product labels specified a rate of 0.03-0.0375 kg ai/ha, the Meeting agreed that it was not appropriate to use four German trials in which the application rates were 0.056-0.075 kg ai/ha in the evaluation.

The Meeting estimated a maximum residue level of 1 mg/kg and an STMR of 0.3 mg/kg for fenbuconazole in grapes, based on the trials complying with French and Spanish GAP. Although the highest residue in the trials was only 0.5 mg/kg, the Meeting noted that there were several residues close to 0.5 mg/kg and that the median residue was relatively high.

Strawberries. GAP was reported for Israel. The maximum application rate is 0.075 kg ai/ha with a PHI of 14 days.

Only one trial was considered to comply with Israeli GAP, with a residue of 0.17 mg/kg. Although additional data were available from Spain, the climatic and agricultural practices were not considered to be comparable to those in Israel and these data have not been used in the evaluation. There were therefore insufficient data to estimate a maximum residue level.

Bananas. GAP was reported for Columbia, Costa Rica, Ecuador, Guatemala, Honduras, Mexico, Panama, Venezuela, Philippines and the USA. GAP in all of these countries was the same with a maximum application rate of 0.105 kg ai/ha and a PHI of 0 days.

The residues in the trials with bagged fruit considered to comply with GAP were <0.01 (4) and 0.01 mg/kg in pulp, <0.01 (4) and 0.03 mg/kg in peel, and <0.01 (3) mg/kg in whole fruit. Those in the trials with unbagged fruit which complied with GAP were <0.01 and 0.02 mg/kg in pulp, 0.09 mg/kg in peel, and <0.01 (3), 0.01 and 0.02 mg/kg in whole fruit. The Meeting estimated a maximum residue level of 0.05 mg/kg and an STMR of 0.01 mg/kg for bananas. Although only 6 trials were reported for bagged bananas, the Meeting considered that there were sufficient data to estimate a maximum residue level since all the residues were well below the practical limit of determination of 0.05 mg/kg.

Melons (except watermelons). GAP for melons was reported for France, Israel, Italy, Portugal and Turkey, and pending GAP for Spain and Morocco. The maximum application rates are 0.0375-0.2 kg ai/ha or 0.005-0.01 kg ai/hl. PHIs are 3-7 days. The Meeting was informed that the PHI in France was 7 days and not the 3 days reported in the original company submission.

The residues in trials considered to comply with Italian GAP were <0.005, 0.009, 0.02 and 0.05 mg/kg, and those in the trials complying with French GAP with a 7-day PHI were <0.02, 0.02 (2), 0.03, 0.07, 0.09, 0.1 and 0.13 mg/kg. The residues all appear to be within the same population and can be combined, giving <0.005, 0.009, <0.02, 0.02 (3), 0.03, 0.05, 0.07, 0.09, 0.1 and 0.13 mg/kg. The Meeting estimated a maximum residue level of 0.2 mg/kg and an STMR of 0.025 mg/kg for melons.

Watermelons. GAP was reported for Israel, Italy, Portugal, Spain and Turkey, and pending GAP for Morocco. The maximum application rates are 0.0375-0.2 kg ai/ha or 0.005 -0.01 kg ai/hl, with PHIs of 3 or 7 days. There is no French GAP for watermelons.

Only one trial was considered to comply with Italian GAP, with a residue of <0.005 mg/kg. There were insufficient data to estimate a maximum residue level.

Cucumbers. GAP was reported for Israel, Spain and Turkey, and pending GAP for France and Morocco. The maximum application rates are 0.0375-0.1 kg ai/ha or 0.005-0.01 kg ai/hl, with PHIs of 3 or 7 days.

One Spanish and two Italian field trials, all with residues of 0.02 mg/kg, and five indoor trials in Spain and Greece with residues of <0.01, 0.02, 0.03 (2) and 0.11 mg/kg were considered to comply with Spanish GAP. One trial in Israel complied with Israeli indoor GAP with a residue of 0.1 mg/kg.

The Meeting concluded that the residues in all the trials according to GAP were in a single population and agreed to combine them to give <0.01, 0.02 (4), 0.03 (2), 0.1 and 0.11

mg/kg. The Meeting estimated a maximum residue level of 0.2 mg/kg and an STMR of 0.02 mg/kg for cucumbers.

Summer squash (courgettes, zucchini). GAP was reported for Israel, Spain and Turkey, and pending GAP for France and Morocco. The maximum application rates are 0.0375-0.2 kg ai/ha or 0.005-0.01 kg ai/hl with PHIs of 3 or 7 days.

The residues in trials considered to comply with the pending French GAP were <0.02, 0.03, 0.03 and 0.08 mg/kg; the trial with the residue of 0.03 mg/kg also complied with Turkish GAP. The residues from trials complying with the pending Moroccan GAP were <0.02, 0.03, 0.04, 0.04, 0.06 and 0.08 mg/kg. Because French and Moroccan GAP is pending the Meeting could not use the results to estimate a maximum residue level.

The outdoor trials considered to comply with Spanish GAP showed residues of <0.01 (2), 0.01, <0.02 (3) and 0.02 mg/kg. Some of these trials were also considered to comply with Israeli GAP. The Meeting estimated a maximum residue level of 0.05 mg/kg and an STMR of 0.02 mg/kg for summer squash, based on Spanish GAP.

Tomatoes. GAP for Israel was reported as 0.0075 kg ai/hl (glasshouse) and 0.05 kg ai/ha (field) with PHIs of 7 days in both cases. Pending GAP for Morocco is 0.01 kg ai/hl with a PHI of 3 days for both field and glasshouse applications.

The residues in trials considered to comply with the pending Moroccan GAP were 0.02, 0.03 (2), 0.05 (2), 0.08, 0.10, 0.13, 0.14, 0.16, 0.18, 0.31 and 0.38 mg/kg, but as the GAP was pending the Meeting could not use them to estimate a maximum residue level.

The residues in trials which complied with Israeli glasshouse GAP were 0.08, 0.19 and 0.21 mg/kg and in those complying with the field GAP <0.02 and 0.02 (2) mg/kg.

The Meeting agreed that the Israeli glasshouse use appeared to lead to higher residues than the field use. Since only three trials were available for each of these uses, the Meeting concluded that there were insufficient data to estimate a maximum residue level.

Peppers. GAP was reported for Israel (field use only), and pending GAP for Morocco. The maximum application rates are 0.0075 and 0.01 kg ai/hl, with PHIs of 7 and 3 days respectively.

The residues in field trials which complied with the pending Moroccan GAP were 0.02 and 0.10 mg/kg and in glasshouse trials 0.18, 0.20, 0.29, 0.38 and 0.41 mg/kg. The glasshouse use appeared to lead to higher residues, but as the GAP was pending the Meeting could not use the results to estimate a maximum residue level.

Two trials in Italy and Spain complied with Israeli GAP, but the Meeting agreed that the climatic conditions and agricultural practices in Italy and Spain could not be equated with those in Israel. There was therefore no basis on which the Meeting could estimate a maximum residue level.

Egg plant. GAP was reported only for Morocco, and only one residue trial was reported which complied with it. There were insufficient data to estimate a maximum residue level.

Sugar beet. GAP was reported for Italy and pending GAP for the USA. The maximum application rates are 0.1 and 0.14 kg ai/ha with PHIs of 14 days.

The residues in the trials which complied with US GAP were <0.01, 0.02 (3), 0.03 (3), 0.04 (4), 0.06, 0.07 (2), 0.08, 0.09 and 0.20 mg/kg in the roots and 0.51, 0.55, 0.80, 0.85, 0.95, 1.0, 1.2, 1.2, 1.4, 2.6 (2), 3.1, 4.2, 4.5, 5.0 and 8.9 mg/kg in the tops. As the GAP was pending the Meeting could not estimate a maximum residue level from these trials.

Only two trials complied with Italian GAP, with residues in the roots of 0.02 and 0.03 mg/kg. There were insufficient data to estimate a maximum residue level.

Wheat. GAP was reported for Belgium, France, Germany, Israel, Morocco, Portugal, South Africa and the UK, and pending GAP for the USA. The maximum application rates are 0.07-0.125 kg ai/ha with PHIs of 35-90 days or expressed as "before beginning of flowering growth stage 59".

The residues in trials which complied with the pending US GAP were 0.005, 0.007, <0.01 (17), 0.01 (3), 0.02 (3) mg/kg in the grain and <0.05 (2), 0.08, 0.10, 0.11, 0.12, 0.23, 0.27, 0.28, 0.41, 0.45, 0.57, 0.58, 0.70, 0.75, 0.76, 0.77, 0.80, 1.4 (2), 1.6, 1.9, 2.4, 3.0 and 4.5 mg/kg in the straw. A number of other US trials complied with the pending GAP, but the samples were stored for 3.5-4 years before analysis. Since data on storage stability indicated that fenbuconazole residues were stable in wheat for at least 36 months the Meeting agreed to regard these results as valid, but emphasized that the storage of trial samples for long periods before analysis was undesirable. As the GAP was pending however the results could not be used to estimate a maximum residue level.

The residues in trials which complied with German GAP were <0.02 (9) and 0.06 mg/kg in the grain and 0.14, 0.17, 0.27, 0.41, 0.51, 0.61, 0.84, 0.91, 1.0, 1.3 and 2.5 mg/kg in the straw.

The residues in trials which complied with Portuguese GAP were <0.01 (3) and <0.02 (3) mg/kg in the grain. The straw was not analysed.

The residues in trials which complied with UK GAP were <0.02 (5), <0.02* (7) and 0.06* mg/kg in the grain and 0.11*, 0.17*, 0.39*, 0.75*, 0.79, 0.85, 0.89*, 0.95*, 1.05* and 1.26* mg/kg in the straw. The residues marked with an asterisk were from trials which also complied with German GAP.

The Meeting agreed that the residues in the trials according to German, UK and Portuguese GAP appeared to be from the same population of data and could be combined to give <0.01 (3), <0.02 (17) and 0.06 mg/kg in the grain and 0.14, 0.17, 0.27, 0.41, 0.51, 0.61, 0.79, 0.84, 0.85, 0.91, 1.0, 1.3 and 2.5 mg/kg in the straw. The Meeting estimated maximum residue levels of 0.1 mg/kg for wheat grain and 3 mg/kg for straw, and STMRs of 0.02 mg/kg for grain and 0.79 mg/kg for straw.

Barley. GAP was reported for France, Germany, South Africa and the UK. The maximum application rates are 0.072-0.125 kg ai/ha with PHIs of 35-45 days or expressed as "before beginning of flowering growth stage 59".

The residues in trials which complied with German GAP were <0.02, 0.03 (5), 0.04, 0.05, 0.08, 0.09 and 0.14 mg/kg in the grain, and 0.21, 0.25, 0.28, 0.35, 0.55, 0.56, 0.68, 1.2, 1.7, 1.9 and 2.1 (2) mg/kg in the straw. Those from trials complying with UK GAP were <0.02 (2), <0.02*, 0.02*, 0.03 (3), 0.03* (2), 0.04 and 0.04* (3) mg/kg in the grain and 0.17*, 0.27*, 0.44*, 0.55, 0.55*, 0.67, 1.13*, 1.2, 1.8, 2.1*, 2.07*, 2.2 and 2.4 mg/kg in the straw. The residues marked with an asterisk were from trials which also complied with German GAP.

The Meeting agreed that the results of the UK and German trials could be combined to give <0.02 (3), 0.03 (8), 0.04 (2), 0.05, 0.08, 0.09 and 0.14 mg/kg in the grain and 0.21, 0.25, 0.28, 0.35, 0.55 (2), 0.56, 0.67, 0.68, 1.2 (2), 1.7, 1.8, 1.9, 2.1 (2), 2.2 and 2.4 mg/kg in the straw. The Meeting estimated maximum residue levels of 0.2 mg/kg for barley grain and 3 mg/kg for straw, and STMRs of 0.03 mg/kg for grain an 0.94 mg/kg for straw.

Maize. GAP was reported to be pending in France, with an application rate of 0.075 kg ai/ha and a PHI of 45 days.

The residues in trials which complied with the pending French GAP were <0.02 (5) mg/kg in maize ears and 0.10, 0.12, 0.15 0.21, 0.26 (2) and 0.27 mg/kg in the fodder. As the GAP was pending the Meeting could not estimate a maximum residue level.

Rye. GAP was reported for Germany, with an application rate of 0.075 kg ai/ha and a PHI of 35 days.

The residues in trials which complied with German GAP were <0.02 and 0.03 mg/kg in the grain, and 0.49 and 1.4 mg/kg in the straw. The Meeting concluded that the residues in wheat grain, resulting from similar GAP, could be used to support those in rye and estimated a maximum residue level of 0.1 mg/kg and an STMR of 0.02 mg/kg for rye grain.

Triticale. Two trials were reported, but there was no information on GAP.

Almonds. GAP was reported for Israel and pending GAP for the USA, with application rates of 0.004 kg ai/hl and 0.105 kg ai/ha and PHIs of 160 and 14 days.

The residues in trials which complied with the pending US GAP were <0.01 (5) in the kernels and 0.13, 0.45, 0.51 and 0.77 mg/kg in the hulls. As the GAP was pending the Meeting could not estimate a maximum residue level or an STMR for almonds or almond hulls.

Pecans. GAP was reported for the USA, with an application rate of 0.14 kg ai/ha and a PHI of 28 days.

The residues in ten trials which complied with US GAP were all <0.01 mg/kg in pecan kernels. The Meeting estimated a maximum residue level of 0.05* mg/kg and an STMR of 0.01 mg/kg for pecans.

Oilseed. GAP for sunflowers was reported only for France. The application rate is either 0.060 or 0.075 kg ai/ha depending on the product, with a PHI of 80 days.

The residues in the seed in trials on sunflowers which complied with French GAP were <0.01 and <0.02 (5) mg/kg. Residues in two further trials with a shorter PHI (34 days) were all <0.02 mg/kg. The Meeting estimated a maximum residue level of 0.05* mg/kg and an STMR of 0.02 mg/kg for sunflower seed.

GAP for rape was also reported only for France, with an application rate of 0.060 kg ai/ha and a PHI of 30 days or 0.075 kg ai/ha and a PHI of 45 days.

The residues in two trials on rape which complied with French GAP were both <0.05 mg/kg. In two other trials with longer PHIs the residues were also <0.05 mg/kg. The Meeting took into account the data on sunflower seed in which no measurable residues were found and estimated a maximum residue level of 0.05* mg/kg and an STMR of 0.05 mg/kg for rape seed.

Animal products. Animal transfer studies were carried out on dairy cattle and hens. Cattle dosed at a level equivalent to 6.5 ppm in the feed, showed total fenbuconazole residues of 0.01 mg/kg in one sample of muscle and up to 0.09 mg/kg in 3 samples of liver. No quantifiable residues were found in the milk, fat or kidney. At a dose level equivalent to 19.5 ppm in the feed the only residues were 0.02 mg/kg in one sample of milk and 0.1-0.2 mg/kg in three samples of liver.

In hens dosed at the equivalent of 0.12, 0.34 or 1.13 ppm in the feed, all the residues in eggs and tissues were below the limit of determination.

The highest residues in fodder crops from trials which complied with GAP were 4.5 mg/kg in wheat straw, 2.1 mg/kg in barley straw, 1.4 mg/kg in rye straw, 0.8 mg/kg in almond hulls and <0.05 mg/kg in rape seed.

Assuming maximum incorporation rates of straw (the feed item with the most significant residues) of 20 and 50% for dairy and beef cattle respectively, the maximum feed intakes will be approximately 1 ppm and 2.5 ppm in the diet. Residues would be expected to be below a limit of determination of 0.05* mg/kg in all cattle products except liver. The Meeting estimated maximum residue levels of 0.05* mg/kg for cattle meat, cattle fat, cattle milk and cattle kidney and 0.05 mg/kg for cattle liver. The Meeting agreed that the STMR should be zero for those cattle commodities in which no measurable residues were found at the equivalent of 6.5 ppm or 19.5 ppm in the diet in the cow feeding studies. Accordingly, the Meeting estimated STMRs of 0.01 mg/kg for cattle meat, milk and liver and 0 mg/kg for cattle kidney and fat.

In poultry, the highest residues would arise from barley grain in which residues were <0.02-0.14 mg/kg. Since cereal grains can constitute up to 70% of the diet the maximum feed intakes will be approximately 0.1 ppm in the diet. The Meeting estimated a maximum residue level of 0.05* mg/kg for poultry fats, poultry meat, edible offal of poultry, and eggs. The Meeting agreed that the STMR should be zero for those poultry commodities in which no measurable residues were found at the equivalent of 1.13 ppm in the diet in the poultry feeding studies. Accordingly, the Meeting estimated an STMR of 0 mg/kg for poultry fats, meat, edible offals, and eggs.

The Meeting agreed not to estimate STMR-Ps for the processed products of apples, peaches or sugar beet since only single samples had been processed on a laboratory scale and the initial residues were low. The Meeting estimated STMR-Ps for wine and grape juice of 0.018 mg/kg (0.3 x 0.06) and 0.03 mg/kg (0.3 x 0.1) respectively. The Meeting also estimated STMR-Ps for bread, flour and bran of 0.0092 mg/kg (0.02 x 0.46), 0.005 mg/kg (0.02 x 0.25) and 0.052 mg/kg (0.02 x 2.6) respectively.

No monitoring data were provided but national MRLs were reported for the USA and several European countries.

Before MRLs can be recommended for the commodities for which GAP is pending confirmation that each proposed GAP has been registered will be required, together with copies of the product labels.

RECOMMENDATIONS

The residue levels shown below are recommended for use as MRLs:

Definition of the residue for compliance with MRL and the estimation of dietary intake: fenbuconazole

Commodity		Recommended MRL, mg/kg	STMR, mg/kg	PHI on which proposal based
CCN	Name			
FP 0009	Pome fruits	0.1	0.025	28
FS 0013	Cherry	1	0.36	0
FS 0240	Apricot	0.5	0.25	0
FS 0247	Peach	0.5	0.25	0
FB 0269	Grapes	1	0.3	21
FI 0327	Banana	0.05	0.01	0
VC 0046	Melon, except watermelon	0.2	0.025	3
VC 0424	Cucumber	0.2	0.025	7
VC 0431	Summer squash	0.05	0.02	7
GC 0654	Wheat	0.1	0.02	35-42
AS 0654	Wheat straw and fodder, dry	3	0.61	35-42
GC 0640	Barley	0.2	0.03	35
AS 0640	Barley straw and fodder, dry	3	0.62	35
GC 0650	Rye	0.1	0.02	35
TN 0672	Pecans	0.05*	0.01	28
SO 0495	Rape seed	0.05*	0.05	30-45
SO 0702	Sunflower seed	0.05*	0.02	80
MM 0812	Cattle meat	0.05*	0.01	N/A
MF 0812	Cattle fat	0.05*	0	N/A
MO 1280	Cattle kidney	0.05*	0	N/A
ML 0812	Cattle milk	0.05*	0.01	N/A
MO 1281	Cattle liver	0.05	0.01	N/A
PF 0111	Poultry fats	0.05*	0	N/A
PM 0110	Poultry meat	0.05*	0	N/A
PM 0111	Poultry, edible offals of	0.05*	0	N/A
PE 0112	Eggs	0.05*	0	N/A
	Wine (from grape)	-	0.018 STMR-P	N/A
	Grape juice	-	0.03 STMR-P	N/A
	Bread	-	0.046 STMR-P	N/A
	Flour	-	0.025 STMR-P	N/A
	Bran	-	0.26 STMR-P	N/A

FURTHER WORK OR INFORMATION

Desirable

1. The method of analysis used for the determination of fenbuconazole in soil and water in the studies of environmental fate.

2. Data on residues in food in commerce or at consumption (i.e. monitoring or total diet data).

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FOLPET

EXPLANATION

Folpet was first evaluated in 1969 and has been reviewed several times since, most recently in 1993 and 1994 for residues.

The 1990 JMPR required, by 1992, results of supervised trials on apples, cherries, cucumbers, grapes, bulb onions, strawberries and tomatoes, as well as current information on 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, raspberries 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 residue studies on citrus fruits, lettuce, melons and potatoes were in progress and that data would be available for the 1994 JMPR. The CCPR decided to maintain CXLs as temporary for all commodities. The 25th Session was informed that the manufacturer had provided information for all commodities with temporary MRLs except cherries and onions (ALINORM 93/24A, para 66).

The 1995 CCPR decided to delete the CXLs for apple, cherries, citrus fruits, head lettuce, melons except watermelon, bulb onion and tomato (ALINORM 95/24A, para 94).

The 28th (1996) Session of the CCPR was informed that data on cucumbers and strawberries as well as on those commodities whose CXLs were deleted in 1995 would be ready for evaluation by the 1997 JMPR, and decided to keep the MRL for cucumbers at Step 3 and to advance the MRL for strawberry to Step 7B (ALINORM 97/24, para 42). The manufacturer confirmed the availability of data on apples, cucumbers, lettuce, melons, onions, strawberries and tomatoes.

The basic manufacturer provided information to the Meeting on metabolism, analytical methods, freezer storage stability, registered uses, data from supervised trials on fruit and vegetable crops, and processing studies. Information on GAP and summary reports of supervised trials were provided by Germany.

METABOLISM AND ENVIRONMENTAL FATE

Plant metabolism

The Meeting received information on the metabolism of folpet in tomato plants, winter wheat, grapes and avocados.

Cheng (1980) treated the roots of tomato plants (7 weeks old) with 4 mg/l [*carbonyl*-^{14}C]folpet in a nutrient solution containing 0.25% acetone, and harvested the plants for analysis 1, 4, 7 and 11 days after treatment. Each plant received 25 ml of the treatment solution while under a growth lamp.

Methanol/water extracts of the tomato roots and tops were examined by TLC for ^{14}C compounds. One day after treatment about 85% of the ^{14}C in the nutrient solution was absorbed into the plants and about 60% of the absorbed ^{14}C was translocated to the tops. By the eleventh day 93% of the ^{14}C had been absorbed from the nutrient solution and of this 90% was in the tops. Folpet itself was a very minor constituent of the residue in the plant.

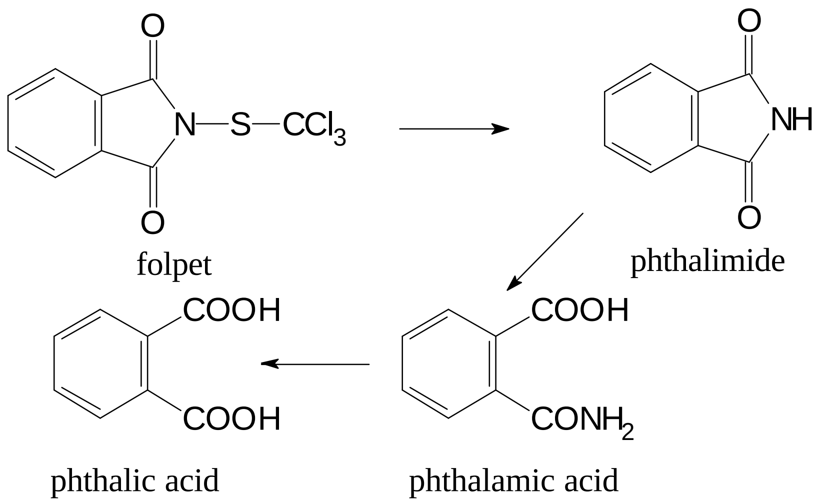
Table 1. Metabolites in tomato plants exposed through the roots to a nutrient solution containing [carbonyl-14C]folpet (Cheng, 1980).

Compound	Compound as % of extractable ^{14}C in roots or tops							
	Day 1		Day 4		Day 7		Day 11	
	Top	Root	Top	Root	Top	Root	Top	Root
Folpet	<0.1	0.2	<0.1	0.1	<0.1	<0.1	<0.1	<0.1
Phthalimide	5.9	1.7	5.4	2.1	2.9	1.9	3.4	1.4
Phthalic acid + phthalamic acid	76	93	67	93	68	93	63	91
Unidentified[1]	15	2.3	25	2.8	26	3.0	30	5.1

[1]Three polar metabolites, possibly ring-hydroxylated phthalamic acid derivatives.

The Rf values of phthalic acid and phthalamic acid were too close for the compounds to be separated by TLC for quantitative measurement, but about 90% of the ^{14}C was estimated to be phthalamic acid from an autoradiogram.

Figure 1. Folpet metabolism in tomato plants.



Crowe (1995) applied [*benzene*-^{14}C]folpet to winter wheat plants twice at a rate equivalent to 1.6 kg ai/ha and sampled the plants 1 day after each application, at maturity, and at harvest, when the ages of the plants were 190, 214, 258 and 269 days respectively.

The levels of ^{14}C were lower in the roots than the straw or grain at each sampling. The plant parts were not washed before measurements were made, so surface residues are included. The recovery of the ^{14}C in the extracts and unextracted residues was high, particularly for straw and grain. Levels of ^{14}C were higher in the harvested crop because the plants had begun to dry out. The composition of the extractable residue is shown in Table 2.

Day	Total ^{14}C as folpet, mg/kg.		
	roots	straw	grain
191	0.03	4.5	3.2
215	0.23	9.4	7.5
258	0.63	13	10
269	0.74	15	24

Treatment of the extracted straw from day 269 with 1M HCl to release bound residues released phthalic acid (1 mg/kg).

Table 2. Composition of the extractable residue in winter wheat straw and grain from plants treated with [*benzene*-^{14}C]folpet at 1.6 kg ai/ha on days 190 and 214 (Crowe, 1995).

Compound	^{14}C as parent or metabolite, mg/kg							
	Day 191		Day 215		Day 258		Day 269	
	Straw	Grain	Straw	Grain	Straw	Grain	Straw	Grain
Folpet	3.5	1.8	4.7	4.8	6.9	4.7	4.7	9.3
Phthalic acid	NDR	NDR	NDR	NDR	0.60	0.57	4.3	6.4
Phthalimide	0.41	0.80	0.98	1.2	0.76	0.98	1.5	3.1
Polar metab					0.43	0.49		
Unknown						0.29		

NDR: no detectable residues

Folpet itself was the major component of the residue in all cases, but in the final stage the levels of phthalic acid + phthalimide exceeded those of folpet. Phthalamic acid was not mentioned in this study.

Mester (1994a) made 3 foliar applications of [*benzene*-^{14}C]folpet at 1-month intervals to Thomson Seedless grape vines, equivalent to 1.5 kg ai/ha at each application, and harvested grapes and leaves 25 days after the final application for analysis and identification of metabolites by O'Connor (1994). Less than 1% of the ^{14}C in the grapes or leaves remained after washing and water/acetonitrile extraction. The water/acetonitrile extract was further divided into dichloromethane- and water-soluble fractions. The disposition of the radiolabel is shown in Table 3.

Table 3. Distribution of radiolabel in rinses and extracts from grapes and leaves of vines treated with 3×1.5 kg ai/ha [*benzene*-^{14}C]folpet and harvested 25 days after the final application (O'Connor, 1994).

Fraction	Grapes		Leaves	
	^{14}C as % of total	^{14}C as folpet, mg/kg	^{14}C as % of total	^{14}C as folpet, mg/kg
Rinse	26	2.0	87.8	258
Organosoluble	19	1.4	6.5	19
Water-soluble	54	4.1	4.6	14
Unextracted	1.5	0.11	1.1	3.2
TOTAL	100.5	7.6	100.0	294

The identities of the components in the rinses and extracts are shown in Table 4. Folpet, phthalic acid and phthalimide constituted 27%, 5.8% and 11% respectively of the residue on the grapes. An unidentified compound in the water-soluble fraction accounted for 41% of the residue. HPLC showed that it was very polar; it was eluted with the solvent front on a reversed phase system. Attempts to identify the material by MS and various combinations of HPLC-MS were not successful. Since acid hydrolysis yielded phthalic acid the material was identified as phthalic acid conjugate(s). Phthalamic acid was not considered as a possible metabolite.

Table 4. Components of the residue on grapes and leaves of vines treated with 3 × 1.5 kg ai/ha [*benzene*-^{14}C]folpet and harvested 25 days after the final application (O'Connor, 1994).

Compound	Residue in grapes expressed as folpet, mg/kg			Residue in leaves expressed as folpet, mg/kg		
	Rinse	Organosoluble	Water-soluble	Rinse	Organosoluble	Water-soluble
Folpet	1.1	0.97		251	15	
Phthalic acid	0.16	0.28			2.2	4.8
Phthalimide	0.74	0.07		7.2	1.6	
Unidentified 1		0.11				
Unidentified 2			3.1			
Unidentified 3						6.7
Unidentified 4						2.0

Mester (1994b) sprayed a small avocado tree in California three times at 21-day intervals with [*benzene*-^{14}C]folpet at the equivalent of 3.4 kg ai/ha for each application, and harvested fruit and leaves 21 and 97 days after the final application for analysis and identification of metabolites by Toia and Collins (1994). The fruit harvested at 97 days were mature.

After aqueous rinsing to release surface residues the samples were thoroughly extracted with ethyl acetate. The distribution of radiolabel in the fruit and leaves is shown in Table 5. The components in the rinses and extracts were identified by TLC and HPLC with the results shown in Table 6.

Table 5. Distribution of radiolabel in rinses and extracts of avocado fruit and leaves from a tree treated with 3×3.4 kg ai/ha [*benzene*-^{14}C]folpet and harvested 21 and 97 days after the final application (Toia and Collins, 1994).

Fraction	^{14}C as folpet, mg/kg			
	Fruit		Leaves	
	21 days	97 days	21 days	97 days
Rinse	0.70	0.014	48	21
Ethyl acetate extract	8.8	14 (peel) 7.5 (pulp)	68	37
Residue after ethyl acetate extract	1.4	3.2 (peel) 0.66 (pulp)	20	15

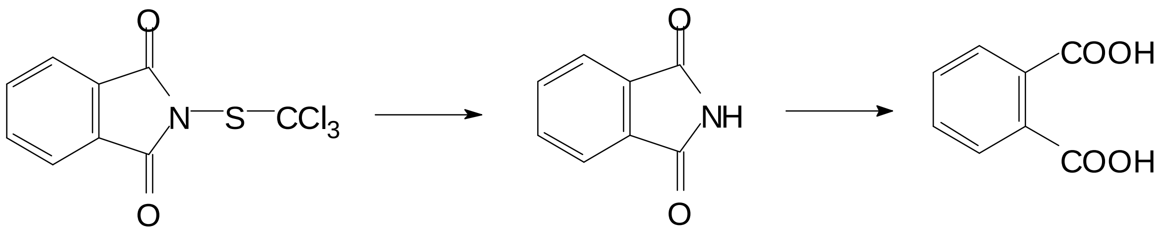
Table 6. Components of the residue on avocado fruit and leaves from a tree treated with 3×3.4 kg ai/ha [*benzene*-^{14}C]folpet and harvested 21 and 97 days after the final application (Toia and Collins, 1994).

Compound	^{14}C expressed as folpet, mg/kg				
	Fruit			Leaves	
	21 days		97 days	21 days	
	rinse	extract	extract	rinse	extract
Folpet	0.29	0.25	0.026	24	54
Phthalimide	0.20	0.55	0.22	10.4	1.2
Phthalic acid	0.077	7.2	4.5	4.0	11
Polar materials	0.018	0.52	0.40	0.94	8.6
Others		0.59	0.34		0.78

Phthalic acid was the main component of the residue in the extracts of the fruit. Extracts of the peel and pulp from mature avocado fruit (97-day) were examined separately; phthalic acid constituted 85% and 67% of the residues in the pulp and peel respectively (all expressed as folpet).

	Residue expressed as folpet, mg/kg on a whole fruit basis	
	Peel extracts	Pulp extracts
Folpet	0.022	0.004
Phthalimide	0.15	0.067
Phthalic acid	0.65	3.8
Polar compounds	0.017	0.38
Others	0.13	0.21

Folpet itself was mostly a surface residue. In the avocados harvested 21 days after the final application it accounted for 47% of the ^{14}C in the rinse, but only 2.7% of that extracted from the fruit. In the fruit harvested 97 days after the final application the residue in the rinses was too low to identify individual components, but in the extracts folpet accounted for only 0.5% of the ^{14}C.



Folpet phthalimide phthalic acid

Figure 2. Folpet metabolism in wheat, grapes and avocados.

METHODS OF RESIDUE ANALYSIS

Analytical methods

The analytical method of Schlesinger (1991) for folpet and phthalimide residues in non-oily crops was reviewed by the 1993 JMPR. Cowlyn (1996) described in detail the methods, developed from the Schlesinger method, used in the supervised trials to analyse apples, lettuce, melons, onions, strawberries and tomatoes, and summarized the validation data. Folpet in the cleaned-up extracts was determined by GLC with an ECD.

The region corresponding to the retention time of folpet in the chromatograms from control extracts was examined for potential interfering peaks. Freedom from peaks in the control was taken to indicate specificity.

Recovery range	Number of values
50-59%	2
60-69%	13
70-79%	88
80-89%	98
90-99%	79
100-109%	45
110-119%	12
120-129%	3

Recoveries of folpet by methods based on that of Schlesinger (1991) were determined during method validation and in supervised trials on apples, apple juice, wet apple pomace, cranberries, cucumbers, grape juice, grapes, lettuce, melons, must, onions, raisins, spirits, strawberries, tomatoes, tomato paste, tomato purée and wine, at levels from 0.05 mg/kg to 5 mg/kg for most commodities, and up to 20 or 50 mg/kg for some. The results were satisfactory down to a level of 0.05 mg/kg, which is the limit of determination (LOD). Recoveries did not appear to depend on the residue level or the type of sample. The 340 determinations showed mean and median recoveries of 87% and 86% respectively.

De Paoli and Bruno (1995a, method MR 52) extracted tomatoes with dichloromethane, cleaned up the extract by passage through a chromatographic cartridge, and determined folpet residues in the extract by GLC with an ECD after the addition of ethion as an internal standard. Recoveries from triplicate samples were 94-110% at 0.05 mg/kg and 97-106% at 0.20 mg/kg. The LOD was 0.05 mg/kg. The same authors (1995b) used method MR 52 to analyse strawberries. Triplicate recoveries were 72-80% at 0.10 mg/kg and 94-101% at 0.50 mg/kg.

Grinbaum (1994) analysed grape samples for folpet and phthalimide residues after extracting the grapes with acetone and cleaning up the extract by solvent partition and column chromatography (method FO 05/89). Folpet was measured with an ECD and phthalimide with a nitrogen-specific thermionic detector. Quantitative recoveries of both analytes were obtained at levels of 0.1 mg/kg and above. The mean recovery of folpet in 13 tests at fortification levels of 0.10 to 3.0 mg/kg was 91% (range 75-114%) and that of phthalimide in 9 tests at levels of 0.07 to 1.0 mg/kg was 90% (range 76-105%).

Williams (1996) tested the Schlesinger method (FP/15/91) and a method for the determination of folpet residues in oily crops (Nishioka *et al.*, 1996) to determine whether they could be successfully and reproducibly used by competent chemists without outside assistance in a laboratory without prior experience with the methods.

Williams suggested minor modifications which improved reproducibility. Dilutions of stock solutions for GLC were prepared in hexane containing 2% di(ethyleneglycol)diethyl ether, which reduced the degradation of folpet during gas chromatography; degradation had varied between runs and with different crop extracts. Additional clean-up was needed to produce clean extracts from onions. Recoveries and repeatability were satisfactory with the modified Schlesinger method for folpet in apples, cantaloupes, cranberries, cucumbers, grapes, lettuce, onions, strawberries and tomatoes. Folpet residues in avocados were successfully determined by the Nishioka method with some additional clean-up.

Stability of pesticide residues in stored analytical samples

Information was made available to the Meeting on the stability of folpet in apple juice, wet apple pomace, apples, cranberries, cucumbers, grape juice, lettuce, onions, tomato paste, tomato purée and tomatoes during frozen storage. The data are shown in Table 7.

Table 7. Freezer storage stability of folpet in various substrates fortified with folpet at 1 mg/kg. Raw agricultural commodities were stored whole. The percentage of folpet remaining was calculated from the analytical results at day 0 and after the storage interval, both of which were uncorrected for batch analytical recoveries.

Commodity	Storage temp	Folpet spike, mg/kg	% folpet remaining	Reference
Apple juice	-12 to -27°C	1.0	106 77 77	95-0059
Wet apple pomace	-12 to -27°C	1.0	99 90	95-0059
Apples, whole	-12 to -27°C	1.0	105 98 111	95-0059
Cranberries	-12 to -27°C	1.0	81 90 109 83	AA950306
Cucumbers	below -10°C	1.0	78 98	95-0065
Grape juice	below -12°C	1.0	111 116 108 105	95-0100
Lettuce	-10 to -27°C	1.0	101 96 100	95-0066
Onions	-12 to -27°C	1.0	106 93	95-0070
Tomato paste	below -10°C	1.0	89 99	95-0060
Tomato purée	below -10°C	1.0	91 89	95-0060
Tomatoes, whole	below -10°C	1.0	92 93 91 80	95-0060

Triplicate samples of raisins (hydrated) from processing trials (95-0100) on grapes were analysed for folpet residues before and after storage in a freezer below -12°C for 21 days. The residues had decreased by an average of 6%.

Folpet residues were stable in the various substrates during freezer storage for the periods tested, but in some cases the periods did not exceed 30 days.

Definition of the residue

The Meeting agreed that the current definition is suitable for assessing compliance with MRLs and for the estimation of dietary intake.

Definition of the residue for compliance with MRLs and for the estimation of dietary intake: folpet.

USE PATTERN

Table 8. Registered uses of folpet. All foliar applications.

Crop	Country	Form	Application			PHI, days
			Rate, kg ai/ha	Spray conc. kg ai/hl	No.	
Apples	Argentina	WP	3.6	0.12	3	10
Apples	Canada	WP	0.8	0.10	8	7
Apples	Chile	WP	2.0	0.11	3	7
Apples	France (north)	SC	1.04	0.104-0.14	11	14
Apples	France (south)	SC	0.98-1.2	0.081-0.12	9	14
Apples	Hungary	WP	1.6	0.104	8	10
Apples	Portugal	WP	1.6	0.13	8	21
Apples	Spain	WP	1.9	0.16	6	10
Apples	Switzerland	WG	2.0	0.10	4	21
Cucumbers	Canada	WP	1.0	0.10	8	7
Cucumbers	Mexico	WP	1.8	0.29-0.88	4	3
Grapes	Argentina	WP	1.02	0.10-0.13	4	7
Grapes	Chile	WP	2.0	0.15	3	14
Grapes, wine	Germany	SC	0.45-1.2	0.075	8	Up to stage 61 and 68-81
Grapes, wine	Germany	SC	0.6-1.6	0.1	6	Up to stage 81
Grapes	Italy	WP	1.6	0.16	5	10/40
Grapes	Mexico	WP	1	0.10-0.25	7	10
Lettuce	Greece	SC	0.61	0.12	4	20
Lettuce	Mexico	WP	1.3	0.25-0.63	4	7
Lettuce	Portugal	WP	0.52	0.13	3	14
Lettuce	Spain	WP	0.78	0.16	4	21
Melons	Greece	SC	0.49	0.061	4	20
Melons	Guatemala	WP	0.48	0.096-0.24	6	3
Melons	Honduras	WP	0.64	0.13-0.21	4	3
Melons	Mexico	WP	1.8	0.35-0.88	6	7
Onions	Chile	WP	2.0	0.13	3	7
Onions	Greece	SC	0.61	0.12	3	20
Onions	Hungary	WP	0.39-0.67	0.078-0.43	3	14
Onions	Mexico	WP	1.5	0.30-0.75	4	7
Onions	Portugal	WP	0.52	0.13	3	7
Onions	Spain	WP	0.623	0.16	3	10
Strawberries	Netherlands	WG	1.36	0.14	2	14
Strawberries	Netherlands	WP	1.34	0.13	2	14
Strawberries	Mexico	WP	1.3	0.25-0.63	4	2
Tomatoes	Chile	WP	1.7	0.15	7	7
Tomatoes	Hungary	WP	0.65	0.13	3	14
Tomatoes	Mexico	WP	2.0	0.40-1.0	5	2
Tomatoes	Portugal	WP	1.3	0.16	4	7
Tomatoes	Spain	WP	1.6	0.097-0.26	6	10

RESIDUES RESULTING FROM SUPERVISED TRIALS

Supervised residue trials on fruit and vegetables are summarized in Tables 9-15.

Table 9	Apples. Argentina, Canada, Chile, France, Germany, Hungary, Portugal, Spain, Switzerland, USA.
Table 10	Grapes. Argentina, Chile, France, Germany, Italy, Mexico.
Table 11	Strawberries. Italy, Mexico, Netherlands.
Table 12	Onions. Chile, Greece, Hungary, Mexico, Portugal, Spain.
Table 13	Cucumbers. Canada, Mexico. Melons. Greece, Guatemala, Honduras, Mexico.
Table 14	Tomatoes. Chile, Hungary, Italy, Mexico, Portugal, Spain, USA.
Table 15	Head lettuce. Greece, Hungary, Mexico, Portugal. Leaf lettuce Greece, Mexico, Spain. Lamb’s lettuce. Germany

Where residues were not detected, they are recorded in the Tables as less than the limit of determination (LOD), e.g. <0.05 mg/kg. Residues, application rates and spray concentrations have generally been rounded to 2 significant figures or, for residues near the LOD, to 1 significant figure. Although all trials included control plots, no control data are recorded in the Tables except where residues in control samples exceeded the LOD. Residues are not corrected for recoveries except in a strawberry trial and a tomato trial where only corrected results were reported.

All trials except German trials on apples and lamb’s lettuce were fully reported as well as being summarized.

Folpet was applied to apple trees in supervised trials in France, Hungary, Portugal, Spain and Switzerland by backpack airblast or lance sprayers. Plot sizes were in the range 86-240 m^2. In the label-rate trials 3 field samples were analysed from each of 2 treated plots (Table 9).

In supervised apple trials at 4 sites in Canada, 2 in Argentina and 2 in Chile folpet was applied with a motorised pump backpack sprayer or an airblast sprayer driven by a power take-off (Table 9). Plot sizes ranged from 190 to 784 m^2. Two field samples, each of 2 kg, were analysed from each plot.

In a series of trials on grapes in Argentina, Chile, Italy and Mexico folpet was applied using backpack sprayers with motorized pumps. Plot sizes ranged from 55 to 520 m^2. Duplicate field samples (2 kg) were taken from each treated plot (1 treated plot per trial). The trials were on table grapes (1 trial each in Argentina, Chile and Italy), wine grapes (1 each in Chile and Italy) and raisin grapes (Mexico). Residues in the grapes in the Mexican trial were much lower than in the others. The maximum daily temperature in the final weeks of this trial was high (41°C) and this may have had an influence.

Folpet was applied by airblast knapsack sprayers 8 or 9 times at 6-15 day intervals at 1.5 kg ai/ha to grapes in 4 supervised trials in France in 1995. Plot sizes were 378-792 m^2. Duplicate 3-kg samples of grapes were harvested from each plot 0-21 days after the final application for analysis. The samples were extracted within 3 days of receipt at the laboratory and the crude extracts were stored below -18°C. Wasser (1997) has shown that folpet residues in crude extracts of grapes were stable during refrigerator storage at 4°C for 1 month. Folpet residues in the control plot of trial EA950170 FR04 resulted from an unexpected application of folpet by the farmer approximately 2 months before harvest.

Folpet was applied by boom sprayer in the strawberry trials in Italy. Plot sizes in the two trials were 18.9 m^2 and 10 m^2. Field sample sizes were in the range 1-1.5 kg. In trial R-8989 rain (400 mm) fell between 7 and 14 days after the final application and may have reduced the residues. In trial R-8986 rain (in total about 80 mm) occurred on 9 successive days immediately after the final application. The residues from trial R-8986 were corrected for recovery, but as recoveries were in the range 84-108%, the adjustments are small.

Strawberries were produced in plastic tunnels in trials in The Netherlands. Three field samples (1 kg each) were analysed from each plot; 2 plots in each trial were treated at the label rate and 1 plot at twice that rate.

Motorised backpack sprayers were used to apply folpet to strawberries growing in 480-1200 m^2 plots in supervised trials in Mexico. Two field samples (2 kg each) were taken from each plot for analysis. Low procedural recoveries (52-53%) were experienced with strawberries from trial AA950310.01, but despite investigations no clear reason was discovered. The recorded results were not corrected for recovery.

In onion trials in Chile and Mexico folpet was applied to the foliage by backpack sprayer with a motorized pump or a CO_2 pressure source. Plot sizes were in the range 108-368 m^2. Onions (8-24 per field sample) were pulled from the ground and allowed to dry for one day in the field, then placed in a freezer after the upper foliage and roots were trimmed off. In onion trials in Greece, Hungary, Portugal and Spain folpet was applied with back boom sprayers. Plot sizes were approximately 50 m^2, with duplicate plots in each trial treated at the label rate and a single plot in each of two Hungarian trials at twice the label rate. One field sample (at least 2 kg, 12 or more onions) was analysed from each plot. The soil was removed mechanically by hand and the whole plant, including the roots and foliage, was analysed.

In cucumber trials folpet was applied with a motorized backpack sprayer in Mexico and a CO_2-pressurised backpack sprayer in Canada. Plot areas ranged from 90 to 280 m^2, each trial consisting of a treated and a control plot. The field sample size from the treated plot was 2 kg. Folpet was applied with a backpack boom sprayer to melons in trials in Greece. Plot sizes were in the range 90-180 m^2. Trials consisted of 2 plots treated at the label rate and a control plot. Duplicate field samples, each of 12 melons about 15 cm in diameter, from each trial were analysed on a “whole melon” basis.

Melons were treated with folpet applied by backpack sprayer in supervised trials in Guatemala, Honduras and Mexico. Plot sizes were in the range 120-540 m^2, with 1 treated plot and 1 control plot in each trial. Each field sample consisted of 12 melons and duplicate field samples were analysed from each plot.

The plot size was 10 m^2 in the single Italian tomato trial. Folpet was applied as a high-volume spray by knapsack. Field samples comprised 24 tomatoes. The reported residues were corrected for recovery.

Folpet was applied from a backpack boom sprayer in the tomato trials in Hungary, Spain and Portugal, except in one trial in Spain (MAK/375-07) where the spray was applied with a lance to staked tomatoes. In each trial two plots were treated at the label rate and one at a double rate. The plot size was 50 m^2. One field sample (2 kg or more) from each plot was analysed. Trials MAK/375-01 and MAK/375-03 were subject to overhead irrigation but the dates were not recorded. Residue levels could be reduced if irrigation occurred while the spray deposits were fresh.

Tomatoes at 5 sites in Mexico and 1 site in Chile were treated with folpet using backpack sprayers with motorized pumps. Plot sizes were 117-224 m^2. Two field samples (2 kg each) were analysed from the single treated plot in each trial.

Folpet was applied by backpack CO_2 boom sprayer to lettuce in two trials in Greece, one in Portugal and one in Spain. In the Greek and Spanish trials the lettuce was irrigated by overhead sprinkler either 1 or 2 days after the final application and in each of these trials the residues were below the LOD, 0.05 mg/kg. Drip irrigation was used in the trial in Portugal and the residues were substantially higher. It is likely that the sprinkler irrigation was the cause of the low residues.

A backpack boom sprayer was used to apply folpet to head lettuce grown in plastic tunnels in Hungarian trials in 1996-97. The plot size was 50 m^2. The field sample from each plot comprised 12 lettuce.

In the Mexican trials on lettuce folpet was applied with a motorized backpack sprayer. Plot areas ranged from 50 to 120 m^2. Field samples of 12 lettuce heads were cut and the outermost leaves were removed in the field. Duplicate field samples from each trial were analysed (Table 15).

Table 9. Folpet residues in apples resulting from supervised trials in Argentina, Canada, Chile, France, Germany, Hungary, Portugal, Spain, Switzerland and the USA. Residues in replicate field samples from single plots or from duplicate plots in the same trial are shown separately. Double-underlined residues are from treatments according to GAP and are valid for the estimation of maximum residue levels.

Country year (variety)	Application			PHI, days	Folpet, mg/kg	Ref
	kg ai/ha	kg ai/hl	no.			
Argentina, 1996 (Cooper 8)	3.6	0.12	3	10	1.1, 1.4	AA950314.07 95-0064
Argentina, 1996 (Red Delicious)	3.6	0.12	3	10	2.6	AA950314.08 95-0064
Canada, 1996 (Cortland)	0.81	0.10	8	7	0.36, 0.43	AA950314.02 95-0064
Canada, 1996 (McIntosh)	0.81	0.10	8	7	1.1, 0.61	AA950314.03 95-0064
Canada, 1996 (McIntosh)	0.81	0.10	8	7	0.65, 0.45	AA950314.04 95-0064
Canada, 1996 (Red Delicious)	0.78	0.10	8	7	1.4 1.3	AA950314.01 95-0064
Chile, 1996 (Imperial Gala)	2.0	0.11	3	7	1.6, 2.0	AA950314.05 95-0064
Chile, 1996 (Royal Gala)	2.0	0.11	3	7	3.2, 3.7	AA950314.06 95-0064
France (north), 1996 (Star Crimson)	0.98	0.10	11	14	0.9, 0.6, 0.7 0.7, 0.8, 0.5	MAK/374-08 R-9162
France (north), 1996 (Star Crimson)	1.0	0.10	11	14	0.7, 1.4, 0.7 0.8, 0.8, 0.6	MAK/374-09 R-9162
France (south), 1996 (Golden Delicious)	1.2	0.10	9	14	1.8, 1.2, 1.8 1.1, 1.5, 1.0	MAK/374-06 R-9162
France (south), 1996 (Golden Delicious)	0.98	0.10	9	14	1.2, 1.4, 0.8 0.7, 0.7, 1.4	MAK/374-07 R-9162
Germany, 1985 (Gloster)	0.75	10×0.075 +0.15	10 11	24 3	0.81 0.85	BBA 85/Ob/12885[1]
Germany, 1985 (Gloster)	0.75	10×0.075 +0.15	10 11	24 3	0.84 0.81	BBA 85/Ob/12885
Germany, 1985 (Gloster)	0.75	10×0.075 +0.3	10 11	24 3	0.54 0.83	BBA 85/Ob/12885
Germany, 1985 (Gloster)	10×0.75 +0.5	10×0.075 +0.1	10 11	24 3	0.32 0.52	BBA 85/Ob/12885
Germany, 1985 (Gloster)	10×0.75 +0.5	10×0.075 +0.2	10 11	24 3	0.54 0.61	BBA 85/Ob/12885
Germany, 1985 (Gloster)	10×0.75 +0.5	10×0.075 +0.2	10 11	24 3	0.32 0.43	BBA 85/Ob/12885

Country year (variety)	Application			PHI, days	Folpet, mg/kg	Ref
	kg ai/ha	kg ai/hl	no.			
Hungary, 1996 (Star King)	1.6	0.10	8	10	5.4, 4.4, 5.1 6.5, 5.9, 8.0	MAK374-01 R-9162
Portugal, 1996 (Jonagold Red)	1.6	0.13	8	21	2.7, 2.8, 2.6 3.0, 3.2, 2.3	MAK/374-05 R-9162
Portugal, 1996 (Jonagold Red)	3.1	0.26	8	21	5.5, 10.8, 9.9	MAK/374-05 R-9162
Spain, 1996 (Red Mornet)	1.9	0.16	6	10	1.7, 2.0, 3.1 2.2, 2.3, 1.7	MAK/374-04 R-9162
Spain, 1996 (Red Mornet)	3.7	0.31	6	10	6.9, 4.1, 3.0	MAK/374-04 R-9162
Switzerland, 1996 (Fiorina)	2.0	0.10	4	21	2.2, 3.1, 2.8 2.7, 3.4, 3.3	MAK/374-03 R-9162
USA (NY), 1995 (Northern Spy)	2.9	0.31	4	7	2.1	SARS-95-50 95-0059

[1]All BBA trials only reported on summary sheets

Table 10. Folpet and phthalimide residues in grapes resulting from supervised trials in Argentina, Chile, France, Germany, Italy and Mexico. Residues in replicate field samples from single plots are shown separately. Double-underlined residues are from treatments according to GAP and are valid for the estimation of maximum residue levels.

Country, year (variety)	Application				PHI, days	Residues, mg/kg		Ref
	Form	kg ai/ha	kg ai/hl	no.		folpet	phthalimide	
Argentina, 1996 (Emperador)	WP	1.0	0.13	4	7	1.6, 1.5		R-9141g AA950313.07 95-0071
Chile, 1996 (Red Globe)	WP	2.0	0.15	3	14	1.8, 2.6		R-9141g AA950313.06 95-0071
Chile, 1996 (Red Globe)	WP	2.0	0.15	3	14	1.5, 3.0		R-9141g AA950313.08 95-0071
France (Beaune), 1992	WG	1.5		8	27	1.9, 0.73, 0.88, 0.93	0.21, 0.095, 0.062, 0.091	R-7194a
					52	0.58, 0.56, 0.46, 0.68	0.071, 0.057, 0.052, 0.071	
France (Bordeaux), 1992	WG	1.5		7	0	3.6, 2.5, 2.5, 2.9	0.20, 0.18, 0.18	R-7194
					21	0.47, 1.6, 0.95, 0.39	0.18, 0.33, 0.24, 0.13	
					60	0.52, 0.14, 0.23, 0.50	0.16, 0.091, 0.091, 0.17	
France (Orange), 1992	WG	1.5		12	0	1.1, 1.5, 3.8, 6.5 c0.066	0.50, 0.31, 0.94, 1.4 c00.070	R-7194a
					15	1.8, 4.3, 1.3, 2.0 c0.098	0.94, 0.91, 0.52, 0.91 c00.11	
					30	0.76, 1.1, 0.42, 0.22 c0.057	0.48, 0.53, 0.31, 0.28 c0.056	
France, 1994 (Ugni blanc)	SC	1.5	0.43	6	52	2.8 0.27 m <0.01 w <0.01 sp		R-8411 R 5011 9401-MAK 94-66-06-22
France, 1994 (Ugni blanc)	WG	1.5	0.43	6	52	2.9 0.73 m <0.01 w <0.01 sp		R-8411 R 5011 9401-MAK 94-66-06-22

Country, year (variety)	Application				PHI, days	Residues, mg/kg		Ref
	Form	kg ai/ha	kg ai/hl	no.		folpet	phthalimide	
France, 1995 (Carignan)	SC	1.6	0.50	7 8	8 0 7 14 21	3.9, 8.1 8.3, 9.0 10.6, 7.1 4.4, 6.0 2.2, 2.2 c 0.012		EA950170 R-9146 FR03
France, 1995 (Chardonnay)	SC	1.4	0.50	8	21	2.4, 2.2		EA950170 R-9146 FR02
France, 1995 (Merlot)	SC	1.5	0.47	8	21	3.1, 2.3		EA950170 R-9146 FR01
France, 1995 (Pinot Noir)	SC	1.5	0.60	8 9	10 0 7 14 21	3.7, 3.1 6.1, 7.2 4.8, 4.0 3.2, 2.5 2.8, 2.3 c 0.06, 0.07 c 0.10, 0.06		EA950170 R-9146 FR04
Germany, 1993 (Müller-Thurgau)	WP	0.6+0.9 +1.5+1.8 +2×2.2 +2×2.6	2×0.17 +2×0.26 +2×0.30 +2×0.35	8	14 28 35 28 28	0.91 0.66 0.66 0.68 m <0.05 w	<0.1 <0.1 <0.1 0.27 m 0.29 w	R-7993 HVA 7/94 UHL08
Germany, 1993 (Müller-Thurgau)	WP	0.7+1.0 +1.7+2.0 +2×2.3 +2×2.6	2×0.17 +0.28 +0.33 +2×0.39 +2×0.44	8	7 14 27 35 27 27	1.4 1.5 1.5 1.5 0.58 m <0.05 w	<0.1 <0.1 0.1 <0.1 0.44 m 0.47 w	R-7993 HVA 7/94 UHL10
Germany, 1993 (Müller-Thurgau)	WP	0.6+0.9 +1.6+1.9 +2.2+2.3 +2.6+2.5	2×0.16 +0.27 +0.32 +0.37 +2×0.43	8	7 14 28 35 28 28	1.0 1.6 1.1 0.51 0.27 m <0.05 w	<0.1 <0.1 <0.1 <0.1 0.39 m 0.39 w	R-7993 HVA 7/94 UHL12
Germany, 1993 (Müller-Thurgau)	SC	0.38+0.54 +0.91+1.1 +2×1.3 +2×1.5	2×0.1 +0.13 +0.16 +2×0.18 +2×0.21	8	14 28 35 28 28	2.1 1.2 0.41 0.25 m <0.05 w	<0.1 <0.1 <0.1 0.26 m 0.31 w	R-7993 HVA 7/94 UHL14
Germany, 1993 (Müller-Thurgau)	SC	0.39+0.60 +1.0+1.2 +2×1.4 +2×1.6	2×0.1 +0.17 +0.20 +2×0.23 +2×0.27	8	7 14 28 35 28 28	0.77 1.1 0.42 0.40 0.27 m <0.05 w	<0.1 <0.1 <0.1 <0.1 0.37 m 0.35 w	R-7993 HVA 7/94 UHL16
Germany, 1993 (Portugieser)	WP	0.7+1.0 +1.7+2.0 +2.3+2.5 +2×2.7	2×0.17 +0.28 +0.33 +2×0.39 +2×0.44	8	7 14 28 35 28 28	3.5 1.9 2.0 2.0 <0.05 m <0.05 w	<0.1 <0.1 <0.1 <0.1 1.8 m 0.99 w	R-7993 HVA 7/94 UHL09
Germany, 1993 (Portugieser)	SC	0.39+0.60 +1.0+1.1 +2×1.4 +2×1.6	2×0.1 +0.17 +0.20 +2×0.23 +2×0.27	8	7 14 28 35 28 28	1.7 0.54 0.29 0.23 <0.05 m <0.05 w	<0.1 <0.1 <0.1 <0.1 0.44 m 0.33 w	R-7993 HVA 7/94 UHL15

Country, year (variety)	Application				PHI, days	Residues, mg/kg		Ref
	Form	kg ai/ha	kg ai/hl	no.		folpet	phthalimide	
Germany, 1993 (Reisling)	WP	0.63+0.89 +1.3+1.5 +1.7+2.0 +2×1.3	0.17	8	0 14 28 35 28 28	9.7 2.2 5.6 4.7 0.83 m <0.05 w	<0.1 <0.1 0.2 <0.1 0.72 m 0.76 w	R-7993 HVA 7/94 UHL07
Germany, 1993 (Reisling)	WG	0.6+1.0 +1.6+1.9 +2×2.2 +2.5+2.6	0.16+0.17 +0.27 +0.32 +2×0.37 +2×0.43	8	0 14 28 35 28 28	2.9 1.3 1.3 1.4 <0.05 m <0.05 w	<0.1 <0.1 <0.1 0.12 0.51 m 0.34 w	R-7993 HVA 7/94 UHL11
Germany, 1993 (Reisling)	SC	0.6+0.8 +1.2+1.4 +1.5+1.8 +2×1.2	0.1	8	0 14 28 35 28 28	12 5.6 3.3 1.9 1.0 m <0.05 w	<0.1 <0.1 0.1 <0.1 0.92 m 0.83 w	R-7993 HVA 7/94 UHL13
Italy, 1996 (Italia)	WG	1.6	0.16	5	10	3.3, 2.9		R-9141g AA950313.03 95-0071
Italy, 1996 (Rondinella)	WG	1.6	0.16	5	41	1.7, 1.7		R-9141g AA950313.04 95-0071
Mexico, 1996 (Perleete)	WP	1.0	0.14	7	10	<0.05, <0.05)		R-9141g AA950313.05 95-0071

c: control sample m: must. w: wine sp: spirit

Table 11. Folpet residues in strawberries resulting from supervised trials in Italy, Mexico and The Netherlands. Residues in replicate field samples from single plots and from duplicate plots in the same trial are shown separately. Double-underlined residues are from treatments according to GAP and are valid for the estimation of maximum residue levels.

Country, year (variety)	Application				PHI, days	Folpet, mg/kg	Ref
	Form	kg ai/ha	kg ai/hl	No.			
Italy, 1995 (Addie)	WP	1.3+3×1.2	0.13	4	0 7 10 14	0.70 0.22 0.10 0.07	R-8986 DA-10/915 IT 219/95
Italy, 1995 (Belruby)	WP	0.84+0.92 +0.89	0.15	3	0 7 14 21	0.86 0.09 <0.01 <0.01	R-8989 95I005R 95046/I1-FFST
Mexico, 1995 (Sweet Charlie)	WP	3×1.3+1.2	2×0.50 +0.52+0.62	4	2	1.7, 1.8	R-9141s 950310.01 95-0068
Mexico, 1995 (Sweet Charlie)	WP	1.2	0.31+3×0.26	4	2	0.92, 1.6	R-9141s 950310.02 95-0068
Mexico, 1995 (Seascape)	WP	1.2	0.38+0.32 +2×0.33	4	2	2.0, 2.2	R-9141s 950310.03 95-0068
Netherlands, 1996 (Elsanta)	WP	1.3+1.4	0.13	2 pt	14	1.3, 0.7, 1.2 1.0, 1.1, 1.9	R-9161 MAK/372-01
Netherlands, 1996 (Elsanta)	WP	2.7	0.27	2 pt	14	1.8, 2.0, 2.6	R-9161 MAK/372-01
Netherlands, 1996 (Elsanta)	WG	1.3	0.13	2 pt	14	0.4, 1.6, 0.8 0.8, 1.2, 1.0	R-9161 MAK/372-01

Country, year (variety)	Application				PHI, days	Folpet, mg/kg	Ref
	Form	kg ai/ha	kg ai/hl	No.			
Netherlands, 1996 (Elsanta)	WP	1.4+1.3	0.13	2 pt	14	1.0, 1.4, 1.2 1.0, 0.7, 1.0	R-9161 MAK/372-02
Netherlands, 1996 (Elsanta)	WP	2.7	0.27	2 pt	14	3.0, 3.6, 1.8	R-9161 MAK/372-02

pt: plastic tunnel

Table 12. Folpet residues in bulb onions resulting from supervised trials in Chile, Hungary, Greece, Mexico, Portugal and Spain. Residues in replicate field samples from single plots or from duplicate plots in the same trial are shown separately. Double-underlined residues are from treatments according to GAP and are valid for the estimation of maximum residue levels. Samples from European trials include roots and foliage.

County, year (variety)	Application				PHI, days	Folpet, mg/kg	Ref.
	Form	kg ai/ha	kg ai/hl	no.			
Chile, 1996 (Grano de oro)	WP	2.0	0.13	3	7	0.36, 0.27	R-9140 AA950307.03 95-0070
Greece, 1996 (Banko)	SC	0.62 +0.61+0.62	0.12	3	20	<0.05, <0.05	R-9163 MAK/377-07
Greece, 1996 (Moranda)	SC	2×0.61 +0.62	0.12	3	20	<0.05, <0.05	R-9163 MAK/377-06
Hungary, 1996 (Deutona)	WP	0.40 +0.66+0.65	0.13	3	14	<0.05, 0.07	R-9163 MAK/377-02
Hungary, 1996 (Deutona)	WP	0.75 +2×1.3	0.26	3	14	0.2	R-9163 MAK/377-02
Hungary, 1996 (Makoi Bronz)	WP	0.40 +0.67+0.65	0.13	3	14	<0.05, <0.05	R-9163 MAK/377-03
Hungary, 1996 (Makoi Bronz)	WP	0.39 +0.65+0.67	0.13	3	14	0.21, 0.09	R-9163 MAK/377-04
Hungary, 1996 (Piroschka)	WP	0.39 +2×0.65	0.13	3	14	0.05, <0.05	R-9163 MAK/377-01
Hungary, 1996 (Piroschka)	WP	0.75 +2×1.3	0.26	3	14	1.0	R-9163 MAK/377-01
Mexico, 1995 (Suprema)	WP	1.5	2×0.56 +0.36+0.51	4	7	0.41, 0.31	R-9141 AA950307.01 95-0070
Mexico, 1995 (Suprema)	WP	1.5	3×0.37 +0.56	4	7	0.41, 0.32	R-9141 AA950307.02 95-0070
Portugal, 1996 (Valenciana tardia)	WP	0.53 +0.54+0.54	0.13	3	7	5.0, 3.6	R-9163 MAK/377-08
Spain, 1996 (Dulce Babosa)	WP	0.62 +2×0.65	0.16	3	10	1.6, 2.5	R-9163 MAK/377-09

Table 13. Folpet residues in cucumbers and melons resulting from supervised trials in Canada, Greece, Guatemala, Honduras and Mexico. Residues in replicate field samples from single plots are shown separately. Double-underlined residues are from treatments according to GAP and are valid for the estimation of maximum residue levels.

Country, year (variety)	Application				PHI, days	Folpet, mg/kg	Ref
	Form	kg ai/ha	kg ai/hl	no.			
Cucumbers							

Country, year (variety)	Application				PHI, days	Folpet, mg/kg	Ref
	Form	kg ai/ha	kg ai/hl	no.			
Canada, 1996 (Panther)	WP	1.0	0.10	8	7	<0.05, 0.073	AA950312.05 95-0065
Mexico, 1995 (Dasher)	WP	1.8	0.50	4	3	0.11, 0.075	AA950312.04 95-0065
Mexico, 1995 (Fancipack)	WP	1.7	0.76	4	3	0.18, 0.36	AA950312.03 95-0065
Mexico, 1995 (pickle)	WP	1.8	0.82+0.78 +0.83+0.67	4	3	0.70, 0.41	AA950312.01 95-0065
Mexico, 1996 (Fancipack)	WP	1.8	0.79	4	3	0.55, 0.56	AA950312.02 95-0065
Melons							
Greece, 1996	SC	0.49	0.061	4	20	<0.05, <0.05	R-9159 MAK/373-03
Greece, 1996	SC	0.49	0.061	4	20	<0.05, <0.05	R-9159 MAK/373-04
Greece, 1996 (Galia)	SC	0.49	0.061	4	20	<0.05, <0.05	R-9159 MAK/373-02
Greece, 1996 (Macmidon)	SC	0.49	0.061	4	20	<0.05, <0.05	R-9159 MAK/373-01
Greece, 1996 (Macmidon)	SC	0.98	0.12	4	20	<0.05	R-9159 MAK/373-01
Greece, 1996 (Macmidon)	SC	0.97	0.12	4	20	<0.05	R-9159 MAK/373-02
Guatemala, 1996 (Cristobal)	WP	0.49	0.10	6	3	0.23, 0.21	R-9141m AA950308.06 95-0067
Honduras, 1996 (Hy-Mark)	WP	0.65	0.13	4	3	0.32, 0.17	R-9141m AA950308.04 95-0067
Honduras, 1996 (Hy-Mark)	WP	0.65	0.13	4	3	0.20, 0.41	R-9141m AA950308.05 95-0067
Mexico, 1996 (Cruiser F1)	WP	1.8	0.86+0.87 +0.85+0.84 +2×0.79	6	7	2.2, 0.94	R-9141m AA950308.01 95-0067
Mexico, 1996 (Cruiser)	WP	1.8+1.6 +1.9+1.8 +1.9+1.8	0.62+0.44 +0.55+0.54 +0.54+0.55	6	7	0.89, 0.72	R-9141m AA950308.02 95-0067
Mexico, 1996 (Hiline)	WP	1.8	0.63	6	7	0.30, 0.40	R-9141m AA950308.03 95-0067

Table 14. Folpet residues in tomatoes resulting from supervised trials in Chile, Hungary, Italy, Mexico, Portugal, Spain and USA. Residues in replicate field samples from single plots or from duplicate plots in the same trial are shown separately. Double-underlined residues are from treatments according to GAP and are valid for the estimation of maximum residue levels.

Country, year (variety)	Application				PHI, days	Folpet, mg/kg	Ref
	Form	kg ai/ha	kg ai/hl	no.			
Chile, 1996 (Conservo)	WP	1.7	1.5	7	7	1.4, 2.4	R-9141t AA950311.06 95-0069
Hungary, 1996 (Kecskemet 407)	WP	0.65	0.13	3	14	<0.05, <0.05	R-9158 MAK/375.01
Hungary, 1996 (Kecskemet 407)	WP	1.3	0.26	3	14	0.098	R-9158 MAK/375.01
Hungary, 1996 (Koral)	WP	1.3	0.26	3	14	0.06	R-9158 MAK/375.02
Hungary, 1996 (Koral)	WP	0.66+0.64 +0.65	0.13	3	14	<0.05, <0.05	R-9158 MAK/375.02
Hungary, 1996 (Prima)	WP	0.65	0.13	3	14	<0.05, <0.05	R-9158 MAK/375.04
Hungary, 1996 (Rio Fiego)	WP	2×0.65 +0.66	0.13	3	14	<0.05, <0.05	R-9158 MAK/375.03
Italy, 1995 (UC 82 VF)	WP	1.2	0.13	4	0 7 10 14	0.95 0.55 0.60 0.20	R-8987 IT 217/95 DA-12/95
Mexico, 1995 (Rio Grande)	WP	2.0	0.58+0.72 +0.67+0.66 +0.67	5	2	0.86, 1.0	R-9141t AA950311.01 95-0069
Mexico, 1995 (SM10)	WP	2.0	0.96+0.91 +0.80 +2×0.71	5	2	0.81, 1.6	R-9141t AA950311.04 95-0069
Mexico, 1995 (SM10)	WP	2.0	0.96+0.86 +0.77 +2×0.66	5	2	1.1, 1.8	R-9141t AA950311.05 95-0069
Mexico, 1996 (Rio Grande Mejorada)	WP	2.0	2×0.80 +0.76+0.75 +0.71	5	2	0.45, 0.33	R-9141t AA950311.02 95-0069
Mexico, 1996 (Rio Grande Mejorada)	WP	2.0	0.87+0.80 +2×0.75 +0.72	5	2	0.64, 1.3	R-9141t AA950311.03 95-0069
Portugal, 1996 (Melero)	WP	1.3	0.16	4	7	0.27, 0.34	R-9158 MAK/375.08
Portugal, 1996 (Petto 95)	WP	1.3	0.16	4	7	0.28, 0.58	R-9158 MAK/375.09
Spain, 1996 (Petto 95)	WP	1.6	0.26 +5×0.20	6	10	1.3, 0.36	R-9158 MAK/375.06
Spain, 1996 (Prieto)	WP	2×1.6 +2×2.2 +2×2.5	0.26 +5×0.16	6	10	0.99, 1.2	R-9158 MAK/375.07
USA, 1995 (Peel Mech)	WP	2.2	0.58	5	7	1.8 <0.05 purée <0.05 paste	R-9101 SARS-95-51 95-0060

Table 15. Folpet residues in head and leaf lettuce resulting from supervised trials in Greece, Hungary, Mexico, Portugal and Spain and from lamb's lettuce from trials in Germany. Residues in replicate field samples from single plots or from duplicate plots in the same trial are shown separately. Double-underlined residues are from treatments according to GAP and are valid for the estimation of maximum residue levels.

Country, year (variety)	Application				PHI, days	Folpet, mg/kg	Ref
	Form	kg ai/ha	kg ai/hl	No.			
Head Lettuce							
Greece, 1996 (Crispa)	SC	0.61	0.12	3	20	<0.05, <0.05	R-9160 MAK/378-07
Hungary, 1996 (Chagal)	WP	0.64 -0.66	0.13	pt 3	14	18, 24	MAK/378-01 MAK378/970321
Hungary, 1996 (Chagal)	WP	1.3	0.26	pt 3	14	50	MAK/378-01 MAK378/970321
Hungary, 1996 (Mildred)	WP	0.65 -0.67	0.13	pt 3	14	29, 21	MAK/378-02 MAK378/970321
Hungary, 1996 (Mildred)	WP	1.3	0.26	pt 3	14	61	MAK/378-02 MAK378/970321
Hungary, 1997 (Oktavo)	WP	0.65	0.13	pt 3	14	12, 9.9	MAK/378-04 MAK378/970321
Hungary, 1997 (Vicky)	WP	0.63 -0.66	0.13	pt 3	14	39, 25	MAK/378-03 MAK378/970321
Mexico, 1995 (Great Lakes 407P)	WP	1.3	0.36+0.42 +0.41 +2×0.44	5	7	1.6, 4.5	AA950309.03 95-0066
Mexico, 1996 (Climax)	WP	1.3	0.46+3×0.45 +0.40	5	7	3.2, 9.8	AA950309.02 95-0066
Mexico, 1996 (Top Gun)	WP	1.3	0.44+0.42 +2×0.41 +0.46	5	7	wl (16, 15) xwl (0.22, 0.26)	AA950309.04 95-0066
Portugal, 1996 (Grand rapids)	WP	0.52	0.13	3	14	4.3, 2.4	R-9160 MAK/378-09
Leaf lettuce							
Greece, 1996 (Romana)	SC	0.63	0.12	4	20	<0.05, <0.05	R-9160 MAK/378-06
Mexico, 1996 (Parris Island)	WP	1.2	0.58+2×0.57 +0.56+0.60	5	7	19, 22	AA950309.01 95-0066
Spain, 1996 (Romana)	WP	0.78	0.16	4	21	<0.05, <0.05	R-9160 MAK/378-08
Lamb's lettuce							
Germany, 1975 (Polar)	WP	0.68	0.096	3	10	55	BBA 15/75
Germany, 1975 (Hild's Vit-Neuheit)	WP	0.68	0.096	2	10	56	BBA 15/75
Germany, 1976 (Stuttgarter)	WP	0.68	0.15	4	15	54	BBA 15/75
Germany, 1976 (Stuttgarter)	WP	0.68	0.15	4	15	51	BBA 15/75
Germany, 1975 (Felma GS)	WP	0.68	0.11	4	11	10	BBA 15/75
Germany, 1975 (Dunkelgrüner Vollherziger)	WP	0.68	0.11	4	10	66	BBA 15/75
Germany, 1975 (Hollander)	WP	0.68	0.11	4	10	44	BBA 15/75
Germany, 1975 (Holländischer Breitblättriger)	WP	0.68	0.11	4	10	12, 20	BBA 14/75
Germany, 1975 (Hilmar)	WP	0.68	0.11	4	10	20	BBA 14/75
Germany, 1975 (Dunkelgrüner Vollherziger)	WP	0.68	0.11	4	10	22, 22	BBA 14/75
Germany, 1975 (Felma GS)	WP	0.68	0.11	4	10	211	BBA 14/75
Germany, 1975 (Dunkelgrüner Vollherziger)	WP	0.68	0.11	4	10	188	BBA 14/75
Germany, 1975 (Stuttgarter Markt)	WP	0.68	0.096	3	10	1.3 c 14	BBA 14/75

Country, year (variety)	Application				PHI, days	Folpet, mg/kg	Ref
	Form	kg ai/ha	kg ai/hl	No.			
Germany, 1975 (Stuttgarter Markt)	WP	0.68	0.084	3	10	33 c 6.7	BBA 14/75
Germany, 1975 (Stuttgarter Markt)	WP	0.68	0.096	2	14	5.6	BBA 14/75
Germany, 1975 (Dunkelgroßer Vollherziger)	WP	0.68	0.11	3	15	2.4	BBA 14/75

c: control
pt: plastic tunnels
wl: with wrapper leaves
xwl: without wrapper leaves

Table 16. Interpretation table for folpet residues on apples from trials in Table 9 and from 1993 Evaluations. GAP and trial conditions are compared for treatments considered valid for MRL and STMR estimation.

	Use pattern				Trials	Folpet, mg/kg
	kg ai/ha	kg ai/hl	No of appl	PHI, days		
Argentina GAP	3.6	0.12	3	10		
Argentina trials	3.6	0.12	3	10	AA950314.07	1.4
Argentina trials	3.6	0.12	3	10	AA950314.08	2.6
Canada GAP	0.8	0.10	8	7		
Canada trial	0.78	0.10	8	7	AA950314.01	1.4
Canada trial	0.81	0.10	8	7	AA950314.02	0.43
Canada trial	0.81	0.10	8	7	AA950314.03	1.1
Canada trial	0.81	0.10	8	7	AA950314.04	0.65
Chile GAP	2.0	0.11	3	7		
Chile trial	2.0	0.11	3	7	AA950314.05	2.0
Chile trial	2.0	0.11	3	7	AA950314.06	3.7
Hungary GAP	1.6	0.10	8	10		
Hungary trials	1.6	0.10	8	10	MAK374-01	8.0
Switzerland GAP	2.0	0.10	4	21		
Switzerland trial	2.0	0.10	4	21	MAK/374-03	3.4
Spain GAP	1.9	0.16	6	10		
Spain trial	1.9	0.16	6	10	MAK/374-04	3.1
Portugal GAP	1.6	0.13	8	21		
[1]Portugal trial	1.3	0.13	10	21	FP/25/91	1.8
Portugal trial	1.6	0.13	8	21	MAK/374-05	3.2
France (nth) GAP	1.04	0.14	11	14		
France (nth) trial	0.98	0.10	11	14	MAK/374-08	0.9
France (nth) trial	1.0	0.10	11	14	MAK/374-09	1.4
France (sth) GAP	1.2	0.12	9	14		
France (sth) trial	1.2	0.10	9	14	MAK/374-06	1.8
France (sth) trial	0.98	0.10	9	14	MAK/374-07	1.4

Table 17. Interpretation table for folpet residues on grapes from trials in Table 10 and from 1993 Evaluations. GAP and trial conditions are compared for treatments considered valid for MRL and STMR estimation.

	Use pattern	Trial	Folpet, mg/kg

[1] From 1993 JMPR

	kg ai/ha	kg ai/hl	No of appl	PHI days		
Mexico GAP	1	0.25	7	10		
Mexico trial	1.0	0.14	7	10	AA950313.05	<0.05
Chile GAP	2.0	0.15	3	14		
Chile trial	2.0	0.15	3	14	AA95013.06	2.6
Chile trial	2.0	0.15	3	14	AA95013.08	3.0
Argentina GAP	1.02	0.13	4	7		
Argentina trial	1.0	0.13	4	7	AA950313.07	1.6
Italy GAP	1.6	0.16	5	10		
Italy trial	1.6	0.16	5	10	AA950313.03	3.3
France trial	1.5	0.60	8	10	R-9146 FR04	3.7
France trial	1.6	0.50	7	8	R-9146 FR03	8.1
Italy trial	1.5	0.25	7	10	IT-302-91	0.75
Italy trial	1.5	0.15	10	10	IT-301-91	0.58
France trial	1.5	1.1	7	10	102/91	1.3
France trial	1.5	1.9	7	10	103/91	2.2

Table 18. Interpretation table for folpet residues on melons from trials in Table 13. GAP and trial conditions are compared for treatments considered valid for MRL and STMR estimation.

	Use pattern				Trial	Folpet, mg/kg
	kg ai/ha	kg ai/hl	No.	PHI days		
Mexico GAP	1.8	0.88	6	7		
Mexico trial	1.8	0.79	6	7	AA950308.01	2.2
Mexico trial	1.8	0.55	6	7	AA950308.02	0.89
Mexico trial	1.8	0.63	6	7	AA950308.03	0.40
Honduras GAP	0.64	0.21	4	3		
Honduras trial	0.65	0.13	4	3	AA950308.04	0.32
Honduras trial	0.65	0.13	4	3	AA950308.05	0.41
Guatemala GAP	0.48	0.24	6	3		
Guatemala trial	0.49	0.1	6	3	AA950308.06	0.23
Greece GAP	0.49	0.061	4	20		
Greece trial	0.49	0.061	4	20	MAK/373-01	<0.05
Greece trial	0.98	0.12	4	20	MAK/373-01	<0.05
Greece trial	0.49	0.061	4	20	MAK/373-02	<0.05
Greece trial	0.97	0.12	4	20	MAK/373-02	<0.05
Greece trial	0.49	0.061	4	20	MAK/373-03	<0.05
Greece trial	0.49	0.061	4	20	MAK/373-04	<0.05

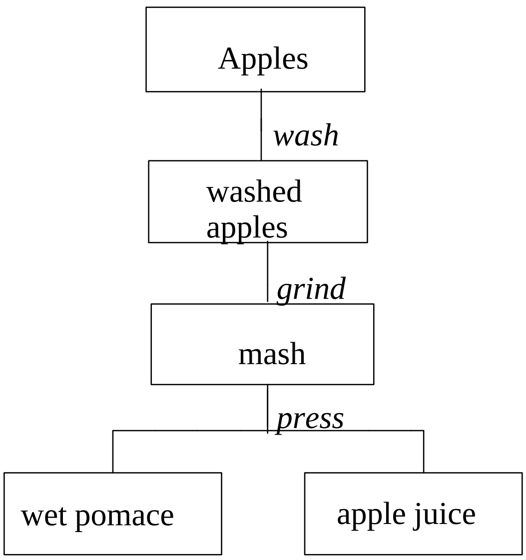
Table 19. Interpretation table for folpet residues on tomatoes from trials in Table 14 and from 1993 Evaluations. GAP and trial conditions are compared for treatments considered valid for MRL and STMR estimation.

	Use pattern				Trial	Folpet, mg/kg
	kg ai/ha	kg ai/hl	No of appl	PHI days		folpet
Chile GAP	1.7	0.15	7	7		
Chile trial	1.7	1.5	7	7	AA950311.06	2.4
Hungary GAP	0.65	0.13	3	14		
Hungary trial	0.63	0.12	5	14	FP/26/91	<0.02
Hungary trial	0.65	0.13	3	14	MAK/375.01	<0.05
Hungary trial	0.65	0.13	3	14	MAK/375.02	<0.05
Hungary trial	0.65	0.13	3	14	MAK/375.04	<0.05
Hungary trial	0.66	0.13	3	14	MAK/375.03	<0.05
Portugal GAP	1.3	0.16	4	7		

	Use pattern				Trial	Folpet, mg/kg
	kg ai/ha	kg ai/hl	No of appl	PHI days		folpet
Italy trial	1.2	0.13	4	7	IT217/95	0.55
Portugal trial	1.3	0.16	4	7	MAK/375.08	0.34
Portugal trial	1.3	0.16	4	7	MAK/375.09	0.58
Mexico GAP	2.0	1.0	5	2		
Mexico trial	2.0	0.67	5	2	AA950311.01	1.0
Mexico trial	2.0	0.71	5	2	AA950311.04	1.6
Mexico trial	2.0	0.66	5	2	AA950311.05	1.8
Mexico trial	2.0	0.71	5	2	AA950311.02	0.45
Mexico trial	2.0	0.72	5	2	AA950311.03	1.3
Spain GAP	1.6	0.26	6	10		
Spain trial	1.6	0.2	6	10	MAK/375.06	1.3

FATE OF RESIDUES IN STORAGE AND PROCESSING

The Meeting received information on the fate of folpet during the processing of apples, grapes and tomatoes.



Leppert (1996a) applied folpet four times at 2.9 kg ai/ha (0.31 kg ai/hl) with airblast equipment to an apple orchard in a processing trial in the USA (NY). The treated plot was 357 m^2. Apples (49 kg) were harvested 7 days after the final application and processed into wet pomace and juice. The residue levels in the unwashed apples are shown in Table 9, trial SARS-95-50).

Armstrong and Luke (1995) processed the apples to simulate commercial practice as closely as possible. The apples were washed, then ground in a hammer-mill to produce a wet mash which was pressed in a hydraulic press to separate the juice and wet pomace. The results and the processing factors are shown in Table 20.

Table 20. Folpet residues in apples, pomace and juice (Leppert 1996a, Armstrong and Luke 1995, Hurley and Farthing 1996e).

Sample	Folpet, mg/kg	Processing factor

Sample	Folpet, mg/kg	Processing factor
Apples, unwashed	2.1	
Apples, washed	1.2	0.60
Wet pomace	5.4	2.6
Juice	0.072	0.035

Singer (1997g) dipped 74 kg of grapes (Thomson Seedless) in 7-10 kg portions for 30 seconds in a vat containing folpet spray mixture at a concentration of 1.25 kg ai/hl, five times the maximum concentration permitted on grapes in Mexico. The grapes were then allowed to dry on polythene sheeting. Because folpet was shown in the metabolism studies to be a surface residue it was considered valid to treat grapes in this way instead of by field spraying. Abdelrahim (1996) processed the grapes into raisins and juice.

Bunches of the unwashed grapes were spread out on stainless steel screens on tables covered with black plastic and sun-dried until the moisture level had dropped to 12-16% to produce unprocessed raisins, samples of which were stored in a freezer. The remaining dried grapes were collected in plastic bags and kept in an incubator at 21°C until removed for destemming and sampling. After destemming, the dried grapes were returned to the incubator at 21°C and subsequently rehydrated to 18-20% moisture to produce raisins.

The grapes were processed in a crusher/destemmer, which crushes the berries and separates the stems from the crushed berries and juice. The crushed berries and juice were treated with an enzyme, heated at 60°C for 2 hours to remove pectin, and then separated by pressing into unclarified juice and pomace. The juice was heated at 88°C to inactivate the enzyme, filtered through diatomaceous earth, and then placed in cold storage for 6 weeks to allow settling. Clear juice was produced by filtration through diatomaceous earth, heated to canning temperature (94°C) and run into cans which were then sealed. The residues and processing factors are shown in Table 21 (Farthing, 1996d).

Table 21. Folpet residues in grapes, juice and raisins after dipping the grapes in a vat containing a 1.25 kg ai/hl folpet spray mixture (Singer, 1997g; Abdelrahim, 1996; Farthing, 1996d).

Sample	Folpet, mg/kg	Processing factor
Grapes	19, 12, 15, 17, 14, 14	
Grape juice	<0.05 (3)	0 (<0.003)
Raisins before rehydration	58, 41, 46	3.2
Hydrated raisins	31, 28, 27	1.9

Folpet residues were not detected in the grape juice and were presumably lost in the filtration and/or heating steps. Residues were concentrated during the drying process to produce raisins.

In two trials in France, Wasser (1996) treated grapes 6 times with folpet (SC and WG formulations) at 1.5 kg ai/ha and harvested them 52 days after the final application. Folpet residues were determined in the grapes and the must, wine and spirits prepared from them. The results are shown in Table 10 (trials R 5011). Some folpet residues appeared in the must, but none in the wine or spirits.

Folpet and phthalimide residues were measured in grapes, must and wine in a series of trials in Germany. The treatment details are recorded in Table 10. The residues in grapes, must and wine and the processing factors are shown in Table 22.

Table 22. Processing factors and residues of folpet and phthalimide in grapes, must and wine after grapes were sprayed with folpet. Application details are provided in Table 10.

Commodity	Residues, mg/kg		Processing factor, folpet	Processing yield, phthalimide[1]	Reference
	folpet	phthalimide			
Grapes Must Wine	5.6 0.83 <0.05	0.2 0.72 0.76	 0.15 0 (<0.009)	 0.24 0.26	R-7993 HVA 7/94 UHL07
Grapes Must Wine	0.66 0.68 <0.05	<0.1 0.27 0.29	 0.97 0 (<0.08)	 0.83 0.89	R-7993 HVA 7/94 UHL08
Grapes Must Wine	2.0 <0.05 <0.05	<0.1 1.8 0.99	 0 (<0.03) 0 (<0.03)	 1.8 0.99	R-7993 HVA 7/94 UHL09
Grapes Must Wine	1.5 0.58 <0.05	0.1 0.44 0.47	 0.39 0 (<0.03)	 0.52 0.56	R-7993 HVA 7/94 UHL10
Grapes Must Wine	1.3 <0.05 <0.05	<0.1 0.51 0.34	 0 (<0.04) 0 (<0.04)	 0.79 0.53	R-7993 HVA 7/94 UHL11
Grapes Must Wine	1.1 0.27 <0.05	<0.1 0.39 0.39	 0.25 0 (<0.05)	 0.72 0.72	R-7993 HVA 7/94 UHL12
Grapes Must Wine	3.3 1.0 <0.05	0.1 0.92 0.83	 0.30 0 (<0.02)	 0.53 0.48	R-7993 HVA 7/94 UHL13
Grapes Must Wine	1.2 0.25 <0.05	<0.1 0.26 0.31	 0.21 0 (<0.04)	 0.44 0.52	R-7993 HVA 7/94 UHL14
Grapes Must Wine	0.29 <0.05 <0.05	<0.1 0.44 0.33	 0 (<0.17) 0 (<0.17)	 3.1 2.3	R-7993 HVA 7/94 UHL15
Grapes Must Wine	0.42 0.27 <0.05	<0.1 0.37 0.35	 0.64 0 (<0.12)	 1.8 1.7	R-7993 HVA 7/94 UHL16

[1]See definition of processing yield below

The processing factors for folpet residues in the process from grapes to must and wine were calculated by dividing the folpet residue level in the must and wine by the residue level in the grapes. The processing factors for folpet from grapes to must were 0, 0, 0, 0.15, 0.21, 0.25, 0.30, 0.39, 0.64 and 0.97, with a mean of 0.29. Folpet was not detected in the wine so the processing factor for folpet from grapes to wine is 0.

Phthalimide residues in the must and wine may arise by transfer of phthalimide from the grapes or conversion of folpet to phthalimide during processing. Processing yields for phthalimide have been calculated from the following formula.

$$\textbf{Processing yield} = \frac{\textbf{phthalimide residue in must or wine}}{\textbf{folpet residue in grapes x 0.496 + phthalimide residues in grapes}}$$

The factor 0.496 is the ratio of the molecular weight of phthalmimide (147.13) to that of folpet (296.55).

The processing yields for phthalimide from grapes to must were 0.24, 0.44, 0.52, 0.53, 0.72, 0.79, 0.83, 1.8, 1.8 and 3.1. The mean was 1.1.

The processing yields for phthalimide from grapes to wine were 0.26, 0.48, 0.52, 0.53, 0.56, 0.72, 0.89, 0.99, 1.7 and 2.3, with a mean of 0.90. These results suggest that most of the folpet on the grapes is converted to phthalimide which finds its way into wine during vinification.

Leppert (1996b) applied folpet five times at 2.2 kg ai/ha (0.58 kg ai/hl) to tomato plants in a processing trial in California. The treated plot was 186 m^2. Tomatoes (152 kg) were harvested 7 days after the final application and processed into purée and paste. The residues in the unwashed tomatoes and processed commodities are shown in Table 14 (trial SARS-95-51).

The tomatoes were initially soaked with 0.5% sodium hydroxide for 3 minutes and then rinsed with a high pressure spray for 30 seconds. The washed tomatoes were crushed, rapidly heated and held for 15 seconds in a steam-jacketed kettle, then separated into pulp and juice. Purée was produced from the juice by evaporation, adjustment of salt and water levels, heating and canning. Paste was produced similarly, but with a higher salt level.

Folpet was not detected (<0.05 mg/kg) in the purée or paste produced from tomatoes containing 1.8 mg/kg of folpet. It is likely that the initial vigorous cleaning of the tomatoes would remove or destroy most of the folpet. The calculated processing factor for the transfer of folpet from tomatoes to purée and paste is <0.028.

Residues in the edible portion of food commodities

A trial on head lettuce in Mexico provided evidence that almost all of the folpet residue was on the wrapper leaves.

The processing factor for folpet residues from unwashed apples to apple juice was 0.035.

Folpet residues were not detected (<0.05 mg/kg) in grape juice produced from folpet-treated grapes containing 12-19 mg/kg. The processing factors for producing dry raisins and hydrated raisins were 3.2 and 1.9 respectively.

Folpet residues were not detected (<0.01 mg/kg) in wine or spirits produced from treated grapes in France, and not detected (<0.05 mg/kg) in wine from treated grapes in a series of trials in Germany. The mean processing yield for phthalimide in wine in the German trials was 0.90, suggesting that most of the folpet on the grapes was converted to phthalimide during vinification.

Folpet residues were not detected (<0.05 mg/kg) in purée or paste produced from tomatoes containing 1.8 mg/kg of folpet.

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

Cugier (1992) reported a 3-year survey for 1990-1992 of residues in grapes and wine in France. Of the 57 grape samples analysed for folpet, residues were detected (with an LOD of 0.05 mg/kg) in 13 and none exceeded the French MRL of 3 mg/kg. Folpet was not detected (LOD 0.02 mg/kg) in the 7 wines analysed.

NATIONAL MAXIMUM RESIDUE LIMITS

The Meeting was aware that the following MRLs had been established for folpet in apples, cucumbers, grapes, lettuce, melons, onions, strawberries and tomatoes.

Country	MRL, mg/kg							
	Apple	Cucumber	Grape	Lettuce	Melon	Onion	Strawberries	Tomato
Argentina	10	15	15		15	2	15	15
Austria	3	0.1	3	2	0.1	0.1	3	3
Belgium	3	0.1	3	2	0.1	0.1	3	3
Brazil	10	2	15	15	2	2	20	
Canada	25	15	25	25	15		25	25
Chile	25		25		15		25	25
Costa Rica	25	15	25	50	15	15	25	25
Croatia	2		2					
Czech Rep	2		2					
Ecuador	25	15	25	50	25	25	25	25
EEC[1]	3		3	2			3	3
France	3	0.1	3	2	0.1	0.1	3	3
Germany	3	0.1	3	2	0.1	0.1	3	3
Greece		3	3	2	3	3	3	3
Guatemala	25	15	25	50	15	15	25	25
Hungary	2	2	5t, 2w[2]	5	5	5	5	5
Israel	10	0.5						
Italy	3	0.1	3	2	0.1	0.1	0.1	3
Korea	5	5	5	2	2	2	5	2
Macedonia	2		2					
Mexico	25	15	25	50	15	15	25	25
Netherlands	3	0.1	3	2	0.1	0.1	3	3
Portugal	3		3	2	0.1	0.1	0.1	3
Romania	2		2					
Slovakia	2		2					
Sth Africa			15					
Spain	3	0.1	3	2	0.1	0.1	3	3
Sweden	3	0.1	3	2	0.1	0.1	3	3
Switzerland	3		15		3			
Uruguay	10	2	25	15	2	2	20	20
USA	25	15	25	50	25	25	25	25
Yugoslavia	2		2					

[1]Directive 76/893 EEC
T: table w: wine

APPRAISAL

Residue aspects of folpet were most recently reviewed in 1993 and 1994. The Meeting received information on metabolism, analytical methods, stability of samples during freezer storage, registered uses, data from supervised trials on fruit and vegetable crops, and processing studies.

The Meeting noted that folpet was scheduled for periodic review by the FAO Panel in 1998.

When the roots of tomato plants were treated with [*carbonyl*-^{14}C]folpet the ^{14}C was rapidly absorbed into the plants (85% within 1 day). After 11 days 90% of the absorbed ^{14}C was in the tops. Folpet itself was a very minor constituent (<0.1-0.2%) of the residue within the plant. The main identified components were phthalimide, phthalamic acid and phthalic acid. Unidentified polar metabolites, possibly ring-hydroxylated phthalamic acid derivatives, accounted for 15-30% of the ^{14}C in the tops.

When wheat was treated with [*phenylene*-^{14}C]folpet at a rate equivalent to 1.6 kg ai/ha and harvested 43 and 54 days after the second treatment the levels of ^{14}C were lower in the roots than in the straw or grain. Folpet was the major component of the residue in the straw (4.7 mg/kg) and grain (9.3 mg/kg) with the metabolites phthalic acid (4.3 mg/kg in straw and 6.4 mg/kg in grain) and phthalimide (1.5 mg/kg in straw and 3.1 mg/kg in grain) also significant constituents.

When Thomson Seedless grape vines were treated 3 times with [*phenylene*-^{14}C]folpet at a rate equivalent to 1.5 kg ai/ha and the grapes harvested 25 days after the final treatment, surface rinsing removed 26% of the grape residue. Folpet itself constituted 27% of the residue in or on the grapes, and phthalic acid and phthalimide 5.8% and 11% respectively. An unidentified compound in the water-soluble fraction accounted for 41% of the residue. It was very polar and yielded phthalic acid on hydrolysis, so was likely to be a conjugate or conjugates of phthalic acid.

A small avocado tree was treated with 3 foliar applications equivalent to 3.4 kg ai/ha of [*phenylene*-^{14}C]folpet and fruit were harvested at maturity 97 days after the final application. Very little residue was removed by rinsing the fruit, but most of it was extractable with ethyl acetate from the peel and pulp. The residues in or on the fruit were folpet 0.026 mg/kg, phthalimide 0.22 mg/kg and phthalic acid 4.5 mg/kg. Polar and other unidentified residues accounted for about 0.7 mg/kg. Folpet and phthalimide residues were mainly on the peel, but most of the phthalic acid residue was in the pulp.

The 1993 JMPR reviewed the Schlesinger analytical method for residues of folpet and phthalimide. The methods used in the supervised trials on apples, lettuce, melons, onions, strawberries and tomatoes were developed from the Schlesinger method. Folpet was determined in the cleaned up extract by GLC with an ECD. The recovery of folpet from various fortified commodities was commonly 70-100%, but with some excursions outside this range. In a total of 340 tests the mean and median recoveries were 87% and 86% respectively. The LOD was 0.05 mg/kg.

Folpet residues were shown to be stable during freezer storage for the intervals tested in apple juice (30 days), wet apple pomace (35 days), apples (149 days), cranberries (176 days), cucumbers (29 days), grape juice (36 days), lettuce (90 days), onions (41 days), tomato paste (30 days), tomato purée (31 days) and tomatoes (136 days).

Information was made available to the Meeting on registered uses of folpet and on supervised trials on apples, grapes, strawberries, onions, cucumbers, melons, tomatoes and lettuce. Relevant data evaluated in 1993 and 1994 were also reviewed where possible.

Folpet is registered in Argentina for use on apples with 3 applications of 3.6 kg ai/ha and harvest 10 days after the final application. Folpet residues in apples from 2 trials according to GAP were 1.4 and 2.6 mg/kg.

Canadian GAP permits folpet to be applied 8 times to apples at 0.8 kg ai/ha with harvest 7 days after the final application. In 4 trials where the use pattern corresponded to GAP the residues were 0.43, 0.65, 1.1 and 1.4 mg/kg.

Folpet residues from 2 trials on apples in Chile where the trial conditions corresponded to the registered use (2.0 kg ai/ha, 3 applications, 7 days PHI) were 2.0 and 3.7 mg/kg.

In a Hungarian trial which complied with GAP (8 applications of 1.6 kg ai/ha and a PHI of 10 days), a Swiss trial according to GAP (4 applications of 2.0 kg ai/ha and a PHI of 21 days), and a Spanish trial complying with GAP (10 applications of 1.9 kg ai/ha and a PHI of 10 days), the folpet residues were 8.0, 3.4, and 3.1 mg/kg respectively.

Folpet may be applied 8 times at 1.6 kg ai/ha to apples in Portugal with harvest 21 days after the final application. In a trial meeting these conditions the residue was 3.2 mg/kg. In a trial reported in 1993 folpet was applied 10 times at 1.3 kg ai/ha, which is within the acceptable range for evaluation, and the resulting residue after 21 days was 1.8 mg/kg

In France folpet may be used up to 11 times on apples at 1.0-1.2 kg ai/ha with harvest 14 days later. In 4 trials in France complying with GAP the residues were 0.9, 1.4, 1.4 and 1.8 mg/kg.

In summary, the folpet residues in apples from trials according to GAP were 1.4 and 2.6 mg/kg in Argentina, 0.43, 0.65, 1.1 and 1.4 mg/kg in Canada, 2.0 and 3.7 mg/kg in Chile, 8.0 mg/kg in Hungary, 3.4 mg/kg in Switzerland, 3.1 mg/kg in Spain, 1.8 and 3.2 mg/kg in Portugal, and 0.9, 1.4, 1.4 and 1.8 mg/kg in France. The residues in rank order (median underlined) in the 17 trials were 0.43, 0.65, 0.9, 1.1, 1.4, 1.4, 1.4, 1.4, 1.8, 1.8, 2.0, 2.6, 3.1, 3.2, 3.4, 3.7 and 8.0 mg/kg.

The Meeting estimated a maximum residue level and an STMR of 10 mg/kg and 1.8 mg/kg respectively for apples.

The folpet residue in grapes was 1.6 mg/kg in a supervised trial that complied with GAP in Argentina (4 applications of 1.0 kg ai/ha and a PHI of 7 days). The residues were 2.6 and 3.0 mg/kg in 2 supervised trials in Chile according to GAP (2.0 kg ai/ha, 3 applications and 14 days PHI), and below the LOD, <0.05 mg/kg, in a Mexican trial in accordance with GAP (1.0 kg ai/ha, 7 applications and a PHI of 10 days).

Italian GAP permits 5 applications of folpet to grapes at 1.6 kg ai/ha with harvest 10 days after the final application. In an Italian trial according to GAP in 1996 and 2 Italian trials reported in 1993 where folpet was used 7 and 10 times at 1.5 kg ai/ha with a PHI of 10 days the folpet residues were 3.3, 0.58 and 0.75 mg/kg.

Four French trials (2 reported in 1993) were evaluated in terms of Italian GAP. The application rates were 1.5 and 1.6 kg ai/ha, with 7 and 8 applications and PHIs of 8 and 10 days, conditions which were acceptably close to GAP. The residues were 1.3, 2.2, 3.7 and 8.1 mg/kg.

In summary, folpet residues in grapes from trials according to GAP were 1.6 mg/kg in Argentina, 2.6 and 3.0 mg/kg in Chile, <0.05 mg/kg in Mexico, and 0.58, 0.75, 1.3, 2.2, 3.3, 3.7 an and 8.1 mg/kg in Italy and France. The residues in rank order (median underlined) in the 11 trials were <0.05, 0.58, 0.75, 1.3, 1.6, 2.2, 2.6, 3.0, 3.3, 3.7 and 8.1 mg/kg.

The Meeting estimated maximum residue and STMR levels for grapes of 10 mg/kg and 2.2 mg/kg respectively.

GAP in Mexico permits 4 applications of folpet to strawberries at 1.3 kg ai/ha with harvest 2 days after the final application, and in The Netherlands 2 applications of 1.4 kg ai/ha and a 14-day PHI. The residues in 3 Mexican and 3 Dutch trials complying with GAP were 1.6, 1.7 and 2.2 mg/kg, and 1.4, 1.6 and 1.9 mg/kg respectively.

The Meeting noted that the results of these 6 trials were in line with the current draft MRL for strawberries of 5 mg/kg, and decided that it would be preferable to estimate an STMR when all the information on residue trials and current GAP become available for the periodic review in 1998.

GAP for onions in Chile allows 3 applications of 2 kg ai/ha and in Mexico 4 applications at 1.5 kg ai/ha, both with harvest 7 days after the final application. Folpet residues in one Chilean and two 2 Mexican trials complying with GAP were 0.36, 0.41 and 0.41 mg/kg.

Two trials in Greece and four in Hungary according to national GAP gave residues of <0.05 (3), 0.07 and 0.21 mg/kg.

The folpet residues in onions in trials in Portugal (5.0 mg/kg) and Spain (2.5 mg/kg) were somewhat higher than in other European countries (<0.05-0.21 mg/kg), and probably related to the drip irrigation system used in Portugal and Spain, whereas sprinkler irrigation is used elsewhere.

In the trials in Greece, Hungary, Portugal and Spain the field sample was described as at least 2 kg consisting of 12 or more onions. The soil was removed mechanically by hand and the whole plant, including roots and foliage, was analysed. The Meeting was informed that this sampling procedure was based on a draft EU guideline, which is unfortunately in conflict with a long-established Codex procedure. Because the correct sample for bulb onions does not include roots or foliage the Meeting could not use the data, and the 3 trials in Chile and Mexico were insufficient to estimate a maximum residue level.

The folpet residue in cucumbers was 0.07 mg/kg in a Canadian trial according to Canadian GAP (8 applications of 1.0 kg ai/ha with a PHI of 7 days), and 0.11, 0.36, 0.56 and 0.70 mg/kg in four Mexican trials complying with national GAP (1.8 kg ai/ha with harvest after the last of 4 applications).

The Meeting noted that the current draft MRL for cucumbers is 0.5 mg/kg and concluded that it would be preferable to evaluate all the residues in terms of relevant GAP at the periodic review in 1998.

In Greece folpet is registered for use on melons at 0.49 kg ai/ha with harvest 20 days after the final application (maximum 4). Folpet residues were below the LOD (<0.05 mg/kg) in melons in 4 Greek trials according to GAP and in 2 others where folpet was applied at twice the GAP rate.

Mexican GAP permits 6 applications at 1.8 kg ai/ha and harvest 7 days after the final application. The residues in 3 Mexican trials complying with GAP were 0.40, 0.89 and 2.2 mg/kg.

In two trials in Honduras according to GAP (4 applications of 0.64 kg ai/ha and a PHI of 3 days), the residues were 0.32 and 0.41 mg/kg, and in a Guatemalan trial according to GAP (6 applications of 0.48 kg ai/ha and a PHI of 3 days), the residue was 0.23 mg/kg.

In summary, folpet residues in melons from trials effectively according to GAP were <0.05 (6) in Greece, 0.40, 0.89 and 2.2 mg/kg in Mexico, 0.32 and 0.41 mg/kg in Honduras and 0.23 mg/kg in Guatemala. The residues in rank order in the 12 trials were <0.05 (6), 0.23, 0.32, 0.40, 0.41, 0.89 and 2.2 mg/kg.

As the residues in the Greek trials appear to belong to a different population from the others, the 6 trials in Mexico, Honduras and Guatemala were used to estimate an STMR.

The Meeting estimated maximum residue and STMR levels for folpet in melons of 3 mg/kg and 0.41 mg/kg respectively. The STMR in this case is for the whole melon because data were not available on residues in the edible portion.

GAP for tomatoes in Chile allows 7 applications of 1.7 kg ai/ha with a 7-day PHI, and in Mexico 5 applications at 2.0 kg ai/ha with a 2-day PHI. Folpet residues in one Chilean and 5 Mexican trials complying with GAP were 2.4 mg/kg and 0.45, 1.0, 1.3, 1.6 and 1.8 mg/kg respectively.

In Hungary folpet is registered for use on tomatoes at an application rate of 0.65 kg ai/ha with harvest 14 days after the final application (maximum of 3). In 4 Hungarian trials according to GAP and in 1 trial reported in 1993 with 5 applications at the GAP rate and PHI the residues were all below the LOD (<0.02 and <0.05 (4) mg/kg).

In one Italian and two Portuguese trials in compliance with Portuguese GAP (4 applications of 1.3 kg ai/ha and 7 days PHI) the residues were 0.34, 0.55 and 0.58 mg/kg. In a Spanish trial according to GAP (6 applications of 1.6 kg ai/ha and a 10-day PHI) the residue was 1.3 mg/kg.

In summary, folpet residues in tomatoes from trials according to GAP were 2.4 mg/kg in Chile, 0.45, 1.0, 1.3, 1.6 and 1.8 mg/kg in Mexico, <0.02 and <0.05 (4) mg/kg in Hungary, 0.55, 0.34 and 0.58 mg/kg in Portugal and Italy, and 1.3 mg/kg in Spain. The residues in rank order in the 15 trials were <0.02, <0.05 (4), 0.34, 0.45, 0.55, 0.58, 1.0, 1.3 (2), 1.6, 1.8 and 2.4 mg/kg.

The residues in the Hungarian trials appear to be in a different population from the others. The 10 trials from Chile, Portugal, Italy and Spain were used to estimate an STMR.

The Meeting estimated maximum residue and STMR levels for folpet in tomatoes of 3 mg/kg and 1.15 mg/kg respectively.

Folpet is registered in Mexico for 4 applications of 1.3 kg ai/ha to lettuce with harvest 7 days after the final application. Folpet residues were 4.5, 9.8 and 16 mg/kg in 3 Mexican trials on head lettuce with 5 applications at the GAP rate and PHI, and 22 mg/kg in one trial on leaf lettuce under the same conditions.

Folpet residues were not detected (<0.05 mg/kg) in head or leaf lettuce in 2 trials in Greece according to Greek GAP (4 applications of 0.61 kg ai/ha and 20 days PHI), except that only 3 applications were made to head lettuce. No residue was detected (<0.05 mg/kg) in leaf lettuce in a Spanish trial according to GAP (4 applications of 0.78 kg ai/ha and 21 days PHI).

In summary, folpet residues in head lettuce were 4.5, 9.8 and 16 mg/kg in Mexico and <0.05 mg/kg in Greece, and in leaf lettuce 22 mg/kg in Mexico, <0.05 mg/kg in Greece and <0.05 mg/kg in Spain. The data populations in Mexico and Europe appear to be different. There were too few results to make a recommendation.

Processing

Field-treated apples were processed to juice and wet pomace to simulate commercial practice as closely as possible. The process included an initial washing step which removed about 40% of the residue. The processing factors for the production of wet pomace and apple juice were 2.6 and 0.035 respectively.

The STMR-Ps for the processed apple commodities calculated from the processing factors and the STMR for apples (1.8 mg/kg) are wet apple pomace 4.7 mg/kg and apple juice 0.063 mg/kg.

Grapes were treated post-harvest by dipping bunches for 30 seconds in a vat containing folpet (1.25 kg ai/hl). The grapes were allowed to dry and then processed into raisins and juice. Because folpet is a surface residue it was considered valid to treat grapes in this way.

The treated grapes were dried in the sun until the moisture level reached 12-16%. After destemming, the dried grapes were rehydrated to 18-20% moisture in an incubator at 21°C to produce raisins. Juice was produced from treated grapes by crushing, enzyme treatment, heating and filtering.

Folpet residues were not detectable (<0.05 mg/kg) in the grape juice. The calculated processing factor for juice is <0.003. Folpet residues in the dried and hydrated raisins were higher than in the original grapes, with processing factors of 3.2 and 1.9 respectively.

The Meeting estimated a maximum residue level for folpet residues in dried grapes or raisins of 40 mg/kg after rounding up, from the processing factor of 3.2 and the maximum residue level estimated for grapes (10 mg/kg).

The STMR-P levels calculated from the processing factors and the STMR for grapes (2.2 mg/kg) are grape juice 0.0066 mg/kg, dried raisins 7.0 mg/kg, and hydrated raisins 4.2 mg/kg.

In 10 trials on grapes in Germany in 1993 residues of folpet were measured in the must and wine produced from treated grapes. The processing factors for folpet transfer from grapes to must ranged from 0 to 0.97, mean 0.29. Folpet was not detected (<0.05 mg/kg) in any wine sample, hence the processing factor for wine is 0. Phthalimide, a metabolite and breakdown product of folpet, was consistently present in both must and wine.

The STMR-P for must calculated from the mean processing factor and the STMR for grapes (2.2 mg/kg) is 0.64 mg/kg.

The Meeting noted that the use of folpet on grapes consistently results in phthalimide residues in wine at levels typically 25-50% of the folpet levels in the grapes. The metabolism study on grapes had shown the formation of a water-soluble conjugate of phthalic acid in grapes which also has the potential to reach the wine.

A tomato crop was treated 5 times with folpet at 2.2 kg ai/ha and harvested 7 days after the final application for processing. The tomatoes were treated in 0.5% sodium hydroxide and then vigorously washed before being processed to juice, purée and paste. Purée was produced from juice by evaporation, adjustment of salt and water levels, heating and canning. Paste was produced similarly, but with a higher salt level.

Folpet residues were not detected (<0.05 mg/kg) in tomato purée or paste produced from tomatoes containing 1.8 mg/kg of folpet. It is quite likely that the initial vigorous cleaning of the tomatoes would remove or destroy most of the folpet residues. The calculated processing factor for the transfer of folpet from tomatoes to purée and paste is <0.028, and the STMR-P calculated from the STMR for tomatoes of 1.15 mg/kg is 0.032 mg/kg.

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 (for compliance with MRL and for estimation of dietary intake): folpet.

Commodity		Recommended MRL, mg/kg		Based on PHI, days	STMR, mg/kg	STMR-P, mg/kg
CCN	Name	New	Previous			
FP 0226	Apple	10	-	7-21	1.8	
DF 0269	Dried grapes (currants, raisins and sultanas)	40				7.0
FB 0269	Grapes	10	2	7-14	2.2	
VC 0046	Melons, except Watermelon	3	-	3-7	0.41	
VO 0448	Tomato	3	-	2-10	1.15	
	Apple juice					0.063
	Apple pomace, wet					4.7
	Grape juice					0.0066

Commodity		Recommended MRL, mg/kg		Based on PHI, days	STMR, mg/kg	STMR-P, mg/kg
CCN	Name	New	Previous			
	Must					0.64
	Raisins, hydrated					4.2
	Tomato paste					0.032
	Tomato purée					0.032
	Wine					0

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GLYPHOSATE (158)

EXPLANATION

Glyphosate was first evaluated by the 1986 JMPR; an ADI of 0.3 mg/kg bw was allocated and maximum residue levels were estimated for some cereal grains and straw, vegetables, oilseeds and animal products.

In 1987 a revised analytical method for glyphosate residues and residue data on milled products and processed cereal commodities were evaluated. MRLs were recommended for kiwifruit, soya beans, soya bean fodder and forage, wheat and wheat bran.

The 1988 JMPR re-evaluated data on wheat, barely and oats resulting from pre-harvest applications of glyphosate and recommended new or revised MRLs for wheat and its milling fractions.

The 1994 JMPR evaluated data on residues resulting from new pre-harvest uses of glyphosate and recommended revised MRLs for soya bean, soya bean fodder, and unprocessed wheat bran.

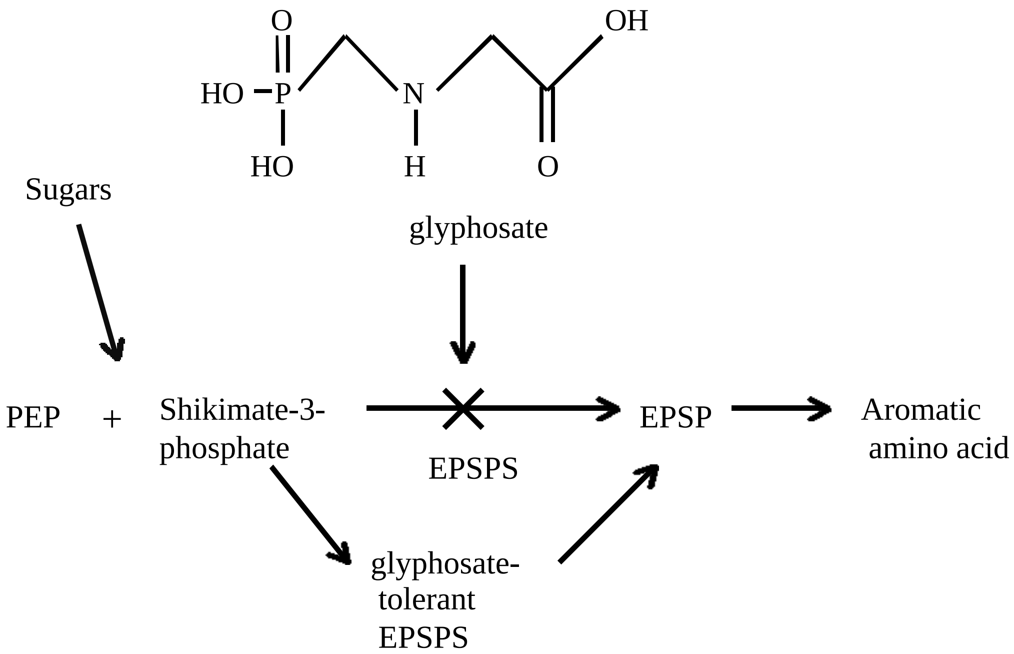
The 1997 JMPR was requested to evaluate the new uses of glyphosate on cotton, maize and sorghum according to GAP. These new uses are (1) pre-harvest topical applications and (2) in-crop applications to cotton and maize crops which have been genetically modified to be resistant to glyphosate. Relevant data on metabolism and residue trials were submitted to the Meeting.

Genetic modification of crops

Glyphosate binds to and blocks the activity of 5-enolpyruvoyl-shikimate-3-phosphate synthase (EPSPS), an enzyme of the aromatic amino acid biosynthetic pathway. The inhibition of EPSPS hyglyphosate prevents the plant from producing the aromatic amino acids essential for protein synthesis. EPSPS is present in all plants, bacteria, and fungi but not in animals, which do not synthesize aromatic amino acids, but receive them from food.

The development of glyphosate-resistant (glyphosate-tolerant) crops has been in progress since the early 1980s, using a “target-site modification” approach in which a glyphosate-resistant EPSPS was identified and its expression induced in plants by genetic manipulation. Glyphosate treatment leaves the plant unaffected because the continued action of the glyphosate-resistant EPSPS enzyme supplies the plant’s need for aromatic amino acids (Figure 1).

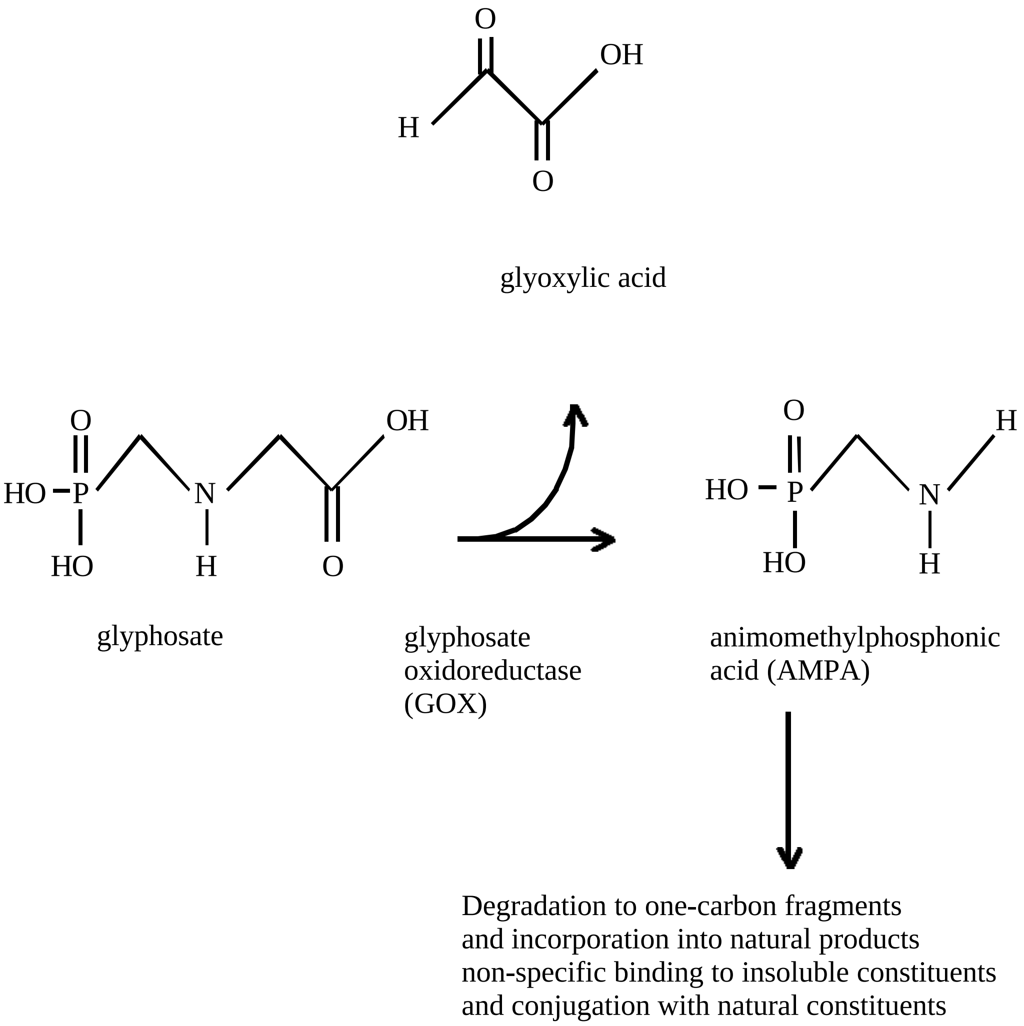
Figure 1. Action point of glyphosate and mechanism of glyphosate resistance.



A search was instituted to identify naturally-occurring EPSPS with not only a high degree of glyphosate resistance, but also tight binding of the substrate phosphoenolypyruvate (PEP). The EPSPS enzyme identified is derived from *Agrobacterium* sp. Strain (CP4 EPSPS), and has been used to develop glyphosate-resistant crops.

While the CP4 EPSPS enzyme has been successful in providing glyphosate resistance in cotton, the activity of CP4 EPSPS alone has been insufficient to ensure adequate resistance in other crops. In maize, a second mechanism of resistance has been developed to allow applications of glyphosate at rates of use necessary for effective weed control. The second mechanism is glyphosate inactivation, which reduces cellular levels of glyphosate by converting it to aminomethylphosphonic acid (AMPA). The inactivating enzyme is glyphosate oxidoreductase (*gox*). The gene encoding *gox* was isolated from a naturally-occurring bacterium. *Achromobacter* sp., and has been modified to optimize its expression in plants. The inactivation process is shown in Figure 2.

Figure 2. Mechanism of glyphosate inactivation in plants.



PLANT METABOLISM

A number of metabolism studies with vegetables, orchard tree, nut and pasture crops were reported to the 1986 JMPR. The 1986 Meeting concluded that glyphosate applied to the soil was absorbed to a very small extent or not at all by the crops and conversion of glyphosate to aminomethylphosphonic acid (AMPA), which is the primary metabolite, was not observed.

Hydroponic administration allows sufficient uptake of glyphosate to elucidate the metabolic transformation in plants. Metabolic studies with the hydroponic administration of glyphosate to maize, wheat, cotton and soya beans showed the conversion of glyphosate to AMPA and further degradation in plant tissues.

Metabolic studies with plants that had been genetically modified to be resistant to glyphosate showed that the metabolism was the same as in susceptible plants: glyphosate is metabolized to AMPA, which is either non-selectively bound to natural plant constituents, further degraded to one-carbon fragments that are incorporated into natural products, or conjugated with naturally-occurring organic acids to give trace-level metabolites. The metabolites are the same in resistant and non-resistant crops; only the relative distribution varies, depending on the speed and extent to which glyphosate is converted to AMPA.

Metabolism in susceptible plants

Maize, cotton, wheat and soya beans were treated with [^{14}C]glyphosate, labelled in the phosphonomethyl group, added to the hydroponic solution (Rueppel, 1973). In order to define the metabolism more completely, glyphosate labelled at the carboxyl- and α-carbon atoms has also been hydroponically administered to soya beans.

In the hydroponic uptake experiments, which lasted for 28 days for all crops except wheat (10 days), the composition of the ^{14}C-labelled residue in both the aqueous plant extracts and the nutrient media has been identified as a function of time on the basis of TLC and/or column chromatography with LSC.

The forage from all four crops could be efficiently extracted with water; 70-90% of the radioactivity was extractable, so the isolated compounds probably include all the major metabolites. The main ^{14}C-labelled compound was glyphosate in all the forage except maize.

The major metabolite (4-28%) of [^{14}C]glyphosate was chromatographically identified as *N*-methylaminomethylphosphonic acid, but its presence was considered to be an artifact on the basis of uptake studies using highly purified [*phosphonomethyl*-^{14}C]glyphosate.

The conclusions were as follows.

1. Significant degradation of glyphosate occurs in plants; AMPA also appears to be degraded. The high extractability indicates that conjugation of glyphosate and AMPA with natural plant components represents a minor pathway at most.

2. The major metabolic pathway of degradation of glyphosate probably involves the formation of AMPA and glyoxylate by enzymatic cleavage of the C-N bond. There is significant incorporation of glyoxylate, aminomethylphosphonate fragments, and/or CO_2 into natural products.

The residues found in maize, wheat, cotton and soya bean forage are shown in Table 1.

Table 1. Metabolites found in maize, wheat, cotton and soya bean forage after hydroponic application of [^{14}C]glyphosate.

Crop	% of the TRR					
	Glyphosate[1]	AMPA[1]	N-MAPA[1,2]	Natural products	Unidentified [2]	Unextractable
Maize	21.1	27.9	NA	4.0	20.0	26.6
Wheat	55.3	4.2	NA	1.0	8.0	31.5
Cotton	61.5	6.8	2.0	8.8	10.9	10.0
Soya bean	69.2	9.0	1.1	9.0	2.3	9.5

[1]*N*-methylaminomethylphosphonic acid
[2]Defined as extractable ^{14}C lost during the analytical procedure

Metabolism in resistant cotton

Cotton that has been genetically modified to be resistant to glyphosate contains the CP4 EPSPS gene. In a study of the metabolism of glyphosate in resistant cotton plants two application of [^{14}C]glyphosate were made to test plots at the 3-4 leaf stage (42 days after planting) and at the 5-6 leaf stage (51 days after planting). The target rates were 0.93 kg/ha for the first application and 1.26 kg/ha for the second application (Bleeke, 1997). The timing and application rates were according to expected GAP for over-the-top treatments. In order to distinguish between radioactive residues resulting from the metabolism of [^{14}C]glyphosate within the plant and those resulting from the uptake of $^{14}Co_z$ formed by microbial degradation of [^{14}C]glyphosate in the soil, the plants were divided into two treatment groups. In one group the soil was covered during application to minimize contact of the test substance with the soil, but was left uncovered in the other.

Crop samples were collected at the forage stage (78 days after planting, or 27 days after the second application) and at maturity (209 days after planting, or 158 days after the second application). At the final harvest the plants were separated into seed, lint, and stalk fractions. The forage samples were extracted with water and the seed samples first with hexane to remove oil, then with 50% acetronitrile in water. The total radioactivity in the forage and seed and its distribution after extraction are shown in Table 2.

Table 2. Distribution of radioactivity in extracts of forage and seed of glyphosate-resistant cotton after treatment with [^{14}C]glyphosate.

Sample	PHI, days	TRR, mg/kg, as glyphosate	[^{14}C], mg/kg (% of TRR)			Total Recovery, %
			Hexane fraction	Aqueous fraction	Unextracted	
TP forage	27	30.4	NA	30.0 (98.5)	0.447 (1.5)	100.0
TU forage	27	15.2	NA	14.7 (96.9)	0.708 (4.7)	101.6
CP forage	27	0.008	NA	NA	NA	NA
CU forage	27	0.039	NA	NA	NA	NA
TP seed	158	0.107	0.012 (11.3)	0.034 (31.9)	0.058 (54.1)	97.3
TU seed	158	0.181	0.027 (14.7)	0.034 (18.6)	0.136 (75.4)	108.7
CP seed	158	0.018	NA	NA	NA	NA
CU seed	158	0.070	0.016 (22.9)	0.006 (8.8)	0.053 (76.1)	107.8

TP: treated protected TU: treated unprotected
CP: control protected CU: control unprotected
NA: not analysed

The total radioactive residues in the forage were 15-30 mg/kg (glyphosate equivalents), but in the seed they were <0.2 mg/kg. There were also relatively high levels of radioactivity in the final harvest control samples (treated with unlabelled glyphosate), particularly in those from the unprotected plots. The level of ^{14}C in the control samples indicated that the uptake of soil-generated $^{14}Co_2$ and its incorporation into the plant made a significant contribution to the total radioactive residues at harvest.

In the plants grown on protected soil the total radioactive residues in the forage were about 30 mg/kg and in the seed about 0.1 mg/kg. In the forage >95% of the TRR was extractable and in the seed hexane and water extracted about 11% and 32% respectively; the unextractable residues accounted for 54%. The extracts were analysed by HPLC with the results shown in Table 3.

Table 3. Compounds identified and characterized in the forage and seed of glyphosate-resistant cotton after foliar application of [^{14}C]glyphosate.

Sample	Compound/or type	TP forage		TP seed	
		% of TRR	mg/kg[1]	% of TRR	mg/kg[1]
Hexane extract	Saponifiable fatty acids	NA	NA	10.4	0.011
	Total	NA	NA	11.3	0.012
	Glyphosate	95.7	29.1	23.7	0.025
	AMPA	0.66	0.201	1.38	0.001
Aqueous extract	Conjugates	0.29	0.087	NA	NA
	Natural products	0.40	0.123	6.93	0.007
	Total	98.5	30.0	31.9	0.034
Seed after hexane and aqueous extraction	Base-extractable	NA	NA	10.7	0.011
	Unextracted	NA	NA	43.4	0.047
	Total	NA	NA	54.1	0.058

[1]As glyphosate

The primary route of metabolism in glyphosate-resistant cotton plants is the gradual conversion of glyphosate to aminomethylphosphonic acid (AMPA), as in susceptible crops. In the forage, glyphosate accounted for almost all the residue with small amounts of AMPA, and in the seed glyphosate constituted more than 64% of the water-extractable residues, or 12-25% of the total radioactive residues. The radioactivity found in the oil and bound in the seed was characterized as being associated with natural products.

Metabolism in resistant maize

Maize that has been genetically modified to be resistant to glyphosate contains both the CP4 EPSPS and *gox* genes. To study its metabolism of glyphosate two post-emergence applications of 0.9 and 0.84 kg glyphosate/ha were made at 43 and 73 days after planting (George, 1995). The timing and application rates are expected to become GAP for over-the-top treatments. Again some plots were protected to prevent uptake by the plants of $^{14}Co_z$ formed by microbial degradation of [^{14}C]glyphosate in the soil.

Crop samples were collected according to normal agricultural practices. Forage and silage samples were collected 3 and 49-53 days after the second application respectively, and fodder and grain samples 83 days after the second application at normal harvest. Ground forage, silage and fodder samples were extracted with water. Ground grain samples were extracted first with hexane to remove oil, then with water. The total radioactivity found in the forage, silage, fodder and grain and its distribution between extracted and unextracted fractions are shown in Table 4. No significant differences were found between the uncovered and covered soil groups, indicating that the radioactivity in the plants was mainly derived from the applied [^{14}C]glyphosate.

Table 4. Distribution of radioactivity in forage, silage, fodder and grain of glyphosate-resistant maize after treatment with [^{14}C]glyphosate.

Sample	PHI, days	TRR, mg/kg	mg/kg , % of TRR		Total recovery, %
			Extracted	Unextracted	
TP forage	3	13.3	12.8 (96.2)	0.38 (2.9)	99.1
TP silage	49-53	9.11	8.52 (93.5)	0.40 (4.4)	97.9
TP fodder	83	14.9	14.2 (95.2)	0.68 (4.5)	99.7
TP grain	83	0.685	0.54 (79.2)	0.14 (20.9)	100.1
TU forage	3	10.8	10.0 (93.0)	0.31 (2.8)	95.8

Sample	PHI, days	TRR, mg/kg	mg/kg , % of TRR		Total recovery, %
			Extracted	Unextracted	
TU silage	49-53	9.59	8.3 (86.8)	0.43 (4.5)	91.3
TU fodder	83	19.1	18.0 (94.4)	1.0 (5.4)	99.8
TU grain	83	1.04	0.84 (81.1)	0.24 (23.2)	104.3

TP: treated protected TU: treated unprotected
CP: control protected CU: control unprotected
NA: not analysed

The total radioactive residues in the forage, silage and fodder of the two groups were 9.11 to 19.1 mg/kg glyphosate equivalents, and in the grain much lower at about 0.7 mg/kg. About 90% of the TRR could be extracted from all samples except grain from which hexane and water extracted about 1% and 80% respectively, with 20-23% remaining in the extracted grain. The aqueous extracts were analysed by HPLC with the results shown in Table 5.

Table 5. Components of the residue found in aqueous extracts of glyphosate-resistant maize forage, fodder and grain after foliar application of [^{14}C]glyphosate.

Metabolite or characterization	mg/kg, (% of ^{14}C in extract)			
	Forage	Silage	Fodder	Grain
Glyphosate	7.77 (71.9)	6.43 (67.1)	14.27 (74.8)	0.03 (2.6)
Glyphosate conjugates	0.04 (0.4)	0.04 (0.4)	0.36 (1.9)	NA
AMPA	1.72 (15.9)	1.26 (13.1)	2.13 (11.2)	0.63 (60.3)
N-glyceryl-AMPA	0.06 (0.5)	0.14 (1.5)	0.31 (1.6)	0.07 (6.9)
Saponifiable fatty acids	NA	NA	NA	0.01 (1.0)
Starch	NA	NA	NA	0.22 (20.9)
Natural products	0.24 (2.2)	0.34 (3.5)	0.65 (3.4)	0.04 (3.6)
Total	9.8 (90.9)	8.2 (85.6)	17.7 (92.9)	1.0 (95.3)

The metabolism of glyphosate in resistant maize, as in cotton, follows the same pathway as in susceptible crops. The results of the study show that glyphosate is gradually metabolized to AMPA and low levels of AMPA conjugates. In grain, AMPA is the major component of the residue, with only trace levels of glyphosate remaining. Grain also contained significant levels of bound radioactivity (about 19% of the TRR). Because glyphosate is rapidly degraded to AMPA owing to the presence of the *gox* protein, further reactions of AMPA become important. These result in higher levels of AMPA conjugates, bound AMPA residues, and natural products derived from the degradation of AMPA to one-carbon fragments.

METHODS OF RESIDUE ANALYSIS

Glyphosate and its major metabolite, AMPA, can be determined by GLC or HPLC after derivatization.

Samples from supervised trials on susceptible cotton were analysed by the GLC method employing anion and cation exchange resin and carbon clean-up, followed by trifluoroacetylation and methylation, which was evaluated by the 1986 JMPR.

The limit of determination was 0.05 mg/kg in cotton seed and hay and the recoveries at 0.05-0.4 mg/kg of glyphosate and AMPA respectively were 66.3-89.4% and 66.0-84.9% in the hay and 56.7-74.8% and 63.4-93.2% in the seed.

Samples from supervised trials on resistant cotton, resistant and susceptible maize, and susceptible sorghum were analysed by two-column switched HPLC with a post-column reactor. The method was evaluated by the 1994 JMPR.

The limit of determination was 0.05 mg/kg in all commodities and the mean recoveries were as shown in Table 6.

Table 6. Fortification levels and mean recoveries of glyphosate and AMPA in several commodities.

	Cotton[1]		Maize[2]		Maize[1]			Sorghum[2]		
	Seed	Gin	Grain	Fodder	Grain	Fodder	Forage	Grain	Fodder	Hay
Spike, mg/kg	0.06-50	0.03-100	0.05-2.0	0.05-200	0.05-25	0.05-50	0.05-30	0.05-3.0	0.05-20.0	0.05-10.0
Glyphosate mean recovery, %	86.33	84.59	77.36	82.30	86.28	88.38	83.43	87.24	83.70	83.60
AMPA Mean recovery, %	87.31	82.59	82.63	84.19	89.88	88.02	82.21	90.25	78.13	79.39

[1]Resistant
[2]Susceptible

USE PATTERN

Glyphosate is a leaf-absorbed, non-selective systemic herbicide which is used for the control of unwanted vegetation. Major international use patterns include pre-planting application to most crops, directed spray in tree and vine crops, silvicultureal site preparation and conifer release, fallow and reduced tillage systems, general land management in non-crop situations, and pre-harvest application to cereals and oilseeds. The rate of application depends on the type and size of the weeds and the degree of control required.

Table 7 shows the use patterns which have recently been approved in the USA for applications to cotton, maize and sorghum.

Table 7. Registered uses of glyphosate on cotton (susceptible and resistant), maize (susceptible and resistant) and sorghum (susceptible) in the USA.

Crop	Application			PHI, days	Remarks
	Type	No.[1]	Rate, kg/ha[2]		
Cotton	Pre-plant, pre-emergence at-planting		0.32-4.2		Applications must be made before emergence.
	Hooded sprayer selective equipment		0.32-4.2	7	
	Spot treatment		0.32-4.2	7	Apply before boll opening.
	Pre-harvest	1	0.32-4.2	7	Apply after sufficient bolls have developed to produce the desired yield of cotton.
Cotton (glyphosate resistant)	Pre-plant, pre-emergence at-planting		0.32-4.2		
	In-crop	2	0.84	7	Apply from ground cracking to pinhead square stage. Applications must be at least 10 days apart.
	Post-directed hooded sprayer	2	0.84	7	Applications must be at least 10 days apart.
	Pre-harvest	1	1.7	7	Applications must be at least 10 days apart.

Crop	Application			PHI, days	Remarks
	Type	No.[1]	Rate, kg/ha[2]		
Maize	Pre-plant, pre-emergence at-planting		0.32-4.2		Applications must be made before emergence.
	Spot treatment		0.32-4.2	7	Apply before silking of maize.
	Pre-harvest (ground)	1	2.5	7	Apply at 35% grain moisture or less.
	Pre-harvest (aerial)	1	0.84	7	Ensure that maximum kernel fill is complete and the maize is physiologically mature.
	Post-harvest		0.32-4.2		
Maize (glyphosate resistant)	Pre-plant, pre-emergence		0.32-4.2		
	In-crop	2	0.84	7 50	Apply from emergence to 12-leaf stage or 30 inches plant height. Applications must be at least 14 days apart. PHI of 50 days is for forage.
	Pre-harvest	1	0.84	7 50	PHI of 50 days is for forage.
	Post-harvest		0.32-4.2		
Sorghum	Pre-plant, pre-emergence at-planting		0.32-4.2		Applications must be made before emergence.
	Spot treatment, wiper		0.32-4.2	7 40	PHI of 40 days is for wiper application.
	Pre-harvest	1	1.7	7	Apply at 30% grain moisture or less.
	Post-harvest		0.32-4.2		

[1]The combined total of all treatments must not exceed 6.7 kg/ha per year if number of applications is not indicated
[2]Expressed as glyphosate acid (*N*-(phosphonomethyl)glycine)

RESIDUES RESULTING FROM SUPERVISED TRIALS

The results of supervised trials on susceptible cotton, maize and sorghum are shown in Tables 8-15.

Cotton. Twelve supervised trials on susceptible cotton were reported to the Meeting. The results are shown in Table 8. The underlined residues are from treatments according to GAP and double-underlined residues have been used for the estimation of STMRs. The application patterns in the trials were as follows.

Application (glyphosate acid)	Pre-emergence, kg/ha	Post-emergence, kg/ha	Pre-harvest, kg/ha	Total application, kg/ha
Type 1	6.7	--	3.4	10
Type 2	6.7	2 x 5.0	3.4	20
Type 3	6.7	3 x 5.0	3.4	25
Type 4	2 x 6.7	8.9 (from 3 applicns.)	3.4	26

Table 8. Residues of glyphosate and AMPA in susceptible cotton seed and hay from supervised trials in the USA in 1974 and 1975. SL 41% formulation (Baszis, 1980).

Location	Application[1]			PHI, days	Residues, mg/kg					
	No. (treatment)	kg ai/ha[2]	kg ai/ha[3]		Glyphosate		AMPA		Total[4]	
					Seed	Hay	Seed	Hay	Seed	Hay
Malone, Florida	2 (1)	10	1.2	9	0.54	15	≤0.05	0.11	0.62	15
Ropesville, Texas	2 (1)	10	1.6	13	3.6	29	0.06	0.17	3.7	29
Sasser, Georgia	4 (2)	20	1.5	9	2.7	7.4	0.08	0.08	2.8	7.5
Mount Pleasant Mississippi	5 (3)	25	0.52	13	2.9	18	0.07	0.13	3.0	18
St.Joseph Louisiana	6 (type 4)	26	1.8	9	5.9	33	0.07	0.24	6.0	33
Five Points California	2 (1)	10	1.1	13	1.9	17	0.05	0.18	2.0	17
Kerman	1	3.7	1.5	3	1.5	98	0.05	0.46	1.6	99
California				10	0.49	84	0.06	0.45	0.58	85
Weldon	1	3.7	2.8	3	2.0	120	<0.05	0.83	2.1	120
North Carolina				10	0.92	11	0.20	0.09	1.2	11
Cheneyville	1	3.7	1.5	7	2.3	24	≤0.05	0.20	2.4	24
Louisiana				14	0.15	11	<0.05	0.10	0.23	11
Sledge	1	3.7	1.8	5	0.63	20	≤0.05	0.18	0.71	20
Mississippi				10	0.27	6.3	<0.05	0.09	0.35	6.4
Ropesville	1	3.7	1.5	3	0.47	60	<0.05	0.21	0.55	60
Texas				10	0.26	19	<0.05	0.20	0.34	19
Dawson	1	3.7	1.2	3	4.1	27	<0.05	0.44	4.2	28
Georgia				8	2.9	3.8	0.07	0.21	3.0	4.1

[1]Expressed as glyphosate acid, *N*-(phosphonomethyl)glycine
[2]Total of all applications
[3]Concentration of last application
[4]Glyphosate + AMPA expressed as glyphosate

Forty eight supervised trials on susceptible cotton were reported to the Meeting. The results are shown in Tables 9 and 10. The application patterns were as shown below.

Application (glyphosate acid)	**Pre-emergence, kg/ha 3-4 leaf**	**Post-emergence, kg/ha 5-6 leaf**	**Post-emergence, kg/ha 7-8 leaf**	**Post-emergence, kg/ha**	**Post-directed, kg/ha**	**Pre-harvest, kg/ha**	**Total Application, kg/ha**
Type 1	3.36	1.26			1.26	1.68	7.56
Type 2	3.36	0.84	1.26		1.26	1.68	8.4
Type 3	3.36	0.84		1.26	1.68	1.68	8.82

Table 9. Residues of glyphosate and AMPA in seed and gin from susceptible cotton (genotype 1445) from supervised trials in the USA in 1994 (Oppenhuizen, 1995a). SL 41% formulation.

Location	Application[1]			PHI, days	Residues, mg/kg					
	No. (type)	kg ai/ha[2]	kg ai/hl[3]		Glyphosate		AMPA		Total[4]	
					Seed	Gin	Seed	Gin	Seed	Gin
Alabama	4 (1)	7.6	0.90	7	0.46	9.7	<0.05	0.09	0.54	9.8
	5 (2)	8.5	0.90	7	0.47	8.3	<0.05	0.08	0.55	8.4
	5 (3)	8.9	0.90	7	0.44	16	<0.05	0.18	0.52	16
Arkansas	4 (1)	7.5	1.4	8	0.41	4.3	0.21	0.05	0.73	4.4
	5 (2)	8.1	1.4	8	0.33	7.2	0.06	0.07	0.42	7.3
	5 (3)	8.5	1.4	8	0.43	8.6	<0.05	0.08	0.51	8.7
Arizona	4 (1)	7.1	1.0	7	1.3	41	0.06	0.28	1.4	41
	5 (2)	7.9	1.0	7	0.43	23	<0.05	0.20	0.51	23
	5 (3)	8.3	1.0	7	1.0	32	<0.05	0.19	1.1	32
California	4 (1)	7.7	1.3	6	2.8	36	0.08	0.39	2.9	37
	5 (2)	8.5	1.3	6	2.0	26	0.07	0.25	2.1	26
	5 (3)	9.0	1.3	6	1.4	20	0.08	0.18	1.5	20
Louisiana	4 (1)	7.6	1.8	17	0.43	1.2	<0.05	<0.05	0.51	1.3
	5 (2)	8.4	1.8	17	0.50	0.79	0.05	<0.05	0.58	0.87
	5 (3)	8.8	1.8	17	0.40	0.86	<0.05	<0.05	0.48	0.94
Mississippi	4 (1)	7.6	1.2	6	3.6	30	0.07	0.26	3.7	30
(Bolivar)	5 (2)	8.4	1.2	6	3.4	31	0.05	0.23	3.5	31
	5 (3)	8.9	1.2	6	0.69	27	<0.05	0.13	0.77	27
Mississippi	4 (1)	7.7	1.2	9	0.22	5.8	<0.05	0.05	0.30	5.9
(Washington)	5 (2)	8.5	1.2	9	0.41	3.7	<0.05	0.05	0.49	3.8
	5 (3)	8.9	1.2	9	0.13	4.0	<0.05	0.05	0.21	4.1
Tennessee	4 (1)	7.7	1.4	8	3.6	34	0.10	0.45	3.8	35
	5 (2)	8.4	1.4	8	5.0	26	0.13	0.26	5.2	26
	5 (3)	8.8	1.4	8	2.5	29	<0.05	0.27	2.6	29
Texas	4 (1)	7.6	1.5	6	2.0	33	<0.05	0.30	2.1	33
(Uvalde)	5 (2)	8.4	1.5	6	2.2	84	<0.05	0.84	2.3	85
	5 (3)	8.9	1.5	6	2.4	32	<0.05	0.33	2.5	33
Texas	4 (1)	7.9	0.92	8	2.5	18	<0.05	0.21	2.6	18
(Willacy)	5 (2)	8.5	0.92	8	2.7	21	<0.05	0.31	2.8	22
	5 (3)	9.0	0.92	8	3.1	19	<0.05	0.25	3.2	19
Texas	4 (1)	7.5	1.2	7	1.7	29	<0.05	0.12	1.8	29
(Hockley)	5 (2)	8.3	1.2	7	2.8	42	<0.05	0.25	2.9	42
	5 (3)	8.7	1.2	7	4.6	31	<0.05	0.15	4.7	31

[1]Expressed as glyphosate acid, *N*-(phosphonomethyl)glycine
[2]Total of all applications
[3]Concentration of last application
[4]Glyphosate + AMPA expressed as glyphosate

Table 10. Residues of glyphosate and AMPA in seed and gin from resistant cotton (genotype 1698) from supervised trials in the USA in 1994 (Oppenhuizen, 1995a). SL 41% formulation.

Location	Application[1]			PHI, days	Residues (mg/kg)					
	No. (type)	kg ai/ha[2]	kg ai/hl[3]		Glyphosate		AMPA		Total	
					Seed	Gin	Seed	Gin	Seed	Gin
Arkansas	4 (1)	7.5	1.4	8	0.67	4.0	<0.05	0.08	0.75	4.1
	5 (2)	8.1	1.4	8	0.58	7.8	<0.05	0.09	0.66	7.9

Location	Application[1]			PHI, days	Residues (mg/kg)					
	No. (type)	kg ai/ha[2]	kg ai/hl[3]		Glyphosate		AMPA		Total	
					Seed	Gin	Seed	Gin	Seed	Gin
	5 (3)	8.5	1.4	8	0.69	6.2	≤0.05	0.10	0.77	6.4
Louisiana	4 (1)	7.6	1.8	17	0.44	2.2	<0.05	<0.05	0.52	2.3
	5 (2)	8.4	1.8	17	0.31	2.0	<0.05	<0.05	0.39	2.1
	5 (3)	8.7	1.8	17	0.30	0.99	<0.05	<0.05	0.38	1.1
Mississippi	4 (1)	7.7	1.2	9	1.2	5.0	≤0.05	0.05	1.3	5.1
(Washington)	5 (2)	8.5	1.2	9	0.98	4.3	≤0.05	≤0.05	1.1	4.4
	5 (3)	8.9	1.2	9	0.60	5.1	≤0.05	0.05	0.68	5.2
Texas	4 (1)	7.6	1.5	6	2.5	26	≤0.05	0.31	2.6	26
(Uvalde)	5 (2)	8.5	1.5	6	2.1	65	≤0.05	0.54	2.2	66
	5 (3)	8.9	1.5	6	1.8	74	0.05	0.67	1.9	75
Texas	4 (1)	7.6	0.92	8	4.0	15	0.14	0.18	4.2	15
(Willacy)	5 (2)	8.5	0.92	8	4.2	16	0.14	0.15	4.4	16
	5 (3)	9.0	0.92	8	4.0	18	0.13	0.16	4.2	18

[1]Expressed as glyphosate acid, *N*-(phosphonomethyl)glycine
[2]Total of all applications
[3]Concentration of last application
[4]Glyphosate + AMPA expressed as glyphosate

Twelve supervised trials on susceptible maize were carried out in the USA in 1993, with the results shown in Table 11

Table 11. Residues of glyphosate and AMPA in susceptible maize grain and fodder from supervised trials in the USA in 1993 (Oppenhuizen, 1995b). SL 41% formulation.

Location	Application			PHI, days	Residues, mg/kg					
	No.	kg ai/ha	kg ai/hl		Glyphosate		AMPA		Total	
					Grain	Fodder	Grain	Fodder	Grain	Fodder
Illinois	1	2.5	1.8	6	0.07	23	0.08	0.66	0.19	24
Indiana	1	2.5	2.1	7	≤0.05	8.5	≤0.05	0.26	≤0.13	8.9
Iowa	1	2.5	1.3	7	≤0.05	28	0.13	0.41	0.25	29
Kentucky	1	2.5	1.9	7	0.05	43	0.06	0.52	0.14	44
Michigan	1	2.6	1.7	6	≤0.05	3.7	≤0.05	0.09	≤0.13	3.8
Minnesota	1	2.5	1.3	6	0.19	82	≤0.05	0.46	0.27	83
Missouri	1	2.5	1.6	6	0.05	8.8	≤0.05	0.13	0.13	9.0
Nebraska	1	2.5	2.7	6	≤0.05	92	≤0.05	0.81	≤0.13	93
Ohio	1	2.5	1.6	6	0.54	11	≤0.05	0.14	0.62	11
South Dakota	1	2.5	2.5	7	≤0.05	55	≤0.05	0.33	≤0.13	56
Texas	1	2.5	1.4	6	≤0.05	54	0.12	0.61	0.23	55
Wisconsin	1	2.5	1.4	7	≤0.05	18	≤0.05	0.12	≤0.13	18

[1]Expressed as glyphosate acid, *N*-(phosphonomethyl)glycine
[2]Total of all applications

Sixty six supervised trials on resistant maize in the USA in 1994 were reported to the Meeting. The results are shown in Tables 12 and 13. The application patterns in the trials were as follows.

Treatment type	Pre-emergence, kg/ha	Early post-emergence, kg/ha	Late post-emergence, kg/ha	Pre-harvest, kg/ha	Total application, kg/ha
Type 1	6.38	0.84			7.2
Type 2	6.38	0.84	0.84		8.1
Type 3	6.38	0.84	0.84	0.84	8.9

Table 12. Residues of glyphosate and AMPA in resistant maize grain and fodder from supervised trials in the USA in 1994 (Oppenhuizen, 1995d). SL 41% formulation.

Location	Application[1]			PHI, days	Residues, mg/kg					
	No. (type)	kg ai/ha[2]	kg ai/hl[3]		glyphosate		AMPA		Total[4]	
					Grain	Fodder	Grain	Fodder	Grain	Fodder
Colorado	2 (1)	7.2	0.50	124	<0.05	<0.05	<0.05	<0.05	<0.13	<0.13
	3 (2)	8.1	0.53	99	<0.05	0.16	<0.05	0.11	<0.13	0.33
	4 (3)	8.9	0.50	6	0.34	8.2	<0.05	0.17	0.42	8.5
Iowa	2 (1)	7.3	0.45	132	<0.05	<0.05	<0.05	0.05	<0.13	0.13
(Butler)	3 (2)	8.1	0.45	117	<0.05	0.09	0.08	0.06	0.17	0.18
	4 (3)	9.0	0.44	7	<0.05	20	0.11	0.35	0.22	21
Iowa	2 (1)	7.2	0.45	143	<0.05	<0.05	<0.05	<0.05	<0.13	<0.13
(Hamilton)	3 (2)	8.1	0.45	130	<0.05	0.14	0.11	0.11	0.22	0.31
	4 (3)	8.9	0.47	7	<0.05	8.0	<0.05	0.17	<0.13	8.3
Iowa	2 (1)	7.2	0.45	132	<0.05	<0.05	<0.05	<0.05	<0.13	<0.13
(Des Moines)	3 (2)	8.1	0.45	99	<0.05	0.12	0.13	0.29	0.25	0.56
	4 (3)	8.9	0.45	7	<0.05	12	0.21	0.50	0.37	13
Illinois	2 (1)	7.2	0.45	124	<0.05	<0.05	<0.05	<0.05	<0.13	<0.13
(Henry)	3 (2)	8.1	0.45	104	<0.05	<0.05	0.13	<0.05	0.25	<0.13
	4 (3)	8.9	0.60	7	<0.05	7.8	0.12	0.12	0.23	8.0
Illinois	2 (1)	7.2	0.62	134	<0.05	<0.05	<0.05	<0.05	<0.13	<0.13
(Clinton)	3 (2)	8.1	0.75	104	<0.05	0.38	0.45	0.34	0.73	0.90
	4 (3)	8.9	0.59	6	<0.05	3.0	0.30	0.41	0.51	3.6
Illinois	2 (1)	7.2	0.51	153	<0.05	<0.05	<0.05	<0.05	<0.13	<0.13
(Warren)	3 (2)	8.1	0.75	121	<0.05	0.10	0.41	0.27	0.67	0.51
	4 (3)	8.9	0.83	7	<0.05	12	0.41	0.47	0.67	13
Indiana	2 (1)	7.2	0.58	111	<0.05	<0.05	0.14	<0.05	0.26	<0.13
	3 (2)	8.1	0.86	97	<0.05	0.41	0.88	0.19	1.4	0.70
	4 (3)	8.9	0.60	6	<0.05	7.1	0.64	1.3	1.0	9.1
Kansas	2 (1)	7.2	0.48	119	<0.05	<0.05	0.33	<0.05	0.55	<0.13
	3 (2)	8.1	0.46	91	<0.05	0.13	0.48	0.13	0.78	0.33
	4 (3)	8.9	0.45	8	<0.05	2.8	0.48	0.21	0.78	3.1
Kentucky	2 (1)	7.2	0.52	109	<0.05	<0.05	0.20	0.13	0.35	0.25
	3 (2)	8.1	0.51	79	<0.05	1.3	1.3	2.74	2.0	5.4
	4 (3)	8.9	0.53	6	0.06	41	1.4	4.7	2.2	48
Michigan	2 (1)	7.2	0.53	135	<0.05	<0.05	<0.05	<0.05	<0.13	<0.13

Location	Application[1]			PHI, days	Residues, mg/kg					
	No. (type)	kg ai/ha[2]	kg ai/hl[3]		glyphosate		AMPA		Total[4]	
					Grain	Fodder	Grain	Fodder	Grain	Fodder
	3 (2)	8.1	0.52	114	<0.05	0.48	0.47	0.13	0.76	0.68
	4 (3)	8.9	0.52	6	<0.05	12	0.36	0.42	0.60	13
Minnesota	2 (1)	7.2	0.45	143	<0.05	<0.05	<0.05	<0.05	<0.13	<0.13
(Polk)	3 (2)	8.1	0.45	112	<0.05	0.30	0.28	0.31	0.48	0.77
	4 (3)	8.9	0.45	7	<0.05	2.0	0.30	0.27	0.51	2.4
Minnesota	2 (1)	7.2	0.52	142	<0.05	<0.05	<0.05	<0.05	<0.13	<0.13
(Rock)	3 (2)	8.1	0.55	118	<0.05	0.14	0.13	0.11	0.25	0.31
	4 (3)	8.9	0.57	6	0.06	34	0.21	0.28	0.38	34
Missouri	2 (1)	7.2	0.65	110	<0.05	<0.05	0.12	<0.05	0.23	<0.13
	3 (2)	8.1	0.50	90	<0.05	0.54	1.33	0.69	2.1	1.6
	4 (3)	8.9	0.47	6	<0.05	14	1.00	0.48	1.6	15
North Carolina	2 (1)	7.2	0.45	95	<0.05	<0.05	<0.05	0.30	<0.13	0.51
	3 (2)	8.1	0.45	76	<0.05	0.06	<0.05	0.39	<0.13	0.65
	4 (3)	8.9	0.45	7	<0.05	6.0	<0.05	0.91	<0.13	7.4
Nebraska	2 (1)	7.2	0.45	128	<0.05	<0.05	<0.05	<0.05	<0.13	<0.13
(York)	3 (2)	8.1	0.45	96	<0.05	0.05	0.24	0.08	0.41	0.17
	4 (3)	8.9	0.45	7	<0.05	1.8	0.32	0.13	0.54	2.0
Nebraska	2 (1)	7.2	0.45	129	<0.05	<0.05	<0.05	<0.05	<0.13	<0.13
(Polk)	3 (2)	8.1	0.45	96	<0.05	0.16	0.20	0.15	0.35	0.39
	4 (3)	8.9	0.45	8	<0.05	1.9	0.24	0.21	0.41	2.2
Ohio	2 (1)	7.2	0.48	145	<0.05	<0.05	<0.05	<0.05	<0.13	<0.13
	3 (2)	8.1	0.50	107	<0.05	0.10	0.35	0.31	0.58	0.57
	4 (3)	8.9	0.49	6	<0.05	8.4	0.22	0.44	0.38	9.1
Pennsylvania	2 (1)	7.2	0.45	114	<0.05	<0.05	<0.05	0.05	<0.13	0.13
	3 (2)	8.1	0.42	99	<0.05	0.38	0.28	0.43	0.48	1.0
	4 (3)	8.9	0.55	6	<0.05	6.2	0.31	0.62	0.52	7.1
South Dakota	2 (1)	7.2	0.51	142	0.08	<0.05	<0.05	<0.05	0.16	<0.13
	3 (2)	8.1	0.59	112	<0.05	18	0.33	0.40	0.55	19
	4 (3)	8.9	0.58	6	<0.05	20	0.32	0.45	0.54	21
Texas	2 (1)	7.2	0.59	118	<0.05	<0.05	<0.05	<0.05	<0.13	<0.13
	3 (2)	8.2	0.61	83	<0.05	0.14	0.16	<0.05	0.29	0.22
	4 (3)	9.1	0.60	7	<0.05	18	0.11	<0.05	0.22	18
Wisconsin	2 (1)	7.2	0.56	118	<0.05	<0.05	<0.05	<0.05	<0.13	<0.13
	3 (2)	8.1	0.56	103	<0.05	0.18	0.10	0.37	0.20	0.74
	4 (3)	8.9	0.46	6	<0.05	6.7	0.14	0.20	0.26	7.0

[1]Expressed as glyphosate acid, *N*-(phosphonomethyl)glycine
[2]Total of all applications
[3]Concentration of last application
[4]Glyphosate + AMPA expressed as glyphosate

Table 13. Residues of glyphosate and AMPA in resistant maize forage from supervised trials in the USA in 1994 (Oppenhuizen, 1995d). SL 41% formulation.

Location	Application			PHI, days	Residues, mg/kg		
	No. (type)	kg ai/ha[2]	kg ai/hl[3]		glyphosate	AMPA	Total[4]
Colorado	2 (1)	7.2	0.50	80	<0.05	<0.05	<0.13
	3 (2)	8.1	0.53	55	0.11, 0.05	0.16, 0.12	0.35, 0.23
Iowa	2 (1)	7.3	0.45	77	<0.05	0.07	0.16
(Butler)	3 (2)	8.1	0.45	62	<0.05, 0.05	0.14, 0.12	0.26, 0.23
Iowa	2 (1)	7.2	0.45	83	<0.05	<0.05	<0.13
(Hamilton)	3 (2)	8.1	0.45	70	0.08, 0.08	0.21, 0.22	0.40, 0.41
Iowa	2 (1)	7.2	0.45	96	<0.05	<0.05	<0.13
(Des Moines)	3 (2)	8.1	0.45	63	0.11, 0.16	0.46, 0.32	0.81, 0.65
Illinois	2 (1)	7.2	0.45	84	<0.05	<0.05	<0.13
(Henry)	3 (2)	8.1	0.45	64	<0.05,<0.05	0.12, 0.12	0.23, 0.23
Illinois	2 (1)	7.2	0.62	84	<0.05	<0.05	<0.13
(Clinton)	3 (2)	8.1	0.75	54	0.27, 0.19	0.43, 0.45	0.92, 0.87
Illinois	2 (1)	7.2	0.51	97	<0.05	<0.05	<0.13
(Warren)	3 (2)	8.1	0.75	65	0.31, 0.08	0.36, 0.37	0.86, 0.64
Indiana	2 (1)	7.2	0.58	75	<0.05	0.12	0.23
	3 (2)	8.1	0.86	61	0.19, 0.52	0.83, 0.93	1.5, 1.9
Kansas	2 (1)	7.2	0.48	76	<0.05	<0.05	<0.13
	3 (2)	8.1	0.46	48	0.21, 0.21	0.52, 0.44	1.0, 0.88
Kentucky	2 (1)	7.2	0.52	55	<0.05	0.19	0.34
	3 (2)	8.1	0.51	25	0.59, 0.73	2.3, 2.5	4.1, 4.5
Michigan	2 (1)	7.2	0.53	93	<0.05	<0.05	<0.13
	3 (2)	8.1	0.52	72	0.24, 0.23	0.35, 0.56	0.77, 1.1
Minnesota	2 (1)	7.2	0.45	98	<0.05	0.05	0.13
(Polk)	3 (2)	8.1	0.45	67	0.12, 0.14	0.37, 0.44	0.68, 0.81
Minnesota	2 (1)	7.2	0.52	93	<0.05	<0.05	<0.13
(Rock)	3 (2)	8.1	0.55	69	0.10, 0.10	0.20, 0.18	0.40, 0.37
Missouri	2 (1)	7.2	0.65	69	<0.05	0.09	0.19
	3 (2)	8.1	0.50	49	0.10, 0.11	0.90, 1.1	1.5, 1.8
North Carolina	2 (1)	7.2	0.45	67	<0.05	0.11	0.22
	3 (2)	8.1	0.45	48	0.05, 0.05	0.33, 0.32	0.55, 0.54
Nebraska	2 (1)	7.2	0.45	93	<0.05	<0.05	<0.13
(York)	3 (2)	8.1	0.45	61	0.12, 0.13	0.32, 0.26	0.61, 0.53
Nebraska	2 (1)	7.2	0.45	96	<0.05	<0.05	<0.13
(Polk)	3 (2)	8.1	0.45	63	0.08, 0.06	0.37, 0.24	0.64, 0.42
Ohio	2 (1)	7.2	0.48	88	<0.05	<0.05	<0.13
	3 (2)	8.1	0.50	50	0.13, 0.24	0.41, 0.55	0.75, 1.1
Pennsylvania	2 (1)	7.2	0.45	86	<0.05	0.07	0.16
	3 (2)	8.1	0.42	71	0.18, 0.18	0.70, 0.57	1.2, 1.1
South Dakota	2 (1)	7.2	0.51	93	<0.05	0.05	0.13

Location	Application			PHI, days	Residues, mg/kg		
	No. (type)	kg ai/ha[2]	kg ai/hl[3]		glyphosate	AMPA	Total[4]
	3 (2)	8.1	0.59	63	0.20, 0.18	0.47, 0.54	0.91, 1.0
Texas	2 (1)	7.2	0.59	84	<0.05	<0.05	<0.13
	3 (2)	8.2	0.61	49	0.09, 0.05	0.06, 0.05	0.18, 0.13
Wisconsin	2 (1)	7.2	0.56	86	0.10	0.11	0.27
	3 (2)	8.1	0.56	71	0.18, 0.14	0.41, 0.26	0.80, 0.54

[1]Expressed as glyphosate acid, *N*-(phosphonomethyl)glycine
[2]Total of all applications
[3]Concentration of last application
[4]Glyphosate + AMPA expressed as glyphosate

Eight supervised trials on susceptible sorghum in the USA were reported in 1994. The results are shown in Tables 14 and 15.

Table 14. Residues of glyphosate and AMPA in susceptible sorghum grain and fodder from supervised trials in the USA in 1992 (Oppenhuizen, 1993). SL 41% formulation.

Location	Application[1]			PHI, days	Residues, mg/kg					
	No.	kg ai/ha	kg ai/hl		Glyphosate		AMPA		Total[2]	
					Grain	Fodder	Grain	Fodder	Grain	Fodder
Arkansas	1	1.7	0.90	8	1.7	16	0.09	0.29	1.8	16
Kansas (Chautauqua)	1	1.7	0.94	6	5.3	33	0.09	0.33	5.4	34
Kansas (Pratt)	1	1.7	1.0	8	13	29	0.09	0.27	13	29
Missouri	1	1.7	0.90	7	6.0	28	0.11	0.22	6.2	28
Nebraska	1	1.7	1.8	8	1.8	7.0	<0.05	0.09	1.9	7.1
Oklahoma	1	1.7	1.2	7	6.3	29	0.22	0.41	6.6	30
South Dakota	1	1.7	0.95	7	13	2.9	0.08	<0.05	13	3.0
Texas	1	1.7	1.4	8	1.4	8.2	0.10	0.16	1.6	8.4

[1]Expressed as glyphosate acid, *N*-(phosphonomethyl)glycine
[2]Glyphosate + AMPA expressed as glyphosate

Table 15. Residues of glyphosate and AMPA in susceptible sorghum hay and fodder from supervised trials in the USA in 1992 (Oppenhuizen, 1993). SL 41% formulation, single application.

Location	Application[1]		PHI, days	Residues, mg/kg		
	kg ai/ha	kg ai/hl		glyphosate	AMPA	Total[2]
Arkansas	1.7	0.90	12	18	0.36	19
Kansas (Chautauqua)	1.7	0.94	11	15	0.22	15
Kansas (Pratt)	1.7	1.0	11	37	0.31	37
Missouri	1.7	0.90	15	15	0.15	15
Nebraska	1.7	1.8	12	4.3	0.08	4.4
Oklahoma	1.7	1.2	14	36	0.45	37
South Dakota	1.7	0.95	10	6.4	<0.05	6.5
Texas	1.7	1.4	11	3.1	<0.05	3.2

[1]Expressed as glyphosate acid, *N*-(phosphonomethyl)glycine
[2]GlyphoaRW+ AMPA expressed as glyphosate

FATE OF RESIDUES IN STORAGE AND PROCESSING

Storage

No information.

Processing

Cotton seed. One processing trial was reported to the Meeting. Cotton which was genetically modified to be susceptible to glyphosate was treated five times with glyphosate at a total application rate of 8.4 kg/ha. Six days after the final treatment, cotton seed was harvested and about 30 kg was processed in a batch operation that simulated industrial practice.

The raw cotton seed was saw-delinted to reduce the amount of lint remaining on the seed from 11-15% to about 3%. The hulls were then mechanically cracked and separated to produce the hull and kernel fractions. The kernels were flaked and extruded, and the extruded material was extracted three times with hot hexane to remove the oil. After removal of the solvent, the extracted material was further dried by passing warm air through it to produce solvent-extracted meal.

A portion of the mesicella (crude oil and hexane mixture) was taken and the hexane evaporated off to supply the crude oil fraction. The remaining mesicella was refined by adjusting the ratio of crude oil to hexane to 60:40, adding sodium hydroxide, and centrifuging to separate the soapstock from the refined oil and hexane. The hexane was removed by evaporation under vacuum and the refined oil bleached by heating with activated bleaching earth. The bleached oil was deodorized by heating under vacuum to yield bleached-deodorized refined oil.

The results are shown in Table 16.

Table 16. Residues of glyphosate and AMPA in processed fractions of susceptible cotton seed, USA (Texas) 1994 (Oppenhuizen, 1995a).

Application, kg ai/ha, and timing	Sample	Residues, mg/kg			Processing factor[2]
		glyphosate	AMPA	Total[1]	
3.36 pre-emergence 0.84 3-4 leaf 1.26 5-6 leaf 1-26 post-directed 1-68 pre-harvest	Raw cotton seed	3.7	<0.05	3.8	1.0
	Delinted seed	0.63	<0.05	0.71	0.19
	Kernels	0.24	<0.05	0.32	0.084
	Hulls	1.2	<0.05	1.3	0.34
	Meal	0.39	<0.05	0.47	0.12
	Crude oil	<0.05	<0.05	<0.13	<0.034
	Soapstock	<0.05	<0.05	<0.13	<0.034
	Refined oil	<0.05	<0.05	<0.13	<0.034
	Bleached-refined deodorized oil	<0.05	<0.05	<0.13	<0.034

[1]Glyphosate + AMPA expressed as glyphosate
[2]Based on total residue

Maize. Two processing trials in the USA were reported, in both of which glyphosate-resistant maize plants were treated with glyphosate pre-harvest at an application rate of 2.5 kg/ha. The maize was harvested six or seven days after treatment and 130-200 kg batches were processed by both dry and wet milling in operations that simulated industrial practice.

Dry milling. The whole maize grain was dried and cleaned by aspiration and screening. The light impurities from the aspiration were classified as chaff and grain dust and were subdivided by sieving. The cleaned whole grain was moisture-conditioned to 20-22%, allowed to temper for 2-2.5 hours, and impact-milled in a Ripple mill. After milling, the cornstock was dried at 54-71°C for 30 minutes, allowed to cool to approximately 32°C after removal from the oven, and passed over a 1/8 inch shaker screen. The material held by the screen was further processed into large grits, germ and hull (bran). That which passed through the screen was separated into medium and small grits, coarse meal, meal, and flour.

The germ was moisture-conditioned to 12%, heated to 88-104°C, flaked and pressed. The resulting fractions were expelled crude oil and presscake with residual crude oil. The presscake was extracted 3 times with hexane at 50-60°C and the mesicella (crude oil and hexane) passed through a separation unit to separate the crude oil and hexane. The crude oil was heated to 72-90°C to remove hexane. The extracted presscake was dried in a current of warm air, and the crude oil-refined according to American Oil Chemists Society Method Ca9a52.

Wet milling. The cleaned whole grain was steeped in water at 50-54°C containing 0.1-0.2% sulfur dioxide for 22-48 hours, then passed through a Bauer mill with devil-toothed plates and the ground product (cornstock) floated in salt water to remove the germ. The cornstock was again ground to separate the hull (bran) and further processed to produce gluten and starch.

The germ was processed as in dry milling to crude and refined oil. The results are shown in Table 17.

Table 17. Residues of glyphosate and AMPA in processed fractions of resistant maize grain, USA, 1993 (Oppenhuizen, 1995c).

Application, Location	Sample	Residues, mg/kg			Processing factor[2]
		Glyphosate	AMPA	Total[1]	
	Raw grain	<0.05	0.08	0.17	1.0
	Cleaned grain	0.05	0.10	0.20	1.2
	Dry-milled crude oil	<0.05	<0.05	<0.13	<0.76
2.5 kg/ha,	Dry-milled refined oil	<0.05	<0.05	<0.13	<0.76
6 days PHI	Flour	<0.05	0.07	0.16	0.94
Illinois	Grain dust	8.3	1.6	11	65
	Grain dust screenings	14	1.7	17	100
	Grits	<0.05	<0.05	<0.13	<0.76
	Meal	<0.05	0.07	0.16	0.94
	Starch	<0.05	<0.05	<0.13	<0.76
	Wet-milled crude oil	<0.05	<0.05	<0.13	<0.76
	Wet-milled refined oil	<0.05	<0.05	<0.13	<0.76
	Raw grain	<0.05	0.12	0.23	1.00
	Clean grain	<0.05	0.09	0.19	0.83
	Dry-milled crude oil	<0.05	<0.05	<0.13	<0.57
2.5 kg/ha,	Dry-milled refined oil	<0.05	<0.05	<0.13	<0.57
7 days PHI	Flour	<0.05	<0.05	<0.13	<0.57
Iowa	Grain dust	0.64	<0.05	0.72	3.1
	Grain dust screenings	0.40	<0.05	0.48	2.1
	Grits	<0.05	0.05	0.13	0.57
	Meal	0.05	<0.05	0.13	0.57
	Starch	<0.05	<0.05	<0.13	<0.57
	Wet-milled crude oil	<0.05	<0.05	<0.13	<0.57

		Residues, mg/kg			
	Wet-milled refined oil	<0.05	<0.05	<0.13	<0.57

[1]Glyphosate + AMPA expressed as glyphosate
[2]Based on total residue

Sorghum. Two processing trials in the USA were reported. In both trials, sorghum was treated with glyphosate by pre-harvest application at a rate of 1.7 kg/ha. It was harvested six or eight days after and processed by both dry and wet milling in 70 kg batches according to industrial practice.

The grain was dried and cleaned by aspiration and screening. The light impurities from the aspiration were classified as grain dust. The cleaned grain was then processed by dry and wet milling.

In the dry process the grain was abrasively milled to remove most of the bran and grits, and the seed was ground into flour.

In wet milling, the cleaned grain was steeped in water and then milled to recover germ, hull, coarse gluten-starch, gluten and starch.

The results are shown in Table 18.

Table 18. Residues of glyphosate and AMPA in processed fractions of susceptible sorghum grain, USA, 1992 (Oppenhuizen, 1994).

Application, location	Sample	Residues, mg/kg			Processing factor[2]
		glyphosate	AMPA	Total[1]	
1.7 kg/ha 6 days PHI Kansas	Raw sorghum	4.5	0.05	4.6	1.0
	Bran	18	0.22	18	3.9
	Clean grain	5.9	0.10	6.1	1.3
	Flour	1.5	<0.05	1.6	0.35
	Germ	<0.05	<0.05	<0.13	<0.028
	Grain dust	28	0.29	28	6.1
	Grits, medium	2.7	<0.05	2.8	0.61
	Starch	<0.05	<0.05	<0.13	<0.028
	Steepwater	3.3	0.05	3.4	0.74
1.7 kg/ha 8 days PHI Texas	Raw sorghum	1.1	<0.05	1.2	1.0
	Bran	6.4	0.12	6.6	5.5
	Clean grain	1.1	<0.05	1.2	1.0
	Flour	0.35	<0.05	0.43	0.36
	Germ	<0.05	<0.05	<0.13	<0.11
	Grain dust	3.8	0.07	3.9	3.3
	Grits, medium	0.35	<0.05	0.43	0.36
	Starch	<0.05	<0.05	<0.13	<0.11
	Steepwater	0.39	<0.05	0.47	0.39

[1]Glyphosate + AMPA expressed as glyphosate
[2]Based on total residue

NATIONAL MAXIMUM RESIDUE LIMITS

The only national MRLs reported were those in the USA which were amended recently. The residue is defined as glyphosate.

Commodity	US MRL, mg/kg
Cotton seed	15
Maize grain	1
Maize forage	1
Maize fodder	100
Sorghum grain	15
Sorghum fodder	40

APPRAISAL

Glyphosate was first evaluated in 1986, and residue aspects were reviewed in 1987, 1988 and 1994. Maximum residue levels were estimated for kiwifruit and a range of vegetables, cereals, oilseeds and animal products.

The 1997 JMPR was requested to evaluate the new uses of glyphosate on cotton, maize and sorghum according to GAP. These new uses are (1) pre-harvest topical applications and (2) in-crop applications to cotton and maize crops which have been genetically modified to be resistant to glyphosate. Relevant data on metabolism and residue trials were submitted to the Meeting.

Genetic modification of crops

Glyphosate binds to and blocks the activity of 5-enolpyruvoyl-shikimate-3-phosphate synthase (EPSPS), an enzyme of the aromatic amino acid biosynthetic pathway. Glyphosate inhibition of EPSPS prevents the plant from synthesizing the aromatic amino acids essential for protein production. Glyphosate-resistant EPSPS is derived from *Agrobacterium sp.* strain CP4 (CP4 EPSPS), and has been used to develop glyphosate-resistant (i.e. glyphosate-resistant) crops.

While CP4 EPSPS has been successful in providing glyphosate resistance in cotton, its activity alone has been insufficient to ensure adequate resistance in other crops. In maize, a second mechanism has been developed to ensure sufficient levels of crop resistance to allow applications of glyphosate at rates necessary for effective weed control. The second mechanism is glyphosate inactivation, which effectively reduces cellular levels of glyphosate by converting it to aminomethylphosphonic acid (AMPA). The enzyme responsible for glyphosate inactivation is glyphosate oxidoreductase (*gox*). The gene encoding *gox* was isolated from a naturally-occurring bacterium, *Achromobacter sp.*, and has been modified to optimize its expression in plants.

Plant metabolism

Numerous plant metabolism studies with vegetable, orchard tree, nut tree and pasture crops were reported to the 1986 JMPR. The 1986 Meeting concluded that glyphosate applied to the soil was absorbed very slightly or not at all by the crops examined and its conversion to AMPA, the primary metabolite, was not observed.

However, hydroponic administration allows sufficient uptake of glyphosate to elucidate its metabolism in plants. Metabolic studies with glyphosate in hydroponically-grown maize, wheat, cotton and soya beans have shown the conversion of glyphosate to AMPA and further degradation in plant tissues.

Metabolic studies in plants that have been genetically modified to be resistant to glyphosate show that the metabolism is the same as in susceptible plants. Glyphosate is metabolized to AMPA, which is either non-selectively bound to natural plant constituents, further degraded to one-carbon fragments that are incorporated into natural products, or conjugated with naturally-occurring organic acids to give trace-level metabolites. The metabolites are the same in resistant and susceptible crops but their relative distribution depends on the speed and extent of conversion to AMPA.

Methods of residue analysis

Glyphosate and its major metabolite AMPA can be determined by GLC or HPLC after derivatization. In the GLC method evaluated by the 1986 JMPR, clean-up on anion exchange, cation exchange and carbon columns is followed by trifluoroacetylation and methylation. The limit of determination was 0.05 mg/kg in cotton seed and hay and recoveries of glyphosate and AMPA respectively at 0.05-0.4 mg/kg fortification levels were 66.3-89.4% and 66.0-84.9% in cotton hay, and 56.7-74.8% and 63.4-93.2% in cotton seed.

HPLC methods were discussed in the 1986 and 1994 monographs. The preferred method employs two-column switched HPLC with a post-column reactor. The limit of determination was 0.05 mg/kg in all commodities and mean recoveries were 77-88% for glyphosate and 78-90% for AMPA.

Residues of AMPA in or on crops and definition of the residue

The Meeting received data on supervised trials on maize into which the *gox* gene had been introduced, which showed that residue levels of AMPA were much higher than those in normal crops.

The Meeting agreed to recommend two MRLs for residues in maize, one as glyphosate to accommodate uses on glyphosate-susceptible crops and the other as AMPA to accommodate uses on glyphosate-resistant crops. A violation would occur if either MRL were exceeded.

The current definition of the residue is "glyphosate" because residues of AMPA in crops are usually very low or undetectable, except in soya beans.

The Meeting agreed that the definition of the residue for estimations of dietary intake should include AMPA but the definition for enforcement purposes for all commodities, including genetically modified crops, should remain as "glyphosate" for the following reasons.

1. Already many commodities have CXLs based on the residue defined as glyphosate. All existing CXLs would have to be reviewed if the definition of the residue were changed.
2. It is not thought appropriate to establish a separate definition of the residue for maize.
3. The existing definition of the residue has already been incorporated into many national regulations, and a change of the definition would be likely to cause difficulties in international harmonization.

The Meeting also noted the significant residue levels of AMPA that occurred in soya beans, and recommended that their significance should be evaluated in a future periodic review even though they are not believed to pose any risk to consumers.

Supervised trials

In the following text the sum of glyphosate + AMPA expressed as glyphosate is referred to as "total glyphosate". The total glyphosate residue was evaluated to estimate STMRs for the assessment of dietary intake.

Cotton. Twelve supervised trials were carried out on glyphosate-susceptible cotton in the USA with pre-harvest application at 3.4 kg ai/ha. US GAP allows pre-emergence (crop) application (including pre-plant or at-planting applications), post-directed application (post-crop-emergence, directed at weeds), spot treatment and pre-harvest application at 4.2 kg ai/ha as the maximum for each treatment. The total application is restricted to 6.7 kg ai/ha per year.

Six of the trials were with pre-emergence and post-emergence applications before a pre-harvest application. The pre-emergence application rate (6.7 kg ai/ha) and the total applied (10-26 kg ai/ha) exceeded the GAP limits, but the Meeting concluded that these trials were comparable with GAP because the rate of the pre-harvest application (3.4 kg ai/ha), which should be most influential on the residue in the harvested crops, was within the GAP rate of 4.2 kg ai/ha and the studies of plant metabolism indicated that the uptake of glyphosate from soil would be negligible. The other six trials with only one pre-harvest application at 3.4 kg ai/ha were according to GAP.

Sixteen supervised trials, with three different application patterns in each, were carried out on glyphosate-resistant cotton in the USA with 4 or 5 applications which included pre-emergent, post-emergent, post-directed and pre-harvest treatments. Eleven trials were with genotype 1445 cotton and five with genotype 1698 cotton but these have the same basic genetic structure and would be expected to show no differences in glyphosate metabolism.

All the application patterns slightly exceeded US GAP: post-emergence (trials: 0.84-1.26 kg ai/ha, GAP: 0.84 kg ai/ha), post-directed (trials: 1.26 kg ai/ha, GAP: 0.84 kg ai/ha), and total application (trials 7.56-8.8 kg ai/ha, GAP: 6.7 kg ai/ha), but the Meeting again concluded that the trials complied with GAP because the most influential final applications were compatible with GAP and earlier applications would be unlikely to have much effect on the residues.

In susceptible cotton seed the residues of glyphosate were 0.54-5.9 mg/kg at 5-9 days and 0.15-3.6 mg/kg at 10-14 days, and those of AMPA were <0.05-0.20 mg/kg at 5-14 days. The residues of total glyphosate were 0.62-6.0 mg/kg at 5-9 days and 0.23-3.7 mg/kg at 10-14 days, and of total glyphosate after maximum GAP treatments 0.62, 0.71, 2.4, 2.8, 3.0 and 6.0 mg/kg.

In resistant cotton seed the residues of glyphosate were 0.13-5.0 mg/kg at 6-9 days and 0.30-0.50 mg/kg at 17 days, and those of AMPA were <0.05-0.21 mg/kg at 7-9 days. The residues of total glyphosate were 0.21-5.2 mg/kg at 6-9 days and 0.38-0.58 mg/kg at 17 days. Those of total glyphosate after maximum GAP treatments were 0.21, 0.30, 0.42, 0.49, 0.51 (2), 0.52, 0.54, 0.55, 0.66, 0.68, 0.73, 0.75, 0.77 (2), 1.1 (2), 1.3, 1.4, 1.5, 1.8, 1.9, 2.1 (2), 2.2, 2.3, 2.5, 2.6 (3), 2.8, 2.9 (2), 3.2, 3.5, 3.7, 3.8, 4.2 (2), 4.4, 4.7 and 5.2 mg/kg.

Since the differences between both the median and maximum total glyphosate residues in resistant and susceptible crops were not significant, the Meeting based the STMR on the combined residues from the two sets of trials.

The total glyphosate residues from the 48 individual trials which complied with GAP (six on susceptible cotton and 42 on resistant cotton) in rank order (median underlined) were 0.21, 0.30, 0.42, 0.49, 0.51 (2), 0.52, 0.54, 0.55, 0.62, 0.66, 0.68, 0.71, 0.73, 0.75, 0.77 (2), 1.1 (2), 1.3, 1.4, 1.5, 1.8, <u>1.9</u>, <u>2.1</u> (2), 2.2, 2.3, 2.4, 2.5, 2.6 (3), 2.8 (2), 2.9 (2), 3.0, 3.2, 3.5, 3.7, 3.8, 4.2 (2), 4.4, 4.7, 5.2 and 6.0 mg/kg.

The Meeting estimated an STMR level of 2.0 mg/kg total glyphosate. Taking into account the residues of glyphosate alone in susceptible (0.54-5.9 mg/kg) and resistant (0.13-5.0 mg/kg) crops, the Meeting estimated a maximum residue level of 10 mg/kg glyphosate and recommended the withdrawal of the CXL of 0.5 mg/kg.

The residues of glyphosate in the hay from susceptible cotton were 3.8-33 mg/kg at 5-9 days and 6.3-84 mg/kg at 10-14 days, and those of AMPA were 0.10-0.46 mg/kg at 5-14 days. The residues of total glyphosate were 4.1-33 mg/kg at 5-9 days and 6.4-85 mg/kg at 10-14 days.

The glyphosate residues (3.8-84 mg/kg) were below the existing CXL for the straw and fodder (dry) of cereal grains (100 mg/kg), although cotton hay is not classified within this group of commodities. The Meeting agreed not to recommend an MRL for cotton hay in view of its insignificance in international trade.

The residues of glyphosate in the gin by-product from resistant cotton were 3.7-84 mg/kg at 6-9 days and 0.79-2.2 mg/kg at 17 days, and those of AMPA were <0.05-0.84 mg/kg at 6-9 days and <0.05 mg/kg at 17 days. The residues of total glyphosate were 3.8-85 mg/kg at 6-9 days and 0.87-2.3 mg/kg at 17 days.

The Meeting did not recommend an MRL because the commodity does not figure in international trade.

<u>Maize</u>. Twelve supervised trials on susceptible maize and 66 on resistant maize were carried out in the USA.

The 12 trials were with one pre-harvest application (2.5 kg ai/ha). US GAP allows pre-emergence application (0.32-4.2 kg ai/ha), spot treatment (0.32-4.2 kg ai/ha) and pre-harvest application (2.5 kg ai/ha for ground, 0.84 kg ai/ha for aerial) but the Meeting considered that the trials were effectively compatible with the maximum GAP application because the residue from pre-emergence application would be expected to be negligible and spot treatment should not affect crops if carried out according to GAP.

The 66 trials on resistant maize were with 2 to 4 applications which included pre-emergent, post-emergent and pre-harvest applications; 22 of the trials were according to maximum GAP.

Grain. The residues of glyphosate, AMPA and total glyphosate in the susceptible maize were <0.05-0.54 mg/kg, <0.05-0.13 mg/kg and <0.13-0.62 mg/kg respectively at 6-7 days. The residues of total glyphosate after maximum GAP treatments were <0.13 (5), 0.13, 0.14, 0.19, 0.23, 0.25, 0.27 and 0.62 mg/kg.

The residues of glyphosate, AMPA and total glyphosate in the resistant maize were <0.05-0.34 mg/kg, <0.05-1.4 mg/kg and <0.13-2.2 mg/kg respectively at 6-8 days. The residues of total glyphosate after maximum GAP treatments were <0.13 (2), 0.22 (2), 0.23, 0.26, 0.37, 0.38 (2), 0.41, 0.42, 0.51 (2), 0.52, 0.54 (2), 0.60, 0.67, 0.78, 1.0, 1.6 and 2.2 mg/kg.

Since the total glyphosate residues in the susceptible and resistant maize clearly belonged to difference populations, the Meeting estimated an STMR of 0.47 mg/kg total glyphosate, based on the residues in the resistant maize.

On the basis of the residues of glyphosate in susceptible (<0.05-0.54 mg/kg) and resistant (<0.05-0.34 mg/kg) maize, the Meeting recommended an MRL of 1 mg/kg for glyphosate to replace the existing CXL (0.1* mg/kg). The Meeting also estimated a maximum residue level of 2 mg/kg for AMPA in maize on the basis of the residues of AMPA found in resistant maize (<0.05-1.4 mg/kg).

Fodder. The residues of glyphosate, AMPA and total glyphosate in the susceptible maize fodder were 3.7-92 mg/kg, 0.09-0.81 mg/kg and 3.8-93 mg/kg respectively at 6-7 days. The corresponding residues in the fodder from resistant maize were 1.8-41 mg/kg, <0.05-4.7 mg/kg and 2.0-48 mg/kg respectively at 6-8 days. The residues in both susceptible and resistant maize fodder were below the existing CXL for the straw and fodder (dry) of cereal grains (100 mg/kg).

The Meeting estimated a maximum residue level of 5 mg/kg for AMPA in maize fodder from the residues in fodder from resistant maize (<0.05-4.7 mg/kg).

Forage. According to GAP, the forage of susceptible crops should be cut before the pre-harvest application of glyphosate, whereas the forage of resistant crops can be cut after the application before harvest. Trials to determine residues in forage were therefore restricted to resistant maize.

The residues of glyphosate, AMPA and total glyphosate in the maize forage were <0.05-0.52 mg/kg, 0.06-1.1 mg/kg and 0.18-1.9 mg/kg respectively after 48-65 days. Those of total glyphosate from maximum GAP treatments were 0.18, 0.23, 0.26, 0.35, 0.55, 0.61, 0.64, 0.81, 0.86, 0.92, 1.0 (2), 1.1, 1.8 and 1.9 mg/kg.

The Meeting estimated maximum residue levels of 1 mg/kg glyphosate and 2 mg/kg AMPA, which are recommended for use as MRLs, and an STMR of 0.81 mg/kg total glyphosate.

Sorghum (pre-harvest applications to susceptible plants). Eight supervised trials were carried out in the USA with one pre-harvest application at 1.7 kg ai/ha. US GAP allows pre-emergence application at 0.32-4.2 kg ai/ha, spot treatment at 0.32-4.2 kg ai/ha and pre-harvest application at 1.7 kg ai/ha. For the reasons given above, the Meeting considered the trials to be compatible with maximum GAP.

Grain. The residues of glyphosate, AMPA and total glyphosate were 1.4-13, <0.05-0.22 and 1.6-13 mg/kg respectively after 6-8 days. Those of total glyphosate in rank order were 1.6, 1.8, 1.9, 5.4, 6.2, 6.6 and 13(2) mg/kg.

The Meeting recommended an MRL of 20 mg/kg for glyphosate to replace the existing CXL (0.1* mg/kg), and an STMR of 5.8 mg/kg for total glyphosate.

Fodder and hay. Residue data said to be on sorghum hay were submitted, but the Meeting concluded that the commodity analysed in the trial should be classified as sorghum fodder.

The residues of glyphosate, AMPA and total glyphosate in fodder were 2.9-33, <0.05-0.41 and 3.0-34 mg/kg respectively at 6-8 days. The corresponding residues in "hay" were 3.1-37, <0.05-0.45 and 3.2-37 mg/kg at 10-15 days.

The glyphosate residues in both fodder (2.9-33 mg/kg) and hay (3.1-37 mg/kg) were below the existing CXL for the straw and fodder (dry) of cereal grains (100 mg/kg).

Processing

Cotton. Although only one study was available the Meeting agreed to calculate STMR-Ps because the processing adequately simulated industrial practice.

Processing factors from cotton seed to delinted cotton seed, cotton kernels, cotton hulls and cotton meal were 0.19, 0.084, 0.34 and 0.12 respectively. They were <0.034 for processing to crude cotton seed oil, cotton soapstock, refined cotton seed oil and bleached-deodorized cotton seed oil.

The Meeting estimated maximum residue levels of 0.05* mg/kg for crude and edible cotton seed oil, and STMR-Ps of 0.38, 0.17, 0.68 and 0.24 mg/kg for delinted cotton seed, cotton kernels, cotton hulls and cotton meal respectively, by calculation from the cotton seed STMR of 2.0 mg/kg.

Maize. Residues of glyphosate and AMPA were determined in the processed commodities but the residue of glyphosate in the raw grain was below the LOD, although AMPA was detected. Information on the conversion of glyphosate to AMPA during the processing was not available. The Meeting could not use the data to estimate STMR-Ps.

Sorghum. The mean processing factors were 4.7, 1.2, 0.36, 4.7 and 0.49 from sorghum to bran, clean grain, flour, grain dust and grits (medium) respectively and <0.028 or <0.11 for processing to germ and starch.

The Meeting estimated STMR-Ps of 0 for sorghum germ and starch because they contained negligible residues of glyphosate and AMPA individually, and 27, 7.0, 2.1, 27 and 2.8 mg/kg for bran, clean grain, flour, grain dust and grits (medium) respectively, by calculation from the sorghum STMR (5.8 mg/kg).

RECOMMENDATIONS

Glyphosate

On the basis of the data on residues from supervised trials, the meeting concluded that the residues listed below are suitable for establishing MRLs and STMR to replace previous recommendation for glyphosate.

Definition of the residue for compliance with MRL: glyphosate

Definition of the residue for estimation of dietary intake: sum of glyphosate and aminomethylphosphonic acid (AMPA) expressed as glyphosate.

Commodity		Recommended MRL, mg/kg		PHI, days	Estimated STMR, mg/kg	Estimated STMR-P, mg/kg
CCN	Name	New	Previous			
SO 0691	Cotton seed	10	0.5	7	2.0	
OC 0691	Cotton seed oil, crude	0.05*				0.0
OR 0691	Cotton seed oil, edible	0.05*				0.0
	Delinted cotton seed					0.38
	Cotton seed kernels					0.17
	Cotton seed hulls					0.68
	Cotton seed meal					0.24
GC 0645	Maize	1	0.1*	7	0.47	
AF 0645	Maize forage	1		50	0.81	
GC 0651	Sorghum	20	0.1*	7	5.8	
	Sorghum, cleaned					7.0
	Sorghum bran					27
	Sorghum flour					2.1
	Sorghum grain dust					27
	Sorghum grits (medium)					2.8
	Sorghum germ					0.0
	Sorghum starch					0.0

AMPA

On the basis of the data on residues from supervised trials, the Meeting concluded that the maximum residue levels listed below are suitable for establishing MRLs.

Definition of the residue for compliance with MRLs: aminomethylphosphonic acid (AMPA)

Commodity		Maximum residue level, mg/kg		PHI, days
CCN	Name	New	Previous	
GC 0645	Maize	2	-	7
AS 0645	Maize fodder	5	-	7
AF 0645	Maize forage	2	-	50

FURTHER WORK OR INFORMATION

<u>Desirable</u>

Processing studies with both susceptible and resistant maize in which the raw grain contains measurable residues of both glyphosate and AMPA.

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GUAZATINE (114)

EXPLANATION

Guazatine, originally evaluated by the JMPR in 1978 and 1980, is included in the CCPR periodic review programme (ALINORM 91/24A, para 321 and Appendix VI, para 18). At the 1992 CCPR the manufacturer provided a list of all available data and indicated that toxicological studies were under way. Guazatine was tentatively scheduled for the 1996 JMPR pending the availability of these studies (ALINORM 93/24, Appendix V, Annex I). The 1995 CCPR postponed the residue review to 1997 (ALINORM 95/24A, Appendix IV). Information on current GAP was requested by circular letters (CL 1991/15-PR and CL 1993/11-PR).

The manufacturer provided data on physical and chemical properties, metabolism in plants and animals, environmental fate, methods of residue analysis, registered uses, residues in cereals, citrus fruits and sugar cane, and national MRLs (Buys *et al.*, 1997). Information on GAP and national MRLs was supplied by Australia (Anon., 1996a) and Germany (Anon., 1996b), and on GAP by Norway (Anon., 1997a) and the UK (Anon., 1997c). The Netherlands provided information on analytical methods, use patterns and national MRLs (Anon., 1997b).

IDENTITY

ISO common name: Guazatine

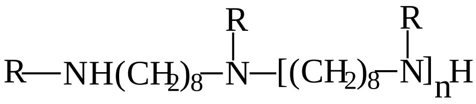
Chemical name: "A mixture of the reaction products from polyamines,
comprising mainly octamethylenediamine,
iminodi(octamethylene)diamine,
octamethylenebis(imino-octamethylene)diamine,
and carbamonitrile" (IUPAC)

The approved common name guazatine was originally defined as applying to 1,1'iminodi(octamethylene)diguanidine (BSI used the name *guanoctine* from 1970-1972). It is now known that the material marketed commercially is a reaction mixture. Produced by the amidination of technical iminodi(octamethylene)diamine, commercial guazatine containes numerous guanidines, in which the amino and imino groups of the polyamine chain form part, and polyamines; many of these bases are fungicidal. A replacement common name, iminoctadine (*q.v.*) has been established for 1,1'-iminodi(octamethylene)diguanidine. (Pesticide Manual, 1994).

CAS No: [115044-19-4] for guazatine acetates

Synonyms: GTA

Structural formula:



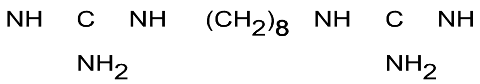
N may be 0,1,2 etc. and any R substituent may be
-H (17-23%) or
-$C(NH_2)=NH$ (77-83%).

A coding system is used for the compounds that make up guazatine in which "N" represents any amino group thus:

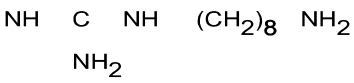
NN represents $H_2N-(CH_2)_8-NH_2$
NNN represents $H_2N-(CH_2)_8-NH-(CH_2)_8-NH_2$ and so on.

"G" represents any guanidated amino group (NH or NH_2) for example:

GG means



and GN means



The guanidated diamines and triamines are the most abundant components of guazatine. A typical composition of free guazatine (not of guazatine acetates, the salts which are used in practice) is as follows.

Component	%	Component	%
NN	0.8	GGG	30.6
GN	9.8	GNNG	1.4
GG	29.5	GGGN	1.4
NNN	$\leq$0.1	GGGG	5.1
NGN	0.8	Other tetramines[1]	3.1
GNN	1.7	GGGGG	1.1
GGN	8.1	Other pentamines[1]	1.4
GNG	4.5	Hexamines and above[1]	0.6
		Total:	99.9

[1]And their guandidated derivates

It can be seen that diamine derivatives account for 40% of the constituents, triamines for 46%, tetramines for 11% and other amine derivatives for 3%. The most abundant individual components are the fully guanidated triamine (GGG, 30.6%) and the fully guanidated diamine (GG, 29.5%) followed by the monoguanidated diamine (GN, 9.8%) and a diguanidated triamine (GGN, 8.1%).

Molecular formula: not applicable for a mixture
Molecular weight: not applicable for a mixture

Physical and chemical properties

Active ingredient as acetates

Vapour pressure: less than 10^{-5} Pa at °C (Boden, 1992a)
Melting point: guazatine acetate begins to melt at 60°C

Octanol/water partition coefficient (Karlsson and Stensiö, 1984, 1988):

pH	Component	log P_{ow}
3	guazatine acetates (GTA)	-1.2
7		ND
10		-0.9
6.5	1-amino-8-guanidino-octane diacetate (NG)	-2.3
6.5	1,8-diguanidino-octane diacetate (GG)	-3.3, -4.8
6.5	bis(8-guanidino-octyl)amine triacetate (GNG)	-3.2
6.5	bis(8-guanidino-octyl)guanidine triacetate (GGG)	-4.8

Solubility (Carlsson, 1992):

Solvent	Solubility at 20°C, g/l
water	> 3000
dimethylformamide	approx. 500
N-methylpyrrolidone	approx. 100
ethanol	200
methanol	510
2-propanol	28
N-octanol	15
acetone	<0.1
dichloromethane	<0.1
ethyl acetate	<0.1
toluene	<0.1
N-hexane	<0.1

r.d. (specific gravity): 1.09 g/cm^3 at 20°C (Boden, 1992b)

Hydrolysis: Guazatine acetates taken from two technical batches were hydrolysed in buffered aqueous solution at pH 5, 7 and 9 at 25°C for 30 days according to EPA Guidelines, and five compounds were determined by an HPLC method. There was no significant hydrolysis of any of the five components (Boden, 1992c).

Photolysis: Samples of GTA 70 in buffered aqueous solutions were irradiated in a Rayonet photochemical reactor at 300 nm for 24 h (Erikson and Stensiö, 1987) with the following results.

pH	Concentration, mg/l		
	Stored in darkness	Irradiated	% loss by photodegradation
5	26	26,3	0
7	8	6,5	19
9	18	13,5	25

Technical material

Purity: Technical guazatine obtained in the production process is a 70% w/w solution of guazatine acetates in water known as GTA 70. This solution is the basis of formulations.

Melting range: not applicable

Stability: GTA 70 is stable for at least 2 years at ambient temperature (can be stored at temperatures between 0°C and 50°C) (Carlsson,1993).

Formulations

LS: Solution for seed treatment
FS: Flowable concentrate for seed treatment
SL: Soluble concentrate
TC: Technical material
WP: Wettable powder
WS: Water-dispersible powder for slurry treatment

All active ingredient contents of the formulations are expressed as guazatine acetate. For example, a 200 SL formulation contains 200 g/l of guazatine acetate or 133 g/l of guazatine. The following products are used.

Post-harvest treatment of citrus fruit

SL 40 g/l guazatine acetate
SL 150 g/l guazatine acetate

SL 200 g/l guazatine acetate
SL 400 g/l guazatine acetate
TC 700 g/l guazatine acetate
Wax 3 g/l guazatine acetate

Seed treatment products

FS 150 g/l guazatine acetate, 12.5 g/l triticonazole
FS 266.7g/l guazatine acetate, 16.7 g/l triticonazole
FS 400 g/l guazatine acetate, 10 g/l flutriafol
LS 25 g/l guazatine acetate, 25 g/l imazalil
LS 150 g/l guazatine acetate, 10 g/l imazalil
LS 200 g/l guazatine acetate, 30 g/l imazalil
LS 200 g/l guazatine acetate, 200 g/l fenfuram, 20g/l imazalil
LS 200 g/l guazatine acetate, 12.5g/l triticonazole, 125g/l fipronil
LS 265 g/l guazatine acetate
LS 300 g/l guazatine acetate
LS 300 g/l guazatine acetate, 2.5 g/l cyproconazole
LS 300 g/l guazatine acetate, 5 g/l propiconazole
LS 300 g/l guazatine acetate, 15 g/l tebuconazole
LS 300 g/l guazatine acetate, 20 g/l imazalil
LS 300 g/l guazatine acetate, 25 g/l imazalil
LS 300 g/l guazatine acetate, 25 g/l triticonazole
LS 300 g/l guazatine acetate, 150 g/l fenfuram
LS 300 g/l guazatine acetate, 100 g/l fenfuram
LS 300 g/l guazatine acetate, 150 g/l fenfuram, 40 g/l imazalil
LS 350 g/l guazatine acetate
LS 400 g/l guazatine acetate
LS 700 g/l guazatine acetate
SL 200 g/l guazatine acetate
SL 400 g/l guazatine acetate
WP250 g/kg guazatine acetate

METABOLISM AND ENVIRONMENTAL FATE

Animal metabolism

Absorption, distribution and excretion

Rats. In a preliminary study (Leegwater, 1975) a male Wistar rat received a single oral dose of an aqueous solution (pH 5) containing guazatine labelled with tritium (19.5 µCi) in the octyl moieties and ^{14}C (5.84 µCi) in the guanidino groups. By the end of the 72-hour collection period the total recoveries of radioactivity were 83% of the ^{14}C and 93% of the ^{3}H. Most of the administered dose was recovered from the faeces (about 64% of the ^{14}C, 39% of the ^{3}H) and urine (about 15% ^{14}C, 42% ^{3}H). Most of the radiolabel in the urine was found during the first 24 hours whereas most of that in the faeces was found between 24 and 48 hours, but it was noted that the rat did not eat during the first 24 hours and this would be likely to reduce the intestinal transit time. Similar proportions of the ^{14}C dose were found in the liver (0.61%) and kidneys (0.67%) but the proportions of ^{3}H differed: liver 1.6%, kidney 0.17%. After 72 hours the gastrointestinal tract accounted for 0.47% of the ^{14}C and 1.3% of the ^{3}H, and the residual carcase for 1.9% of the ^{14}C and 9.3% of the ^{3}H.

In a later study (Leegwater, 1980) two male Wistar rats were dosed by oral gavage with [^{14}C]guazatine (pH 5.8) labelled in the guanidino groups at the nominal level of 10 mg/kg body weight. Samples were collected until 120 hours after dosing. The mean total recovery of radioactivity was 93% with about 60% in the urine and 30% in the faeces. Elimination in the urine was rapid, with 93% of the total recovered being eliminated in the first 24 hours. In the faeces about 52% of the total was found during the first 24 hours. After five days a mean total of about 2.5% of the administered dose remained in the body. The liver contained 0.6% of the administered radioactivity, the kidneys 0.08%, the blood 0.41%, the gastrointestinal tract 0.22% and the carcase 1.2%.

In a third study (Cameron *et al.*, 1989) four groups of five rats were dosed orally with [^{14}C]guazatine labelled in the octyl chains. The first group received a single dose of 20 mg/kg bw, two groups received single doses at 2 mg/kg bw (one for an ADME[1] investigation and the other for a bile elimination investigation), and the fourth group received daily doses of 2 mg/kg bw over a period of fourteen days. The mean recoveries of the total radioactive residue (TRR) were about 100% for all three single dose groups (during 96 hours), distributed as shown in Table 1.

[1] Absorption, distribution, metabolism and excretion

Table 1. Distribution of ^{14}C in rats (Cameron *et al.*, 1989).

Sample	^{14}C, % of dose					
	20 mg/kg		2 mg/kg ADME		2 mg/kg Bile[1]	
	Male	Female	Male	Female	Male	Female
Urine	6.9	7.5	6.3	6.06	7.16	4.87
Cage wash	0.55	0.63	0.75	0.75	0.26	3.35
Faeces	92.9	94.0	95.2	95.71	54.08	55.75
CO_2[1]	0.84	0.73	0.66	0.54	na	Na
Bile	Na	na	na	na	0.04	0.23
GI tract	Na	na	na	na	40.3	23.88
Tissue	0.91	1.1	0.74	0.89	na	Na
Carcase	0.4	0.42	0.12	0.15	3.49	11.12
Total	101.8	103.8	103.2	103.7	105.3	99.2

ADME: Absorption, distibution, metabolism, excretion
na: not analysed
[1]Collected for first 24 hours

The TRR in the tissues after 96 hours was low. The highest levels were found in the kidneys (low dose 0.06-0.2, high dose 0.7-3.03 mg guazatine equivalents/kg) and liver (low dose 0.07-0.38, high dose 1.14-5.1 mg/kg). The high recovery of ^{14}C from the faeces and the low levels in the urine, tissues and bile indicate that the [^{14}C]guazatine was poorly absorbed.

After fourteen daily administrations of [^{14}C]guazatine there was some evidence that radioactivity had accumulated to a slight extent in the liver (0.31-1.9 mg guazatine equivalents/kg), kidneys (0.09-1.2 mg/kg) and fat (0.01-0.17 mg/kg) but not in the plasma or carcase. These levels all decreased over a 14-day depuration period leaving mean levels of 0.14 mg/kg in the liver, 0.55 mg/kg in the kidneys, 0.05 mg/kg in the fat, <0.01 mg/kg in the plasma and 0.05 mg/kg in the carcase.

The most recent study (Kato *et al*., 1985) was published in the literature. Male rats were administered [^{14}C]guazatine triacetate labelled in the guanidine groups ([G-^{14}C]) or at the terminal positions of the octamethylene groups ([M-^{14}C]) according to the schedule shown in Table 2.

Table 2. Dose schedules in studies of metabolism of guazatine in rats (Kato *et al*., 1985).

Group	Type of study	Administration	Radiolabel	Dose, mg/kg bw	Specific activity, µCi/mg	No of rats
1	elimination/balance	oral	[G-^{14}C]	3	45.4	4
2[1]	elimination/balance	oral	[G-^{14}C]	30	14.3	4
3[1]	elimination/balance	intravenous	[G-^{14}C]	3	45.4	4
4	elimination/balance	intravenous	[M-^{14}C]	3	13.0	4
5	metabolism	intraperitoneal	[M-^{14}C]	15	13.0	4

Group	Type of study	Administration	Radiolabel	Dose, mg/kg bw	Specific activity, µCi/mg	No of rats
6	bile	intravenous	[G-^{14}C]	3	45.4	4
7	bile	intravenous	[M-^{14}C]	3	13.0	4

[1]Blood samples taken for pharmacokinetic analysis

The mean total recoveries of radioactivity after seven days were µ95% from both oral and intravenous doses and yielded bioavailability values of about 8% for the oral administration. About 90% of the administered dose was recovered from the faeces of both oral dose groups with the urine containing about 4.6% of the dose. In both these groups about 1.2% of the dose remained in the body after seven days. The recovery of radioactivity from the faeces was similar for the two intravenous dose groups at about 27% of the dose. Higher proportions were eliminated in the urine, about 56% of the guanidine label and 38% of the methylene label. In both intravenous groups biliary excretion was low with means of 0.6-1.3% of the dose recovered in the first 24 hours. Faecal elimination of both radiolabels was about 9.3%, indicating that it was not entirely accounted for by biliary excretion. Urinary excretion of the two radiolabels again differed, about 20% of the guanidine and 5% of the methylene label being eliminated. Whole-body autoradiography provided evidence that salivary and gastrointestinal secretions were playing a role in the faecal elimination of guazatine.

The data on blood pharmacokinetics indicated that the mean concentration after oral administration at 30 mg/kg was 0.13 ± 0.03 mg guazatine equivalents/kg, with a maximum about 10 minutes after dosing. The elimination half-life was calculated to be 26.7 ± 2.1 hours. Tissue concentrations varied with the dose route. The oral groups showed a dose proportionality and similar distribution profile, with the highest concentrations in the kidneys. The intravenous dose groups showed a difference in the distribution profiles from the two labels, although the kidneys of both groups contained the highest TRR.

Cows. A study by Cameron and Phillips (1986) designed to investigate the disposition of [^{14}C]guazatine in six lactating cows was in two phases. In phase 1 two cows were given single intraruminal doses of [^{14}C]guazatine, one at 0.1 mg/kg bw, a level consistent with normal exposure to residues in the feed, and the other at ten times his level. Excreta and milk were collected for the whole period of the experiment, and blood, expired air and saliva were sampled periodically to assess the plasma and excretion kinetics of the distribution. In phase 2, six cows received [^{14}C]guazatine by intraruminal administration twice daily for 10.5 days (total 21 doses). Three cows were dosed at 0.5 mg/kg bw and three at 0.05 mg/kg bw. All the milk was collected and blood was sampled repeatedly. Each cow was slaughtered at the peak ^{14}C level in the plasma for identification of the major tissue residues.

The mean recoveries of radioactivity from the faeces after the single doses of 1 and 0.1 mg/kg were both about 93% of the dose and the recovery from the urine during 168 hours reached about 1.6%, giving a mean total recovery of about 95%.

Plasma concentrations after the administration of 1 mg/kg were very low with a peak of about 0.003 mg guazatine equivalents/l at 12 hours, and those from the 0.1 mg/kg dose were below the limit of determination (0.0012 mg/l). After repeated doses of 0.5 mg/kg bw the levels of activity rose steadily to reach a plateau at day 6 which was maintained throughout the remainder of the dosing regime. The plateau levels in individual animals ranged from 0.007 to 0.014 mg/l. The levels in the animals dosed at 0.05 mg/kg bw were below the limit of reliable determination (<0.002 mg/l).

Excretion into the milk after single applications was minimal with levels below the limit of determination (0.0012 mg/l) from the low dose and a maximum recovery of about 0.02% of the dose after 24 hours from the high dose. After repeated dosing at 0.5 mg/kg the ^{14}C in the milk reached a plateau between 0.012 and 0.028 mg/l (in three cows) at 48-56 hours. After repeated dosing at 0.05 mg/kg residues were extremely low and appeared to reach a plateau by 48 hours between 0.0011 and 0.0038 mg/l. The highest proportion of the TRR found in the milk was in the whey fraction (a mean for all six animals of 58%), with 30% in the curd and 12% in the fat.

The liver and kidneys of the high-dose animals were found to contain the highest levels of absorbed radioactivity with respective means of 0.084 and 0.082 mg guazatine equivalents/kg. Muscle and fat contained negligible levels (<0.02 mg/kg).

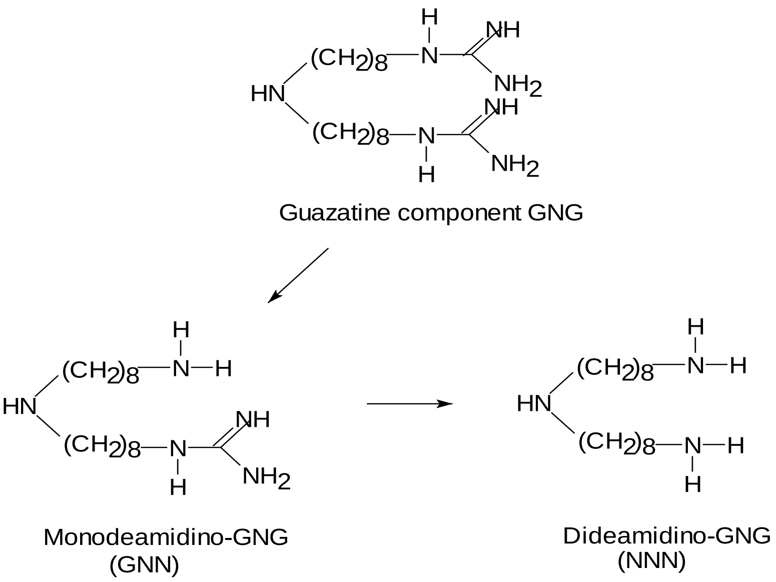
Biotransformation

Rats. In a preliminary study by Leegwater (1975) the differing ratios of $^{14}C/^{3}H$ in the various samples implied that some biotransformation of the administered guazatine had taken place but TLC of urine and faecal extracts did not produce any conclusive evidence of this. In the later study (Leegwater, 1980) TLC indicated the presence of one major and four minor radiolabelled components in the 0-24 hour urine which also appeared to be present in the administered guazatine preparation. A similar result was obtained with faecal extracts.

A review of the results obtained by Cameron *et al.* (1989) was recently reported (Prout, 1996). The results showed that up to 94% of an oral dose of [^{14}C]guazatine had been eliminated as the parent material within 96 hours in the faeces, with 6-7% of the dose being absorbed but largely eliminated in the urine in the same time. The biliary route appeared to play no, or very little, part in the elimination of absorbed material. The absorbed radioactivity appeared to be largely associated with two components of guazatine, G and GGG. The residues in the tissues support the biokinetic evidence for concluding that guazatine is poorly absorbed after oral administration. The metabolism of guazatine components in the rat, irrespective of the number of guanidino groups, mainly involved deamidination to the corresponding amino compounds which were largely excreted in the urine.

The most recent investigation (Kato *et al.*, 1985) indicated the presence of 16 components of which two were major. The major residues identified in the kidney extracts were guazatine, 1,1'-iminodi(octamethylene)-8-amino-8'-guanidine and 1,1'-iminodi(octamethylene)diamine. In the faecal extracts guazatine was the major component with monodeamidinoguazatine also identified. The metabolites in the urine were relatively hydrophilic. Thirteen components were detected but only monodeamidino-guazatine was identified. A general view of the metabolism is shown in Figure 1.

Figure 1. Proposed general metabolic pathway of guazatine components in mammals, using component GNG as an example (Kato *et al.*, 1985).



In general the results of this study are consistent with those obtained in the preceding studies on rats. The deamidination of a component of guazatine was shown to be one of the main routes of biotransformation in rats before the amines are largely excreted in the urine. The metabolic pathway is similar for the other guanidated components of guazatine (GG, GN, GGG, GGN).

Cows. Cameron *et al.* (1984) investigated the degradation of octyl- and guanidine-labelled [^{14}C]guazatine in a rumen fermentation system *in vitro* and a liver homogenate system. No significant degradation of either labelled compound was noted during incubation with fresh rumen contents although some radioactivity was recovered in the traps for gaseous products: 1.4% of the original guanidine radioactivity and 0.067% of the octyl. Aerobic incubation with fresh bovine liver homogenates however produced about 40% loss of radioactivity from the incubation mixture containing [*guanidine*-^{14}C]guazatine and about 10% loss of the octyl label. These losses were not accounted for by the radioactivity in the trapped gases which only accounted for 1.9% and 0.4% of the guanidine and octyl labels respectively. Analyses by TLC did not reveal any significant qualitative differences from the test materials.

In the study by Cameron and Phillips (1986) the radioactivity in the faeces (93% of the dose) and liver had similar chromatographic properties to the test material. The urine was found to contain polar component(s) with a chromatographic profile similar to that from the kidneys. Definitive identifications were not possible.

Plant Metabolism

The uptake, translocation and metabolism of [^{14}C]guazatine labelled at the terminal carbons of the octyl chains was investigated in winter wheat (Caley *et al.*, 1990a). The compound was applied as a seed dressing formulation at a level of 1.05 g/kg seed. Samples were collected about 5 weeks after emergence and again at maturity.

The uptake of guazatine from the seeds was low. At the first harvest the mean uptake into the plants was 0.07 % of the applied radioactivity, equivalent to 0.18 mg/kg as guazatine (Table 3). At maturity the ^{14}C in the plants still represented 0.07 % of the applied radioactivity but because of dilution by growth the concentrations in the plants from the treated and control plots were now similar and all mean values were less than 0.05 mg guazatine equivalents/kg (Table 4). As most of the residues at the final harvest were below the limit of reliable determination (0.01 mg guazatine equivalents/kg), the small differences observed between individual components and between test and control plots are not considered to be significant, but the mean concentrations of radioactivity in the grain were consistently lower than those in the straw and chaff. The levels of radioactivity in the soil samples were very low, almost all below the limit of reliable determination. The levels were too low to determine the nature of the residues or the extent of metabolism.

Table 3. ^{14}C in wheat plants 5 weeks after emergence, following application of [^{14}C]guazatine as a seed dressing (Caley *et al.*, 1990a).

^{14}C, mg/kg as guazatine equivalents, in plants (fresh weight)			
Plant no.	Plot 1	Plot 2	Plot 3
2	0.04	0.12	0.02
3	0.07	0.13	0.02
4	0.01	0.12	<0.01
5	0.03	0.16	<0.01
6	0.03	0.16	<0.01
7	0.05	0.76[1]	<0.01
8	<0.01	0.19	<0.01
9	0.01	0.17	<0.01
10	0.01	0.35	<0.01
Mean	0.03	0.18	0.01

Plots 1 and 3 untreated; plot 2 treated
[1]Outlier

Table 4. ^{14}C in wheat plants at final harvest following application of [^{14}C]guazatine as seed dressing (Caley *et al.*, 1990a).

Plant no.	^{14}C, mg/kg as guazatine equivalents								
	Plot 1			Plot 2			Plot 3		
	Straw	Chaff	Grain	Straw	Chaff	Grain	Straw	Chaff	Grain
1	0.04	0.09	<0.01	0.025	0.02	<0.01	<0.01	0.01	<0.01
2	0.01	0.06	0.01	0.03	0.02	<0.01	0.01	0.02	<0.01
3	0.04	0.03	0.01	0.01	0.01	<0.01	0.01	0.01	<0.01
4	0.02	0.01	<0.01	0.01	0.02	<0.01	<0.01	0.01	<0.01
5	<0.01	0.04	0.01	0.02	0.01	<0.01	0.01	0.02	<0.01
6	0.01	0.04	<0.01	0.03	0.02	<0.01	0.02	0.02	0.01

Plant no.	^{14}C, mg/kg as guazatine equivalents								
	Plot 1			Plot 2			Plot 3		
	Straw	Chaff	Grain	Straw	Chaff	Grain	Straw	Chaff	Grain
7	0.02	0.05	<0.01	0.02	0.01	<0.01	0.02	0.02	0.01
8	0.02	0.03	<0.01	0.03	0.09	<0.01	0.01	0.02	0.01
9	<0.01	0.01	<0.01	0.01	0.02	<0.01	0.02	0.02	<0.01
10	0.01	0.04	0.01	0.02	0.01	<0.01	0.02	0.03	0.01
Mean	0.02	0.04	0.01	0.02	0.02	<0.01	0.01	0.02	<0.01

Plots 1 and 3 untreated; plot 2 treated

The foliar application of [^{14}C]guazatine at a rate of 1.1 kg ai/ha to wheat plants 11 weeks before harvest resulted in mean ^{14}C residues equivalent to 29 mg guazatine/kg in the straw and 18 mg/kg in the chaff. The levels in the grain were significantly lower at to 0.8 mg/kg, equivalent to 1.3% of the total radioactivity present at maturity (Caley *et al.*,1990b).

In a laboratory study by Lowden *et al.* (1996), a mixture of GG, GN and GGG were applied to seed surfaces, and the seeds were planted in soil in metabolism vessels. Most of the seeds germinated; it was possible to distinguish the seeds from the soil and extract the seeds separately up to 29 days after planting. Analysis of these extracts indicated a change in the profile of components present on the seed with levels of the three compounds generally decreasing. There was a concomitant generation of $^{14}CO_2$ (Table 5; see also "Environmental fate in soil" below).

Table 5. Levels of GG, GGG and GN in or on treated wheat seed (Lowden *et al.*, 1996).

	^{14}C, % of applied							
	Loamy sand		Sandy loam		Low organic loamy sand		Clay loam	
Day 0	Unit							
	1	2	36	37	72	106	107	
GG	13.2	15.4	21.2	9.82	9.79	16.6	18.1	
GN	3.36	6.64	4.62	2.02	0	1.77	6.62	
GGG	9.4	13.1	6.92	7.01	15.1	14.9	2.64	
Day 7	Unit							
	11	14	40	54	80	82	110	120
GG	13.7	15.6	16.1	26.6	15.85	4.41	12.8	29.8
GN	5.57	1.64	2.0	0	28.0	0	7.65	9.4
GGG	3.7	0	3.05	21.7	12.2	0	0	3.81
Day 15	Unit							
	4	5	48	55	79	95	115	116
GG	12.1	7.01	3.7	5.56	8.07	9.77	3.91	6.65
GN	3.21	1.43	0.25	0.78	31.1	1.31	0.96	0

	^{14}C, % of applied							
	Loamy sand		Sandy loam		Low organic loamy sand		Clay loam	
GGG	9.55	5.26	0.88	8.6	2.97	1.76	0.7	1.18
Day 29	Unit							
	19	20	49	50	85	99	118	121
GG	3.38	0.6	10.1	0	6.27	0	0	0
GN	1.24	0	0.3	0	1.5	0	0	0
GGG	2.69	7.46	5.15	0.01	0	0	0	0

The penetration, translocation and metabolism of [^{14}C]guazatine applied to dwarf apple trees were determined over a period of 12 weeks under laboratory conditions (Sato *et al.*, 1985). When the guazatine was applied to the leaf surface or the fruit by brushing with 0.05 or 0.1 kg ai/hl its translocation during 12 weeks was extremely limited. Autoradiography showed no observable movement in the leaves, and in the fruit the radiolabelled material was principally retained on the surface. Quantitative determination of the TRR in treated leaves indicated a slow disappearance (half-life 67 weeks). After 12 weeks approximately 87% of the applied [^{14}C]guazatine was recovered, and only 21% had penetrated the leaf tissues. The results are given in Table 6.

Table 6. Distribution of radioactivity in fractions from apple leaves (Sato *et al.*,1985).

Fraction	Identity	^{14}C, % of applied, at weeks after treatment					
		0	1	2	4	8	12
Surface washings		93.5	85.6	86.2	84.5	75.5	66.0
	Guazatine	na	67.9	67.0	64.2	61.9	52.8
	PM	na	7.4	9.0	10.3	7.7	6.3
	Others	na	10.2	10.2	10.0	5.9	6.9
Acetic methanol extracts		1.7	4.8	4.0	4.2	5.9	7.2
	Guazatine	na	2.5	1.3	1.1	2.6	1.7
	PM	na	0.3	0.4	0.4	0.7	0.8
	Others	na	2.0	1.8	2.7	2.6	4.6
Methanolic NaOH extracts		3.6	6.0	6.2	6.5	9.0	10.3
Aqueous		0.1	1.0	1.2	1.4	2.2	2.8
$CHCl_3$		3.5	5.0	5.0	5.1	6.8	7.5
	Guazatine	na	4.4	4.6	4.4	5.7	6.5
	PM	na	0.1	0.1	0.3	0.3	0.2
	Others	na	0.5	0.2	0.4	0.8	0.8
Unextractable residues		0.3	1.4	2.2	2.5	2.6	3.8
Total		99.2	97.9	98.6	97.7	93.0	87.3

PM: major photodegradation product
na: not analysed

Table 7. Distribution of radioactivity in fractions from apple fruits, 12 weeks after treatment (Sato *et al.*,1985).

Fraction	Identity	^{14}C, %	
Surface washings		61.8	
	Guazatine		56.6
	PM		2.9
	Others		2.7
Acetic acid methanol exctracts		9.4	
	Guazatine		5.7
	PM		1.0
	Others		2.7
Methanolic NaOH extracts		23.6	
Aqueous phase		3.0	
$CHCl_3$ phase		20.6	
	Guazatine		18.7
	PM		0.6
	Others		1.3
Unextractable residues		5.2	

PM: major photodegradation product
Each value is the mean of duplicate experiments

Quantitative determination of the TRR in the fruits also confirmed the results of the autoradiography. Table 7 shows that 81% of the TRR in or on fruits analysed 12 weeks after treatment was still the parent mixture. The remainder comprised a major photodegradation product (4.5%), other extractable compounds (6.7%), and unextractable residues (5.2%). A similar distribution (61% of the residues as the parent mixture) was seen in leaves (Table 6).

The availability of guazatine residues in the soil to soya bean and rice plants was investigated under laboratory conditions with three Japanese soils (Kumagaya clay loam and Chiba loam under upland conditions, Nagaoka clay loam under flooded conditions) which were treated with 5 mg/kg of [^{14}C]guazatine triacetate (dry weight basis); some samples were kept for 26 weeks before planting to produce aged soil residues (Sato *et al.*, 1984).

The ^{14}C was determined in separate parts of soya bean plants grown in the Kumagaya and Chiba upland soils. Four weeks after planting, the TRR expressed on a dry weight basis in each part of the plants was less than one-fifth of that in the surrounding soil. The total recoveries of ^{14}C from the foliage and whole plants in the Kumagaya soil were only 0.08 and 0.13% of the applied radioactivity respectively (Table 8).

Table 8. Uptake of ^{14}C from upland soils by soya bean plants (Sato *et al*., 1984).

Sample	Kumagaya soil Weeks after planting 2	4	9	Chiba soil Weeks after planting 2	4	9
^{14}C as guazatine equivalents, mg/kg dry weight						
Stalk	0.036	0.478	-	0.034	0.473	-
First true leaves	0.035	0.259	-	0.030	0.229	-
Second true leaves	0.033	0.337	-	0.034	0.291	-
Third true leaves	0.038	0.77	-	0.035	0.392	-
Fourth true leaves	0.039	0.605	-	0.038	0.621	-
Developing leaves	-	0.329	-	-	0.443	-
Root	0.770	0.909	-	0.127	0.587	-
Pods	-	-	0.052	-	-	0.076
Seeds	-	-	0.053	-	-	0.084
% of applied ^{14}C						
Foliage	0.004	0.077	-	0.003	0.116	-
Whole plant	0.026	0.125	-	0.010	0.179	-

The residues in flooded Nagaoka soil were also not available to rice plants, which absorbed only 0.13% of the ^{14}C applied to the soil during a period of 4 weeks when the residues had been aged for 26 weeks (Table 9).

Table 9. Uptake of aged and freshly deposited [^{14}C]guazatine residues from flooded soils by rice plants (Sato *et al*., 1984).

	Residues aged 1 hour			Residues aged 26 weeks		
Time after planting (weeks)	1	2	4	1	2	4
^{14}C as guazatine equivalents, mg/kg dry weight						
Shoot	15.4	5.74	2.37	0.84	0.48	0.23
Root	19.6	6.61	2.36	0.91	0.55	0.34
% of applied ^{14}C						
Shoot	0.52	0.76	0.67	0.04	0.07	0.05
Whole plant	0.98	1.27	1.17	0.07	0.11	0.13

When the roots were treated with a nutrient solution containing 5 mg/kg of [^{14}C]guazatine they absorbed the extremely high concentration of ^{14}C of 2700 mg/kg in days, but there was little translocation into the shoots which contained only 7 mg/kg after 7 days.

Environmental fate in soil

Degradation

In a laboratory study (Lowden *et al.*, 1996) a mixture of radiolabelled GGG, GG and GN, the three main components of guazatine, was applied to wheat seeds at the commercial rate of 0.6 g ai/kg seed and the seeds were planted in four soils. The treatment rate is equivalent to an application to the soil of about 0.12 kg ai/ha at a sowing rate of 200 kg of wheat seeds per hectare. The seeds were planted in flasks containing 100 g (oven-dried equivalent) of soil, one seed per flask. The soils were a loamy sand, a sandy loam, a low organic matter loamy sand and a clay loam. The three compounds all decreased steadily with time. As early as day 7 significant quantities of $^{14}CO_2$ (4-14% of the applied radioactivity) were observed: similar behaviour of the compounds was observed in a leaching study (McMillan-Staff and Austin, 1996). The decrease of the initially-applied compounds was accompanied by the appearance of many minor degradation products, at low levels at all times (<0.05 mg guazatine equivalents/kg). These could be chromatographically characterized as diamines and monoamines, but were not positively identified owing to their very low concentrations. The deguanidated products GN and GNG have been confirmed in soil extracts. The results show that degradation occurs by two routes: deguanidation and oxidation of the octyl chains. This is consistent with a previous Swedish study on guazatine (Björk and Siirala-Hansen, 1986) which postulated deguanidation followed by oxidative degradation of the hydrocarbon groups. The other components in the commercial guazatine mixture would be expected to show a similar pattern of degradation. The components of technical guazatine not represented in this study (about 30%) comprise about 15% triamines, 11% tetramines and 4% pentamines or higher amines. The study has shown rapid degradation of the fully guanidated triamine and the other triamines would be expected to show similar or more rapid degradation. There is no reason to believe that the mechanisms of degradation of the higher oligomers would be significantly different since the components are chemically very similar to those studied. Figure 2 shows the proposed metabolic pathways using the fully guanidated triamine, GGG, as an example. For the sake of clarity the deguanidation reactions have been shown as occurring before the oxidations. In compounds that are only partially guanidated (either originally or as a result of the degradative process) the reactions are likely to be concomitant (Björk and Siirala-Hansen, 1986).

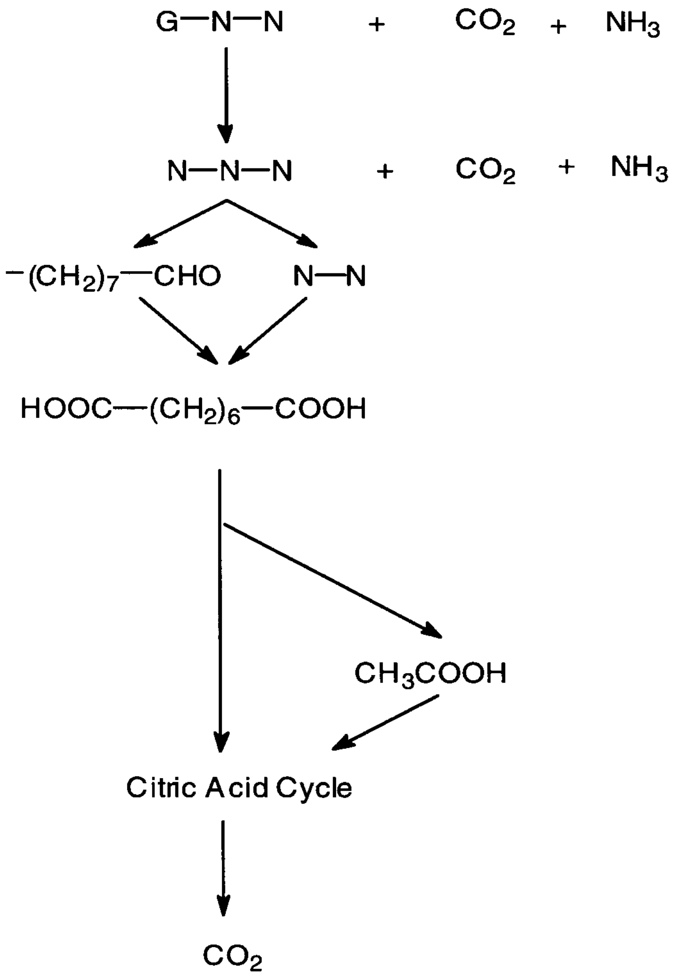
Half-lives of the components of guazatine in this study have been calculated as 62 days in loamy sand, 104 days in clay loam, 106 days in loamy sand with low organic matter, and 176 days in sandy loam. However, these were based on the total radioactivity unextractable by KOH reflux plus the CO_2 generated and so represent an extreme case. It should also be noted that the seeds were not allowed to develop far beyond germination and under field conditions, where the plants become established, degradation might be expected to be more rapid.

These results are also consistent with the previously mentioned study by Björk and Siirala-Hansen (1986). The half-life of guazatine dressed on to wheat seed at 0.75 g/kg seed was 20 days when the seed was incubated in Petri dishes at 20°C, and 80 days when the seed was sown in pots of soil stored outside. The data demonstrate a substantial influence of the test system on the rate of degradation of guanidated amine acetates.

<u>Leaching</u>

A study by McMillan-Staff and Austin (1996) was designed to investigate the mobility of three of the main compounds of guazatine when applied to wheat seeds which were subsequently planted in the tops of columns of three different soils which were leached to simulate rainfall. The compounds were labelled with ^{14}C and two seeds were planted in the top of each column. After the equivalent of 50 cm of rain the guazatine components were still associated with the seeds or the soil surrounding them. The compounds that had moved from the seeds to the soil were not leached as only very small amounts of radiolabelled material were found either in the soil below that in which the seeds had been planted or in the leachate from the columns. During the leaching period there was significant mineralization of these compounds to carbon dioxide, showing that they are readily degraded in soil (probably at the seed surface).

Figure 2. Proposed pathways of degradation of guazatine components in soil, exemplified by the fully guanidated triamine (GGG).



The major components of guazatine (GG, GN and GGG) have been shown to be immobile in a range of soil types under conditions of simulated rainfall, while being mineralized to carbon dioxide. It is reasonable to extrapolate the results to the other components of guazatine. They are all similar physico-chemically in that they are ionic and differ only in the number of hydrocarbon chains and the combination of guanidated and unguanidated amine groups, and so would be likely to show similar leaching behaviour and degradation. The results, therefore, indicate that no movement of guazatine to deeper soil layers would be expected and that the potential for groundwater contamination would be extremely low.

METHODS OF RESIDUE ANALYSIS

Analytical methods

Such a complex mixture as guazatine presents a problem in choosing a residue analytical method. It is not practical to attempt analysis for all the components so some alternative is necessary. Two approaches have been used: (1) "total residue" methods involving the generation of a single compound and (2) the use of a major component as a "marker", with the inclusion of a correction factor to give the total residue (GG, representing 30% of the total free amine content of the product, is the marker of choice. All the residues are expressed as guazatine.

Citrus fruits. In a total residue method (Thornberg, 1979a; Thorstensson and Stensiö, 1984; Stensiö and Thorstensson, 1990) the guanidines are extracted as ion-pairs with picric acid into butanol, then extracted with sulfuric acid and hydrolysed to the parent amines. The amines are extracted into benzene and converted to trifluoroacetylated bis(8-amino-octyl)amine, which is determined by GC-MS. The method was valided by Stensiö (1986). The limits of determination for fortified orange pulp, wet peel and dried peel were 0.05, 0.2 and 1 mg/kg respectively. Table 10 shows the results of recovery studies.

Table 10. Recoveries of guazatine from fortified orange products (Stensiö, 1986).

Sample	Fortification, mg/kg	Recovery, %	Relative SD, %
Finisher pulp	0.05	128	11
	0.1	118	7
	0.2	119	7
Wet peel	0.2	113	20
	0.4	96	24
	0.8	110	5
Dried peel	1	138	12
	2	113	10
	4	111	12

Sugar cane. The method described above for citrus fruits was used by Thornberg (1979b) to determine guazatine in sugar cane. It was stated in the report that the limit of detection was 0.1 mg/kg but no validation of the method was reported.

Cereals. A method based on the aqueous acid or methanolic alkaline extraction of the active ingredient from the crop, followed by alkaline hydrolysis to bis(8-amino-octyl)amine was reported (Anon., 1974). The triamine was extracted into butanol and determined by GLC with a flame ionisation or nitrogen-selective detector. The recovery from oats fortified with guazatine at 1 mg/kg averaged 63% and ranged from 56 to 71% (7 values).

A residue method has been applied to rice by Kobayashi *et al.* (1977). After extraction with alkaline methanol, clean-up by liquid-liquid partition and treatment with hexafluoroacetyl acetone, the residues were determined by GLC with an AFID in the nitrogen mode as bis[3,5-bis(trifluoromethyl)pyrimidyl-1-amino]-8,8´-dioctylamine. The limit of determination was 0.05 mg/kg and recoveries ranged from 80 to 99%.

Guazatine was extracted as an ion-pair with picric acid from cereal grains into butanol (Thornberg, 1976a). After extraction with sulfuric acid, the guazatine was hydrolysed to the triamine which was extracted into benzene and, after trifluoroacetylation, determined by GC-MS. A detection limit of 0.1 mg/kg was reported but the recovery was only 10-20%.

In a later method (Thornberg, 1979c) grain or straw samples were extracted with hydrochloric acid and the guanidines extracted as ion-pairs with picric acid from the aqueous phase into butanol. After extraction with sulfuric acid the guanidines were hydrolysed to the parent amines. The amines were extracted into benzene and converted to the trifluoroacetylated derivative, *N,N*-bis(8-trifluoroacetamido-octyl)trifluoroacetamide, which was determined by GC-MS using multiple ion detection. A limit of detection of 0.05 mg/kg and a recovery of about 50% were claimed in the text but no validation was reported.

This method was improved by Risholm-Sundman *et al.* (1988) by the addition of an internal standard before the extraction. The limit of determination was stated to be 0.05 mg/kg but the lowest fortification level was 0.5 mg/kg. The recoveries from grain were 68-118 % and from straw 100-116 % after fortification with 0.5, 3 and 10 mg/kg.

A new method was developed by Stensiö (1990). The guanidino compounds of guazatine are all extracted, but the purification and derivatization steps are designed for the determination of only one of the major components, 1,8-diguanidino-octane (GG). After addition of internal standard (1,6-diguanidinohexane), guazatine is extracted from the sample with hydrochloric acid. Part of the extract is diluted with water and a 25% ammoniacal solution is added. Purification is carried out on a cation exchange column which is eluted with a 1:1 mixture of water and glacial acetic acid. Guazatine and the internal standard are derivatized in pyridine with hexafluoroacetylacetone, and the derivatives are cleaned up on an alumina column. The derivative of 1,8-diguanidino-octane is determined by GC-MS with internal standard calibration. The limit of determination is 0.05 mg/kg guazatine. The relative standard deviations calculated from 28 analyses of spiked wheat grain samples ranged from 2.9 to 20% in the concentration range 0.05-1 mg/kg guazatine, with recoveries of 64 to 82%.

This performance was maintained in more recent work (Fuchsbichler, 1992a,b) carried out with the compound GG and determination as the same derivative, again by GC-MS with internal standard calibration. The recoveries from cereal ears, straw and grain are shown in Table 11.

Table 11. Recoveries of GG from cereal fractions (Fuchsbichler, 1992a,b).

Sample	GG fortification, mg/kg	Recovery, %
Ears	0.2	82 (mean of 2)
Ears	0.1	97 (mean of 2)
Ears	0.05	96 (mean of 2)
Straw	1.0	82
Straw	0.1	78
Grain	0.2	110 (mean of 4)
Grain	0.05	97 (mean of 4)

Stability of residues in stored analytical samples

The stability of residues of guazatine in cereals was studied by storing analysed wheat samples (straw, grain and ears) at -20ºC and reanalysing after 2 years (Risholm-Sundman and Jonsson,1989). The study was inadequate as the analytical method had not been validated.

Definition of the residue

The metabolism of guazatine has not been fully characterized in either animals or plants. The Meeting concluded that its residues in products of animal origin could not be defined.

The main uses of guazatine are for the seed treatment of cereals and post-harvest application to citrus fruits. The Meeting concluded that the available studies are adequate only to define the residue arising from the seed treatment of cereals. Should further uses be planned in future (e.g. foliar sprays or use on plants other than cereals), detailed metabolism studies would be required.

The determination of total guazatine residues is based on conversion to the triamine bis(8-amino-octyl)amine, which also occurs as a metabolite. Modern analytical methods using 1,8-diguanidino-octane (GG), one of the main components of guazatine, as a marker are more specific.

The Meeting recommended that the definition of the residue for enforcement purposes should be changed to "octane-1,8-diyldiguanidine", i.e. 1,8-diguanidino-octane, "GG". Assuming that the content of GG is 30% of the total free base content, a conversion factor of 3 is required for risk assessment purposes for commodities of plant origin.

Residue definition for enforcement purposes for cereal grains: octane-1,8-diyldiguanidine ("GG"), expressed as octane-1,8-diyldiguanidine.

Residue definition for risk assessment purposes for cereal grains: guazatine.

USE PATTERN

Guazatine is a non-systemic contact fungicide which disturbs the membrane function of fungi, decreasing the cellular permeability. The decrease in oxidative capacity is probably due to the inhibition of the uptake of certain substrates rather then a direct effect on enzymes. It acts at several sites, which prevents resistance.

Guazatine controls a wide range of seed-borne diseases of cereals, e.g. seedling blight *(fusarium spp.)*, glume blotch *(septoria)*, common bunt *(tilletia spp.)*, common root rot *(helminthosporium)* and smut *(ustilago)*. It is used on citrus fruits as a bulk dip after harvest, in the packing line as a spray and in washing installations to disinfect the process water. It controls sour rot *(geotrichum candidum)*, green mould *(penicillium digitatum)* and blue mould *(penicillium italicum).*

The Meeting received information on GAP from the manufacturer and the governments of Australia (Anon., 1996a), Germany (Anon., 1996b), Norway (Anon., 1997a), The Netherlands (Anon., 1997b) and the UK (Anon., 1997c). Tables 12-14 show the registered uses of guazatine for seed treatment, post-harvest application, and other uses. The application rates refer only to guazatine, although some formulations are mixed.

Table 12. Registered uses of guazatine for seed treatment. All single applications.

Crop	Country	Application	
		Form	Rate, a ai/kg seed
Wheat, rye, barley, oats, triticale	Austria	LS 350 g/l LS 300 g/l	0.875 0.6
	Belarussia	LS 300 g/l	0.45-6
	Belgium	LS 350 g/l LS 300 g/l	1.05 0.9
	Brazil	WP 250 g/kg	0.75
	Bulgaria	LS 300 g/l	0.6
	Croatia	LS 300 g/l	0.45-6
	Czech Republic	LS 350 g/l LS 300 g/l	0.7 0.45-0.6
	Denmark	LS 300 g/l LS 200 g/l LS 25 g/l	0.6 0.45 0.05
	Finland	LS 300 g/l	0.6
	France	LS 265 g/l FS 400 g/l	0.8
	Germany	LS 350 g/l LS 300 g/l LS 200 g/l FS 300 g/l FS 150 g/l[1]	0.7 0.6 0.4 0.6 0.6
	Hungary	LS 350 g/l LS 300 g/l	0.7-1.05 0.6-0.9
	Italy	LS 300 g/l LS 325 g/l	0.6
	Kahzahkstan	LS 300 g/l	0.45-0.6
	Macedonia	LS 350 g/l	0.7-1.05
	The Netherlands	LS 350 g/l LS 300 g/l	0.7 0.6-0.7
	Norway	LS 300 g/l LS 25 g/l	0.6 0.05
	Poland	LS 350 g/l LS 300 g/l	0.7-1.05 0.45- 0.6
	Romania	LS 350 g/l LS 300 g/l	0.7 -1.05 0.45- 0.6

Crop	Country	Application	
		Form	Rate, a ai/kg seed
	Russia	LS 350 g/l LS 300 g/l	0.7 0.45- 0.6
	South Africa	SL 400 g/l	0.8
	Spain	LS 300 g/l	0.6-0.9
	Sweden	LS 400 g/l LS 350 g/l LS 300 g/l LS 150 g/l LS 25 g/l	0.6 0.6 0.6 0.6 0.05
	UK	LS 300 g/l	0.6-0.9
	Ukraine	LS 350 g/l LS 300 g/l	0.7 0.45-0.6
	Uzbekistan	LS 350 g/l	0.7

[1]Pending

Table 13. Registered post-harvest uses of guazatine. All single applications.

Crop	Country	Form	Application	
			Method	Rate, kg ai/hl
Citrus	Argentina	SL 40 g/l	watering	0.2
	Australia	SL 400 g/l	watering	0.052
	Greece	SL 400 g/l	watering	0.16
	Morocco	SL 200 g/l	watering	0.1
	Spain	SL 200 g/l	watering	0.06-0.1
	South Africa	Wax 3 g/l[1] SL 200 g/l	 dipping	0.3 0.1
	Uruguay	SL 40 g/l	watering	0.2
Rockmelons	Australia	SL 400 g/l	Dipping[2]	0.052
Tomatoes	Australia	SL 400 g/l	Spraying[3]	0.12

[1]Apply to freshly washed and dry citrus at a rate between 1.3 and 1.5 l per 1000 kg of fruit to obtain a residue of 3.9 to 4.5 mg/kg
[2]Dipping for 1 minute within 24 h of harvest
[3]Spraying for 30 seconds as soon as possible, not later than 24 h after harvest

Table 14. Other registered uses of guazatine.

	Country	Form	Application			PHI, days
			Method	Rate	Number	
Rape	Germany	SL 600 g/l	spraying	1.2 kg ai/ha	1	56

	Country	Form	Application			PHI, days
			Method	Rate	Number	
Sugar cane	South Africa	SL 400 g/l	treatment of plant segment before planting	0.08 kg ai/hl water	1	

RESIDUES RESULTING FROM SUPERVISED TRIALS

The results of supervised residue trials on cereals, sugar cane and citrus fruit are shown in Tables 15 to 17. There were no trials of spraying rape, for which there is German GAP, or on post-harvest applications to rockmelons and tomatoes (Australian GAP).

Underlined residues in the Tables reflect current GAP. Double underlined residues have been selected for the estimation of supervised trials median residue (STMR) levels. All residues are expressed as guazatine.

Cereals (Table 15). The results of 84 trials on barley (21), oats (12), rye (4) and wheat (47) were reported from field trials in Brazil (Risholm-Sundman, 1984, 1986), France (Müller, 1996a-g), Germany (Fuchsbichler, 1995, 1996; Thornberg, 1978; Jonsson and Risholm-Sundman, 1986, 1987, 1988; Strätz, 1994, 1996), Italy (Müller, 1996h,i), Sweden (Thornberg, 1977), South Africa (Thornberg, 1976b) and the UK (Anon., 1972, 1973). As the analytical methods used before 1990 were not satisfactory only the wheat trials in 1994 and 1995 could be used for evaluation.

Table 15. Residues of guazatine in cereals from seed treatments. All single applications.

Crop, Country, Year	Form	Application, g ai/kg seed	Sample	PHI, days	Residues, mg/kg	Report
Barley, Spring						
Germany, 1979	WP	0.9	grain straw	120 120	<0.05 <0.05	438249 R 15
Germany, 1978	LS	0.9	grain straw	153 153	<0.05 <0.05	438248 R 42
Germany, 1979	LS	0.6	grain straw	151 151	<0.05 <0.05	438249 R 13
Germany, 1979	LS	0.4	grain straw	120 120	<0.05 <0.05	438249 R 17
Sweden,	LS	0.6	grain	112	<0.1	438245

Crop, Country, Year	Form	Application, g ai/kg seed	Sample	PHI, days	Residues, mg/kg	Report
1974						E-län
Sweden, 1974	LS	0.6	grain	127	<0.1	438245 D-län
Sweden, 1974	LS	0.6	grain	144	<0.1	438245 C-län
UK, 1972	LS	0.8	grain	154	<0.1	RT/19/72
UK, 1972	WP	0.8	grain	154	<0.1	RT/25/72
UK, 1972	LS	0.8	grain	150	<0.1	RT/20/72
UK, 1972	WP	0.8	grain	150	<0.1	RT/26/72
UK, 1973	LS	0.8	grain	151	<0.1	RT/12/73
UK, 1973	LS	0.8	grain	135	<0.1	RT/13/73
UK, 1973	WP	0.6	grain	151	<0.1	RT/15/73
UK, 1973	WP	0.6	grain	135	<0.1	RT/14/73
UK, 1973	WS	0.6	grain	151	<0.1	RT/16/73
UK, 1973	WS	0.6	grain	135	<0.1	RT/17/73
UK, 1973	WS	0.6	grain	151	<0.1	RT/19/73
UK, 1973	WS	0.6	grain	135	<0.1	RT/18/73
Barley, Winter						
Germany 1977	LS	0.4	grain straw	287 287	<0.05 <0.05	438248 R 48

Crop, Country, Year	Form	Application, g ai/kg seed	Sample	PHI, days	Residues, mg/kg	Report
Germany 1977	LS	0.4	grain straw	258 258	<0.05 <0.05	438248 R 49
Oats						
Germany, 1978	LS	0.9	grain straw	123 123	<0.05 <0.05	438248 R 44
Germany, 1978	LS	0.9	grain straw	140 140	<0.05 <0.05	438248 R 170
Germany, 1978	LS	0.9	grain straw	145 145	<0.05 <0.05	438248 R 171
Germany, 1979	LS	0.6	grain straw	123 123	<0.05 <0.05	438249 R 12
Germany, 1985	LS	0.6	foliage grain straw	59 132 132	<0.05 <0.05 <0.05	86-AC-0322 Rs 8501 B3
Germany, 1985	LS	0.6	foliage grain straw	44 127 127	<0.05 <0.05 <0.05	86-AC-0322 Rs 8501 E2
Sweden, 1974	LS	1.0	grain	154	<0.1	438245
Sweden, 1974	LS	1.0	grain	178	<0.1	438245
UK, 1972	LS	0.8	grain	154	<0.1	RT/22/72
UK, 1972	WP	0.8	grain	154	<0.1	RT/24/72
UK, 1972	LS	0.8	grain	149	<0.1	RT/21/72

Crop, Country, Year	Form	Application, g ai/kg seed	Sample	PHI, days	Residues, mg/kg	Report
UK, 1972	WP	0.8	grain	149	<0.1	RT/27/72
Wheat						
Brazil, 1985	LS	1.5	grain	109	<0.05	86AC0325
Brazil, 1985	LS	1.5	grain	114	<0.05	86AC0325
Brazil, 1985	LS	1.5	grain	114	<0.05	86AC0326
Brazil, 1985	LS	1.5	grain	109	<0.05	86AC0326
South Africa[1], 1976	LS	0.4	grain		<0.1	438247
South Africa[1], 1976	LS	0.8	grain		<0.1	438247
South Africa[1], 1976	LS	1.2	grain		<0.1	438247
Wheat, Spring						
France, 1995	FS	0.8	grain straw	140 140	<0.05 <0.1	RD/CRLD/ AN/9615878 95507BX1
France, 1995	FS	0.8	grain straw	128 128	<0.05 <0.1	RD/CRLD/ AN/9615878 95507AM1
France, 1995	FS	0.8	grain straw	131 131	<0.05 <0.1	RD/CRLD/ AN/9615878 95507RS1

Crop, Country, Year	Form	Application, g ai/kg seed	Sample	PHI, days	Residues, mg/kg	Report
France, 1995	FS	0.8	grain straw	145 145	≤0.05 ≤0.1	RD/CRLD/ AN/9615878 95507LY1
France, 1995	FS	0.8	shoot grain straw	25 47 74 131 131	0.17 <0.1 <0.1 ≤0.05 ≤0.1	RD/CRLD/ AN/9615911 95518RS1
Germany, 1979	LS	0.6	grain straw	117 117	<0.05 <0.05	438249 R 14
Germany, 1979	LS	0.4	grain straw	115 115	<0.05 <0.05	438249 R 18
Germany, 1979	WP	0.9	grain straw	120 120	<0.05 <0.05	438249 R 16
Germany, 1981	LS	0.6	grain straw	144 144	<0.05 <0.05	438249 R 46
Germany, 1981	LS	0.6	grain straw	156 156	<0.05 <0.05	438249 R 47
Germany, 1985	LS	0.6	foliage grain straw	81 162 162	<0.05 <0.05 <0.05	86-AC-0322 Rs 8501 B1
Germany, 1985	LS	0.6	foliage grain straw	59 140 140	<0.05 <0.05 <0.05	86-AC-0322 Rs 8501 E1
Germany, 1986	LS	0.6	foliage grain straw	32 125 125	<0.05 <0.05 <0.05	87-AC-0172 Rs 8602 B2
Germany, 1986	LS	0.6	foliage grain straw	80 167 167	<0.05 <0.05 <0.05	88-AC-0215 CGD 34-86R

Crop, Country, Year	Form	Application, g ai/kg seed	Sample	PHI, days	Residues, mg/kg	Report
Germany, 1986	LS	0.6	foliage grain straw	80 167 167	<0.05 <0.05 <0.05	88-AC-0215 CGD 35-86R
Germany, 1986	LS	0.6	foliage grain straw	72 144 144	<0.05 <0.05 <0.05	88-AC-0215 CGD 36-86R
Germany, 1987	LS	0.6	foliage grain straw	85 157 157	<0.05 <0.05 <0.05	88-AC-0215 CGD 36-87R
Germany, 1987	LS	0.6	foliage grain straw	84 176 176	<0.05 <0.05 <0.05	88-AC-0215 CGD 37-87R
Germany, 1987	LS	0.6	foliage grain straw	86 157 157	<0.05 <0.05 <0.05	88-AC-0215 CGD 38-87R
Germany, 1994	FS	0.6	grain straw	125 125	≤0.05 ≤0.1	R1/94 RPA 21083 RP 94-593
Germany, 1994	FS	0.6	grain straw	114 114	≤0.05 ≤0.1	R1/94 RPA 21084 RP 94-593
Germany, 1994	FS	0.6	grain straw	143 143	≤0.05 ≤0.1	R1/94 RPA 21085
Germany, 1994	FS	0.6	grain straw	105 105	≤0.05 0.1	R1/94 RPA 21086
Germany, 1995	FS	0.6	grain straw	122 122	≤0.05 ≤0.1	R2/95 RPA 21087 RP 95-682
Germany, 1995	FS	0.6	grain straw	150 150	≤0.05 ≤0.1	R2/95 RPA 21088

Crop, Country, Year	Form	Application, g ai/kg seed	Sample	PHI, days	Residues, mg/kg	Report
						RP 95-682
UK, 1972	LS	0.8	grain	150	<0.1	RT/23/72
UK, 1972	WP	0.8	grain	150	<0.1	RT/28/72
Wheat, Winter						
France, 1995	LS	0.8	shoot grain straw	20 27 39 88 159 265 265	0.14 <0.1 <0.1 <0.1 <0.1 ≤0.05 ≤0.1	RD/CRLD/ AN/9615860 95506OR1
France, 1995	LS	0.8	shoot grain straw	109 144 215 215	<0.1 <0.1 ≤0.05 ≤0.1	RD/CRLD/ AN/9615860 95506AV1
Germany, 1986	LS	0.6	foliage grain straw	212 286 286	<0.05 <0.05 <0.05	87-AC-0172 Rs 8532 E1
Italy, 1994	FS	0.6	grain straw	213 213	≤0.05 ≤0.1	RD/CRLD/ AN/9516718
Italy, 1994	FS	0.6	grain straw	245 245	≤0.05 ≤0.1	RD/CRLD/ AN/9516716
Italy, 1995	FS	0.6	grain	219	≤0.05	RD/CRLD/ AN/9615957 95610BO1
Italy, 1995	FS	0.6	grain	219	≤0.05	RD/CRLD/ AN/9615957 95610BO2
Italy, 1995	FS	0.6	grain straw	219 219	≤0.05 ≤0.1	RD/CRLD/ AN/9615958 95609BO1

Crop, Country, Year	Form	Application, g ai/kg seed	Sample	PHI, days	Residues, mg/kg	Report
Italy, 1995	FS	0.6	grain straw	219 219	<0.05 <0.1	RD/CRLD/ AN/9615958 95609BO2
Italy, 1995	FS	0.6	grain straw	219 219	<0.05 <0.1	RD/CRLD/ AN/9615959 95611BO1
Italy, 1995	FS	0.6	grain straw	219 219	<0.05 <0.1	RD/CRLD/ AN/9615959 95611BO2
Italy, 1995	FS	0.6	grain straw	219 219	<0.05 <0.1	RD/CRLD/ AN/9615992 95611BO1
Italy, 1995	FS	0.6	grain straw	219 219	<0.05 <0.1	RD/CRLD/ AN/9615992 95611BO2
Rye, spring						
Germany, 1985	LS	0.6	foliage grain straw	46 135 135	<0.05 <0.05 <0.05	86-AC-0322 Rs 8501 B2
Rye, winter						
Germany, 1986	LS	0.6	foliage grain straw	214 295 295	<0.05 <0.05 <0.05	87-AC-0172 Rs 8532 B1
Germany, 1986	LS	0.6	foliage grain straw	219 304 304	<0.05 <0.05 <0.05	88-AC-0215 CGD 04-86R
Germany, 1987	LS	0.6	foliage grain straw	230 334 334	<0.05 <0.05 <0.05	88-AC-0215 CGD 10-87R

[1]No report available, only summary table

Sugar cane (Table 16). The data are from a summary table (Anon., 1976) because the full study report was not available to the manufacturer. The use pattern (treatment of cane segments before planting) is such that no residues would be expected at harvest.

Table 16. Residues of guazatine in sugar cane products from treatments before planting. USA, Hawaii, 1976. (Only summary available).

Form	Application No	Application kg ai/hl	Sample	Residues, mg/kg	Report
LS	1	0.025 (cold solution)	cane bagasse molasses raw sugar	<0.1 <0.1 <0.1 <0.1	1098-55
LS	1	0.01 (hot solution)	cane bagasse molasses raw sugar	<0.1 <0.1 <0.1 <0.1	1098-55

Citrus fruits (Table 17). Trials on oranges (10), mandarins (1), lemons (3) and grapefruit (2) were carried out in Australia (Thornberg, 1980a), Israel (Bodin, 1978), Italy (Thornberg, 1980b) and the USA (Karlsson and Risholm-Sundman, 1988). In some cases the pulp and peel were weighed and analysed separately and the residue in the whole fruit calculated. The results are shown in Table 17.

In two of the trials Bodin (1978) used ^{14}C- and ^{3}H-labelled guazatine acetate to determine the penetration of the active ingredient into treated oranges after storage up to 50 days at +4°C. In two of four trials ethylene was used for degreening; this treatment had no significant influence on the residual content of guazatine.

Table 17. Residues of guazatine in citrus fruits, post-harvest treatment. All single applications of SL formulation.

Fruit, Country, year	Application		Sample	Residues, mg/kg	Storage, days	Report
	kg, ai/hl	Method				
Oranges						
Australia, 1979	0.05	flooding	whole fruit peel pulp	0.3 3 <0.05	7 7 7	80AC0184
Israel, 1978[1]	0.2	dipping	Whole fruit peel white pulp	5.5[2] 17 1.3 0.1	50 50 50	78-08-01
Israel, 1978[1]	0.1	Dipping	Whole fruit peel white pulp	1.8[2] 6.3 0.4 0.03	50 50 50	78-08-01
Israel, 1978[1]	0.2	dipping, degreening with ethylene[3]	Whole fruit peel white pulp	5.5[2] 15 0.7 0.1	50 50 50	78-08-01
Israel, 1978[1]	0.1	dipping, degreening with ethylene[3]	Whole fruit peel white pulp	1.8[2] 4.8 0.1 0.03	50 50 50	78-08-01
Israel, 1988	0.2 (in wax)	dipping	Whole fruit peel pulp	1.8[2] 7[4] <0.05[4]		88AC0343 48526-4-88
Italy, 1978	0.1 (in water)	dipping	Whole fruit peel pulp	0.6 (2), 0.5 4, 0.4, 2 <0.05	2 5 2 5 2	80AC0187 1065-78

Fruit, Country, year	Application		Sample	Residues, mg/kg	Storage, days	Report
	kg, ai/hl	Method				
Italy, 1978	0.2 (in wax)	dipping	whole fruit	0.5 0.6 0.5 0.3 0.2	0 2 5 10 20	80AC0187 1065-78
USA, 1983	0.1 (in water)	spraying	whole fruit peel pulp	0.2[2] 0.7 <0.05		84AC0232
USA, 1983	0.2 (in wax)		whole fruit peel pulp	0.7[2] 3 <0.05		84AC0232[5]
Mandarins						
Australia, 1979	0.05	flooding	whole fruit peel pulp	0.5 2 <0.05	7 7 7	80AC0184
Lemons						
Australia, 1979	0.05	flooding	whole fruit peel pulp	<0.2 2 <0.05	7 7 7	80AC0184
USA	0.1 (in water)	spraying	whole fruit peel pulp	0.45[2] 0.8 0.05		84AC0232[5]
USA	0.2 (in wax)	spraying	whole fruit peel pulp	0.8[2] 1.3 0.13		84AC0232[5]
Grapefruit						
USA, 1983	0.1 (in water)	spraying	whole fruit peel pulp	0.08[2] 0.13 <0.05		84AC0232[5]

Fruit, Country, year	Application		Sample	Residues, mg/kg	Storage, days	Report
	kg, ai/hl	Method				
USA, 1983	0.2 (in wax)	spraying	Whole fruit Peel Pulp	0.33[2] 0.83 0.09		84AC0232[5]

[1]Treatment with ^{3}H- and ^{14}C-labelled guazatine, residues calculated as guazatine equivalents
[2]Residues in whole fruit calculated from residues in pulp and peel
[3]Fruits were drenched with guazatine solution, dried and degreened with gaseous ethylene (10-20 ppm for 24 h).
[4]Mean of 10 fruits
[5]Only summary report available

FATE OF RESIDUES IN STORAGE AND PROCESSING

In storage

In a study of the stability of guazatine residues during storage Bodin (1978) drenched oranges with 0.1 or 0.2 kg ai/hl (1000 or 2000 ppm) of ^{14}C- and ^{3}H-labelled guazatine acetate in water at 12-18°C for 30-60 seconds. Half of the oranges were degreened with ethylene. After drying and waxing, the fruits were stored at 4°C in plastic bags in a refrigerator. Samples were taken after 0, 1, 8, 24 and 50 days. The oranges were separated into yellow peel, white, and pulp, which were pulverized and frozen at -20°C. The results are shown in Table 18. Residue concentrations were reported as mg/kg dry weight. Fruits stored for 50 days at 4°C were reported as being "storage-damaged".

Table 18. Storage stability of guazatine in oranges (Bodin, 1978).

Storage, days	^{14}C or ^{3}H, mg/kg dry wt. as guazatine acetate							
	0.1 kg ai/hl				0.2 kg ai/hl			
	^{14}C deg	^{14}C not deg	^{3}H deg	^{3}H not deg	^{14}C deg	^{14}C not deg	^{3}H deg	^{3}H not deg
Peel								
0	22	13	19	16	53	53	55	51
1	19	41	17	39	68	84	76	93
8	31	36	27	27	60	95	61	96
24	21	28	18	29	83	63	100	69
50	22	25	18	24	54	56	56	74
White								
0	0.41	2.4	0.59	4.4	1.8	1.2	1.7	2.4
1	0.70	0.99	0.69	1.1	1.4	3.5	1.7	4.0
8	0.47	0.96	0.46	1.3	2.6	2.7	3.9	2.9
24	1.2	1.6	0.88	1.4	4.9	5.1	9.9	4.5
50	0.89	3.5	0.77	4.0	4.0	17	4.4	23
Pulp								
0	0.52	0.36	0.25	0.19	0.68	1.2	0.39	1.1
1	0.51	0.02	0.31	0.30	0.94	0.39	5.1	0.27
8	0.21	0.36	0.07	0.27	0.55	0.32	0.39	0.35
24	0.31	0.26	0.15	0.11	0.90	0.78	0.71	0.67
50	0.67	0.77	0.48	0.94	4.3	4.1	5.5	4.5

The stability of guazatine in Zivdar wax treated at 0.1 and 0.2 kg ai/hl and stored in closed vessels at 35°C for three months was studied by Karlsson and Risholm-Sundman (1988). The guazatine concentrations in the wax were 1025 and 1792 mg/kg before storage and 1126 and 2056 mg/kg after three months, the increase in concentration being due to water evaporation. No decomposition of guazatine could be detected.

In processing

A processing study on grapefruit and oranges was reported by Stensiö and Thorstensson (1987). The results are shown in Table 19. The "peel" of the whole fruit is defined as that obtained by hand peeling. "Wet peel" is from machine-peeled fruit where a water spray is constantly used in the peeler. "Dried peel" is produced by the addition of lime, $Ca(OH)_2$, to the wet peel fraction followed by drying in a tunnel dryer.

Table 19. Residues of guazatine acetate in processed fractions of citrus fruit (Stensiö and Thorstensson, 1987), USA.

Fruit	Application, kg ai/hl	Sample	Residue, mg/kg	Processing factor
Grapefruit	0.1	whole fruit peel pulp	0.08[1] 0.13 <0.05	 1.6 <0.6
		peel oil emulsion	0.18	2.3
		press liquor	<0.06	<0.75
		cold press grapefruit oil	<0.05	<0.6
		juice	<0.05	<0.06
		dried peel	1.2	15
		finisher pulp	<0.05	<0.6
		molasses	1.4	18
Grapefruit	0.2	whole fruit peel pulp	0.33[1] 0.83 0.09	 2.5 0.3
		peel oil emulsion	0.59	1.8
		press liquor	0.39	1.2
		cold press orange oil	<0.05	<0.15
		juice	<0.05	<0.15
		dried peel	2.1	6.4
		finisher pulp	<0.05	<0.15
		molasses	1.2	3.6
Oranges	0.1	whole fruit peel pulp	0.2[1] 0.7 <0.05	 3.5 <0.25
		peel oil emulsion	0.1	0.5
		press liquor	0.25	1.3
		cold press orange oil	<0.05	<0.25
		juice	<0.05	<0.25
		wet peel	0.4	2
		dried peel	max. 2.6	13
		finisher pulp	<0.05	<0.25

Fruit	Application, kg ai/hl	Sample	Residue, mg/kg	Processing factor
		molasses	0.33	1.7
Oranges	0.2	whole fruit peel pulp	0.7[1] 3 <0.05	 4.3 <0.07
		peel oil emulsion	0.49	0.7
		press liquor	0.69	1
		cold press orange oil	<0.05	<0.07
		juice	0.09	0.13
		wet peel	0.83	1.2
		dried peel	max. 3.4	4.9
		finisher pulp	0.1	0.14
		molasses	0.7	1

[1]Residues in whole fruit calculated from residues in pulp and peel

Processing factors for the peel and pulp fractions of the fruit can be calculated from the results of the supervised trials on citrus fruits in Table 17 as shown in Table 20.

Table 20. Residues of guazatine in peel and pulp fractions of citrus fruits.

Fruit	Country	Application, kg ai/hl	Sample	Residue, mg/kg	Ratio to whole fruit
Oranges	Israel	0.2	whole fruit peel white pulp	5.5 17 1.3 0.1	 3.1 0.24 0.02
Oranges	Israel	0.1	whole fruit peel white pulp	1.8 6.3 0.4 0.03	 3.5 0.2 0.02
Oranges	Israel	0.2	whole fruit peel white pulp	5.5 15 0.7 0.1	 2.7 0.13 0.02
Oranges	Israel	0.1	whole fruit peel white pulp	1.8 4.8 0.1 0.03	 2.7 0.06 0.02
Oranges	Isreal	0.2	whole fruit peel	1.8 7	 3.9

Fruit	Country	Application, kg ai/hl	Sample	Residue, mg/kg	Ratio to whole fruit
			pulp	<0.05	<0.03
Oranges	Italy	0.1	whole fruit peel pulp	0.6 4 <0.05	 6.7 <0.08
Oranges	Italy	0.1	whole fruit peel pulp	0.5 2 <0.05	 4 <0.1
Oranges	Australia	0.05	whole fruit peel pulp	0.3 3 <0.05	 10 <0.17
Mandarins	Australia	0.05	whole fruit peel pulp	0.5 2 <0.05	 4 <0.1
Lemons	Australia	0.05	whole fruit peel pulp	<0.2 2 <0.05	
Lemons	USA	0.1	whole fruit peel pulp	0.45 0.8 0.05	 1.8 0.1
Lemons	USA	0.2	whole fruit peel pulp	0.8 1.3 0.13	 1.6 0.16

Residues in the edible portion of food commodities

The data on citrus fruits (Tables 17, 19 and 20) show that almost all the residues were found in the peel with a maximum level of 17 mg/kg. The pulp and juice never contained more than 0.13 mg/kg guazatine.

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

No data were received.

NATIONAL MAXIMUM RESIDUE LIMITS

The following national MRLs were reported. The residues are defined as guazatine.

Country	Commodity	MRL, mg/kg

Country	Commodity	MRL, mg/kg
Australia	Citrus fruits	5
	Melons, except watermelon	5
	Tomato	5
Austria	Citrus fruits	5
Belgium	Fruits	0.1
	Vegetables	0.1
	Potato	0.1
Finland	Citrus fruits	5
Germany	Citrus fruits	5
	Citrus juice	0.5
	Melons	5
	Oil seeds	0.5
	Cereals	0.2
	Other commodities of plant origin	0.05
Italy	Cereal grains	0.1
The Netherlands	Fruits, except citrus	0.1
	Citrus fruits	5
	Melons	5
	Cereal grains	0.1
New Zealand	Citrus fruits	5
Norway	Citrus fruits	5
Spain	Fruits, except citrus	0.1
	Citrus fruits	5
	Melons	5
	Potato	5
Sweden	Citrus fruits	5
	Melons	5
Switzerland	Cereal grains	0.05

APPRAISAL

Guazatine was evaluated by the JMPR in 1978 and 1980, and is now re-evaluated in the CCPR periodic review programme. It is a non-systemic contact fungicide which disturbs the membrane function of fungi. It controls a wide range of seed-borne diseases of cereals, e.g. seedling blight *(fusarium spp.)*, glume blotch *(septoria)*, common bunt *(tilletia spp.)*, common root rot *(helminthosporium)* and smut *(ustilago)*. On citrus fruit, guazatine is used as a bulk dip after harvest, in the packing line as a spray and in washing installations to disinfect the process water. It controls sour rot *(geotrichum candidum)*, green mould *(penicillium digitatum)* and blue mould *(penicillium italicum).*

Guazatine is a mixture of reaction products from polyamines, comprising mainly octa-methylenediamine, iminodi(octamethylene)diamine, octamethylenebis(imino-octamethylene)diamine, and carbamonitrile. A coding system is used for the compounds that make up guazatine in which "N" represents any amino group. Thus NN stands for $H_2N-(CH_2)_8-NH_2$, NNN stands for $H_2N-(CH_2)_8-NH-(CH_2)_8-NH_2$ and so on. "G" stands for any amino group (NH or NH_2) of the above which is guanidated. For example GG stands for $H_2N-C(NH)NH-(CH_2)_8-NH-C(NH)-NH_2$.

The fate of residues has been studied in animals, plants and soil.

Studies on rats and lactating cows showed poor absorption from the gastrointestinal tract, rapid elimination mainly in the faeces (>90%), excretion largely as the unchanged parent mixture and no accumulation in any organs, tissues or milk.

When cows were dosed daily with 0.5 mg/kg bw for 10.5 days, 93% of the administered radioactivity was recovered in the faeces as unchanged guazatine, and the low levels in plasma indicated minimal absorption. ^{14}C in the milk and plasma, expressed as guazatine, reached plateau levels of about 0.02 and 0.015 mg/l respectively by day 3 in milk and day 6 in plasma. Following slaughter after the last dose residues of about 0.08 mg/kg were found in the liver and kidney with only very low levels in other edible tissues (<0.02 mg/kg in skeletal muscle and fat).

Adequate metabolism studies with full characterization of the metabolites in farm animals, an animal transfer study on ruminants and an analytical method for commodities of animal origin were not submitted. The Meeting was therefore unable to establish a definition of the residue of guazatine in animal products and could not estimate maximum residue levels for products of animal origin.

When wheat seeds were dressed with [^{14}C]guazatine at 1.05 g ai/kg seed there was no difference between the total radioactive residue (TRR) levels in the harvested grain, straw or chaff from the treated and the control plots. The method of application was according to GAP.

The foliar application of [^{14}C]guazatine to wheat at 1.1 kg ai/ha, 11 weeks before harvest, resulted in mean TRRs of 29 mg/kg guazatine equivalents in the straw, 18 mg/kg in the chaff, and 0.8 mg/kg in the grain.

When [^{14}C]guazatine was applied to the leaf surface or the fruit of apples (brushing with 0.05 or 0.1 kg ai/hl) its translocation was extremely limited. Autoradiography showed no observable movement in the leaves or fruit and this was confirmed by quantitative determination of the TRR: 87% of the applied radioactivity was recovered from the leaves after 12 weeks, 66% from the surface and 21% from the leaf tissues (61% was identified as the parent mixture). In the fruit 62% of the TRR was located on the surface and 38% in the tissues after 12 weeks, with 81% of the TRR identified as the parent. The remainder comprised a major photodegradation product (4.5%), other extractable compounds (9.7%), and unextractable residues (5.2%).

The uptake of guazatine residues from soil by soya beans and rice plants was investigated by treating soils with 5 mg/kg of [^{14}C]guazatine and planting soya bean and rice plants after 26 weeks.

Four weeks after planting, the TRR in soya beans amounted to only 0.08% of the applied radioactivity in the aerial part and 0.12% in the whole plant. The residues expressed as guazatine equivalents on a dry weight basis were 2.8 mg/kg in the aerial part and 3.7 mg/kg in the whole plant. The pods contained 0.052 mg/kg on a dry weight basis 9 weeks after planting.

Guazatine residues taken up from flooded soil were low in the whole rice plant, which absorbed only 0.13% of the applied ^{14}C (0.57 mg/kg on a dry weight basis) during a period of four weeks, with 0.05 % of the applied radioactivity or 0.23 mg/kg (dry weight) in the shoot.

Guazatine has been shown to be metabolized in about 100 days when applied to wheat seeds planted in soil, via deguanidation and subsequent mineralization. The test system had a substantial influence on the degradation time.

When guazatine was applied to wheat seeds which were subsequently planted in soil and the soil leached to simulate rainfall, the guazatine components were found to be associated with the seeds or the soil surrounding the seeds. The compounds that had moved from the seeds to the soil showed no tendency to migrate. Significant mineralization to carbon dioxide occurred during the leaching period.

The Meeting concluded that these studies were adequate for the use of guazatine for the seed treatment of cereals, and that no further studies on rotational crops were necessary for such uses.

The use of such a complex mixture as guazatine presents a problem in choosing a residue analytical method. It is not considered practical to attempt the determination of all the components so some alternative is necessary. Two approaches may be applicable.

1. Development of a "total residue" method by conversion to a single compound.

2. The choice of a major component as a "marker", with the inclusion of a correction factor to give the total residue.

Many of the residue studies used the first approach, involving the hydrolysis of residues to bis(8-amino-octyl)amine (NNN) and its determination either directly or after derivatization. This method was used, e.g., for the analysis of citrus fruits, where the LODs (expressed as guazatine) were 0.05 mg/kg for finisher pulp, 0.2 mg/kg for wet peel and 1 mg/kg for dried peel. The metabolites are determined by the total residue method together with the parent material.

Better results were achieved with cereals, however, by using the marker GG (octane-1,8-diyldiguanidine, H_2N-C(NH)NH-$(CH_2)_8$-NH-C(NH)-NH_2), one of the major guazatine components, for quantification. This method incorporates a correction factor to allow for the fact that GG represents only 30% of the total guazatine. The homologue GG-C6 (1,6-diguanidinohexane, H_2N-C(NH)NH-$(CH_2)_6$-NH-C(NH)-NH_2), is used as an internal standard. The analytical method for grain and straw consists in extraction of samples fortified with the internal standard with hot 1M HCL, clean-up on a cation exchange column, derivatization with hexafluoroacetylacetone (HFAA), clean-up on basic Al_2O_3, and determination of the HFAA derivatives of GG and the internal standard GG-C_6 by GC-MS.

Samples fortified with guazatine showed LODs of 0.05 mg/kg for cereal grains and 0.1 mg/kg for straw with recoveries of 88% and 94% respectively. The lowest fortification levels at the LOD of the marker GG were also 0.05 mg/kg for grain and 0.1 mg/kg for straw (recoveries: grain 97%, straw 82%).

The Meeting concluded that 0.05 mg/kg is a practical limit of determination for GG.

The justification for the choice of GG as representative of the total guazatine residues in cereals has been supported by the following facts.

1. Guazatine shows low uptake and translocation in cereals. This is consistent with the lack of detectable residues reported in crops after seed treatments.

2. Where the material has been applied as a foliar spray on dwarf apples trees there is little evidence of significant metabolism or hence of changes in the proportions of the components of the guazatine mixture.

3. In a situation where metabolism is demonstrably occurring (see below), GG remains a significant component after 29 days.

Evidence for GG still being present under "metabolizing" conditions comes from an aerobic soil degradation study. In this, a mixture of GG, GN and GGG was applied to seed surfaces, and the seeds were planted in soil in metabolism vessels. Most of the seeds germinated. It was possible to distinguish the seeds from the soil and extract the seeds separately up to 29 days after planting. Analysis of these extracts indicated a change in the profile of components present on the seed with GGG levels decreasing. This is consistent with the generation of $^{14}CO_2$ in the study. However at day 29 GG was still the predominant single compound on the seed, despite the degradation which had been occurring at the seed surface or in the soil in contact with it.

On this basis, it is considered that GG represents a satisfactory marker compound to represent guazatine residues in seed-treated cereals.

The storage stability of analytical samples was investigated by storing analysed samples of wheat grain, ears and straw at -20EC and re-analysing them after two years. The study was not satisfactory as an unvalidated analytical method was used.

Definition of the residue. The metabolism of guazatine in animals has not been fully elucidated, and the Meeting concluded that the residue of guazatine in products of animal origin could not be satisfactorily defined.

The metabolism of guazatine in plants has also not been fully characterized. The main uses of guazatine are for the seed treatment of cereals and the post-harvest protection of citrus fruits. The Meeting concluded that the available studies were adequate only for the seed treatment of cereals. Should further uses (e.g. foliar spray or treatment of plants other than cereals) be planned in future, detailed metabolism studies would be required.

Guazatine has been determined by a total residue method based on conversion to the corresponding triamine, bis(8-amino-octyl)amine, which also occurs as a metabolite. Modern analytical methods using octane-1,8-diyldiguanidine (GG), one of the main components of guazatine, as a marker are more specific.

The Meeting concluded that the residue should be defined for enforcement purposes as "octane-1,8-diyldiguanidine" (GG). Assuming that the content of GG is 30% of the total guazatine content, the GG content should be multiplied by 3 for risk assessment purposes for commodities of plant origin.

Definition of the residue for enforcement purposes: octane-1,8-diyldiguanidine (GG), expressed as octane-1,8-diyldiguanidine.

Definition of the residue for risk assessment purposes: guazatine.

Supervised trials

Citrus fruits. Concentrations of 0.05 to 0.2 kg ai/hl water or 0.3 kg ai/hl wax are registered for post-harvest treatment.

In Australia, guazatine is registered for the post-harvest treatment of citrus fruits with 0.052 kg ai/hl. Three residue trials according to GAP (one each on oranges, mandarins and lemons) were reported and showed residues of <0.2, 0.3 and 0.5 mg/kg (calculated as guazatine) in the whole fruit.

South African GAP specifies 0.3 kg ai/hl in wax for the treatment of citrus fruits. Five trials (3 on oranges, one each on lemons and grapefruit) at the lower rate of 0.2 kg ai/hl in wax were reported. The residues in the whole fruit ranged from 0.33 to 1.8 mg/kg, calculated as guazatine. These results and the data on the validation of the method were submitted only as summaries.

After dipping oranges in water with 0.2 kg ai/hl guazatine, residues of 5.5 mg/kg were calculated in the whole fruit (2 trials). These results are inconsistent with the results found after waxing and indicate a more critical residue situation. Furthermore, no data were available on residues in small citrus fruits (e.g. mandarins) after treatment with 0.2 kg ai/hl.

The Meeting concluded that the residue data were not adequate for citrus fruits as a major crop and recommended the withdrawal of the existing CXL of 5 mg/kg.

Tomatoes and melons, except watermelons. Post-harvest uses of guazatine exist in Australia but no residue data were received.

No maximum residue level could be estimated for tomatoes, and the Meeting recommended the withdrawal of the existing CXL of 5 mg/kg for melons, except watermelon.

Pineapples and potatoes. Since no residue data or information on GAP were received, the Meeting recommended the withdrawal of the existing CXLs of 0.1* mg/kg for pineapple and potato.

Cereal grains. The use of guazatine for seed treatment is registered in many countries with application rates from 0.05 to 1.05 g ai/kg seed (mainly 0.45-0.6 g ai/kg). A total of 84 supervised trials with treatments at 0.4, 0.6, 0.8, 0.9, 1, 1.2 or 1.5 g ai/kg seed were reported to the Meeting. The samples from 61 trials carried out from 1972 to 1987 were analysed by an unvalidated analytical method and could not be used for evaluation. Valid results from 23 trials carried out in 1994/95 on wheat in France (7), Germany (6) and Italy (10) were submitted. No residues were found above the LOD of 0.05 mg/kg, calculated as guazatine.

In view of the non-systemic character and particular use pattern of guazatine as a seed treatment, the Meeting concluded that the residue in cereal grains was "essentially zero" and estimated an STMR of 0 mg/kg.

The Meeting estimated a maximum residue level of 0.05* mg/kg expressed as GG for cereal grains as a practical limit of determination.

Sugar cane. Guazatine is registered in South Africa for the treatment of plant segments before planting with a solution of 0.08 kg ai/hl water. Only two trials, not complying with GAP, were reported. Sugar cane was treated in Hawaii with solutions of 0.01 or 0.025 kg ai/hl. Residues in cane, bagasse, molasses and raw sugar were reported as <0.1 mg/kg. The report was submitted only as a summary with little information (e.g. the PHI and analytical method were not stated).

The Meeting recommended the withdrawal of the existing CXL (0.1 mg/kg).

Rape seed. The use of guazatine as a foliar spray is registered in Germany but no residue data were received. No maximum residue level could be estimated.

Straw and fodder of cereal grains. After treatment of wheat with 0.6-0.8 kg ai/kg seed the residues found in 21 trials carried out in 1994/95 in France (7), Germany (6) and Italy (8) were all <0.1 mg/kg calculated as guazatine.

As there was no residue definition for guazatine in animal products, the Meeting did not recommend an MRL for the straw and fodder of cereal grains as a feed item.

Animal products. No transfer study was carried out on ruminants, no definition of the residue in products of animal origin could be proposed, and no maximum residue levels were estimated for any animal feed items.

The Meeting concluded that there was insufficient information to estimate maximum residue levels for guazatine in products of animal origin.

No feeding or metabolism studies were reported for laying hens. As no residues occur in cereal grains after seed treatment, the Meeting concluded that further studies and the estimation of maximum residue levels for residues in poultry commodities resulting from seed treatment were not necessary.

A study of the storage stability of radiolabelled guazatine on oranges after drenching with 0.1 or 0.2 kg ai/hl showed no decrease of the residues after 50 days.

The results of commercial processing studies on citrus fruits indicate that the residues are on the peel surface. Processing factors calculated for dried peel were 4.9, 6.4, 13 and 15, mean 9.8, median 9.7, and for molasses 1, 1.7, 3.6 and 18, mean 6.1, median 2.7. There was a clear reduction of the residue during processing to pulp and juice. The analysis of fresh peel in 15 supervised trials showed ratios of the residues in the peel to those in the whole fruit ("processing factors") of 1.6 (2), 1.8, 2.5, 2.7 (2), 3.1, 3.5 (2), 3.9, 4 (2), 4.3, 6.7 and 10. with a mean of 3.7 and a median of 3.5.

Residues in the edible portions of citrus fruits were low. After treatment according to GAP, most pulp and juice samples contained guazatine residues at or about the LOD (#0.05 mg/kg) and never more than 0.13 mg/kg.

No information was provided on residues in commodities in commerce or at consumption.

The Meeting estimated the maximum residue level shown in Annex I (Part 2). As the Meeting withdrew the ADI for guazatine this is recorded only as a Guideline Level.

FURTHER WORK OR INFORMATION

<u>Desirable</u>

Any further evaluations for uses apart from the seed treatment of cereals would require the following data.

1. Clarification of the metabolism of all major components in ruminants.

2. Animal transfer studies on ruminants including an analytical method for the determination of residues in products of animal origin.

3. Clarification of the metabolism of all major components in plants.

RECOMMENDATIONS

The Meeting estimated the maximum residue level shown below. As the Meeting withdrew the ADI for guazatine this is recorded only as a Guideline Level. Other previous estimates were withdrawn.

Pesticide (Codex ref. no.)	Commodity	GL, mg/kg		Previous MRL, mg/kg	STMR, mg/kg
	CCN	Name			
Guazatine	FC 0001	Citrus fruits	W	5 Po	
(114)	FI 0353	Pineapple	W	0.1*	
	GC 0080	Cereal grains	0.05*	0.1*	0
	GS 0659	Sugar cane	W	0.1*	
	VC 0046	Melons (except Watermelon)	W	5 Po	

	VR 0589	Potato	W	0.1*	
	Residue for GLs: octane-1,8-diyldiguanidine ("GG"), expressed as octane-1,8-diyldiguanidine for STMRs: guazatine				

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METHAMIDOPHOS (100)

EXPLANATION

Methamidophos was first evaluated in 1976 with further reviews of residue aspects in 1979, 1981, 1984, 1989, 1990, 1994, and 1996. The 1994 JMPR recommended an MRL of 0.5 mg/kg for pome fruits, based on a 21-day PHI.

At the 28th (1996) Session of the CCPR the delegations of The Netherlands, the UK and the EU expressed concern about the intake. At its 29th Session the CCPR retained the MRL for pome fruits at Step 7 pending the evaluation of new data on residues (ALINORM 97/24A, para 61).

Data on supervised trials and information on GAP were submitted to the Meeting to support the estimation of a maximum residue level for pome fruits.

METHODS OF RESIDUES ANALYSIS

Analytical methods

The analytical methods used in supervised trials were the same as those reported in earlier evaluations. Methamidophos can be extracted with ethyl acetate, acetone/water, or acetonitrile. The extract is cleaned up by silica gel or gel permeation chromatography. The residues are quantified by GLC with NP or FP detection.

The analytical method used in most trials submitted to the present Meeting was described by Specht and Thier (1992) and modified by Thier and Kirchhoff (1992). Extraction is with ethyl acetate in the presence of anhydrous granulated sodium sulfate and the final extract is analysed by GLC with an NP detector. The limit of determination (LOD) for methamidophos was 0.01 mg/kg and recoveries were >80%.

Stability of pesticide residues in stored analytical samples

Studies of the stability of residues in frozen samples of several commodities (Lai, 1988a,b) were evaluated by the 1996 JMPR, but the stability of methamidophos was not established because most of the samples were of crops which had been treated with acephate and contained substantially higher levels of acephate than of methamidophos, and acephate could well be degraded to methamidophos during storage. The studies did not include apples and no studies of the stability methamidophos on apples have been submitted to the present Meeting.

USE PATTERN

Methamidophos is an organophosphorus insecticide registered for use on many commodities to control a broad spectrum of insects. Information on registered uses of methamidophos on pome fruits in France, Greece and Italy was provided to the Meeting. Since residues of methamidophos can arise from the use of acephate, the uses of both pesticides have to be taken into account.

Table 1. Registered uses of methamidophos on pome fruits.

Crop	Country	Form.	Application			PHI, days
			kg ai/ha	kg ai/hl	No.	
Apples and pears	France	SL	0.5	0.05	1-2	21
Apples and pears	Greece	SL	0.9-1.2	0.045-0.06	1-2	21
Pome fruit	Italy	EC	0.5-0.75	0.025-0.0375	1	21
Pome fruit	Morocco	SL	0.9			28
Pome fruit	Spain	SL	0.68			28

Table 2. Registered uses of acephate on pome fruits.

Crop	Country	Form.	Application			PHI, days
			kg ai/ha	kg ai/hl	No.	
Pome fruit	Germany	50 WP	0.75		3	42
Pome fruit	South Africa			0.05	1-2	

RESIDUES RESULTING FROM SUPERVISED TRIALS

Residues arising from the use of methamidophos

Pome fruits. Three supervised trials on apples and two on pears in France in 1992 were evaluated in 1994 and reviewed again by the present Meeting. Methamidophos SL was applied twice with a 21-day interval to apples on plots of 32-96 m^2. Samples were analysed after gel permeation clean-up by GLC with FP detection. The SAIs were 210-245 days. Residues 21 days after the second application were between 0.01 and 0.1 mg/kg and similar to those immediately before it. The data lacked both control and sample chromatograms.

Three trials at different locations in Italy on different varieties of apples were evaluated in 1994. Methamidophos was sprayed twice on to plots of 22.5 m^2 (2 trees) to 48 m^2 (4 trees), with a 15-day interval. The SAIs were 180-210 days. The residues were 0.02-0.33 mg/kg at a 21-day PHI.

Field trials on apples were carried out in 1995 and 1996 at six test sites in Greece and Spain. In Greece methamidophos 600 g/l was applied according to Greek GAP, with 13-16 days between applications, using a motorised knapsack sprayer. The plot sizes were 3-8 trees. The residues of methamidophos were 0.31-0.49 mg/kg. Residues in untreated samples were always below the LOD. The SAIs were 16-88 days.

In Spain Methamidophos SL 600 g/l was applied twice with a hand-gun sprayer to three different varieties of apples at intervals of 13-16 days. The SAIs were 38-83 days. Residues of methamidophos at 21-day PHI were between 0.08 and 0.24 mg/kg.

Summary data from two trials on apples in 1970 in Israel were submitted to the Meeting. The residues of methamidophos at 19 days PHI were 0.14 and 0.23 mg/kg.

Two field trials on pears were conducted in France in 1992 according to French GAP. Residues 21 days after the last application were 0.15 and 0.21 mg/kg, similar to those in apples.

The results of the trials are shown in Table 3.

Table 3. Residues of methamidophos in pome fruit. All SL formulations. The underlined residues are from treatments according to GAP and were used to estimate maximum residue levels.

Crop, Country, Year	Application			PHI, days	Residues, mg/kg	Ref
	No.	kg ai/ha	kg ai/hl			
APPLES						
France 1992 Montfavert	2	0.675-0.688	0.05		0.04[1]	Bayer 1992a
				0	0.24	
				7	0.08	
				21	0.1	
				28	0.05	
France 1992 Montfavert	2	0.49	0.05		<0.01[1]	
				0	0.09	
				7	0.04	
				14	0.02	
				21	0.01	
				28	0.01	
France 1992, Pers les Fortaines	2	0.442	0.05		0.04[1]	
				0	0.5	
				7	11	
				14	0.04	
				21	0.04	
				28	0.06	
Greece 1995	2	1.2	0.06	20	0.4	Tomen 1996
Greece Pipera 1996	2	1.2	0.06	21	0.49	
Greece 1996 Agras	2	1.2	0.06	0	1.53	
				3	0.42	
				6	0.38	
				13	0.24	
				21	0.31	
Israel 1970	1	1.416	0.0885	3	0.17	Bayer, 1970
				7	0.15	
				14	0.1	
				19	0.04	
				27	0.06	
Israel 1970	2	1.416	0.0885	6	0.25	
				11	0.22	
				19	0.23	
Italy, 1992 Ravenna	2	0.731	0.049		0.19[1]	Bayer 1992b
				0	0.66	
				7	0.24	
				14	0.39	
				21	0.29	
				28	0.18	
Italy, 1992 S. Rommaldo	2	0.731	0.049	0	1.3	
				7	0.59	
				14	0.64	
				21	0.33	
				28	0.17	
Italy, 1992 Fondi	2	0.731	0.049		0.03[1]	
				0	0.51	
				7	0.07	
				14	0.05	
				21	0.02	
				28	0.02	
Spain, 1995	2	1.2	0.08	23	0.24	Tomen 1995b
Spain, 1995	2	1.2	0.08	0	1.32	
				3	0.35	

Crop, Country, Year	Application			PHI, days	Residues, mg/kg	Ref
	No.	kg ai/ha	kg ai/hl			
				7	0.17	
				14	0.09	
				21	0.08	
Spain, 1995	2	1.2	0.08	0	0.37	
				3	0.11	
				7	0.17	
				14	0.06	
				21	0.14	
PEARS						
France 1992	2	0.545	0.05		0.13[1]	Bayer 1992c
				0	1.02	
				7	0.43	
				14	0.34	
				21	0.21	
				28	0.12	
France 1992	2	0.416-0.452	0.05		0.16[1]	
				0	0.55	
				7	0.36	
				14	0.3	
				21	0.15	
				28	0.11	

[1]Before last application

Residues arising from the use of acephate

One field trial in Greece in 1995 in which apple trees were treated with acephate was reported to the Meeting. Acephate (75% SP) was applied three time at 11- and 30-day intervals. Samples harvested at a 15-day PHI showed residues of 0.38 and 0.24 mg/kg acephate and 0.03 mg/kg methamidophos. Samples were stored frozen at -20ºC until analysis. Residues in control samples were below the LOD.

No data were available from earlier evaluations on residues of methamidophos resulting from the use of acephate on pome fruits.

Table 4. Residues of acephate and methamidophos in apples after applications of acephate in Greece, 1995 (Tomen 1995a).

Application				PHI, days	Residues, mg/kg	
Form	No.	kg ai/ha	kg ai/hl		Acephate	Methamidophos
SP, 750 g/kg	3	1.55-1.60	0.0075	15	0.38, 0.24	0.03, 0.03

FATE OF RESIDUES IN STORAGE AND PROCESSING

No information on the effects of processing pome fruit was provided.

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

No data were submitted to the Meeting. A market basket study conducted in Australia in 1992, in which pears were included, was reported at the 1994 Meeting.

NATIONAL MAXIMUM RESIDUE LIMITS

The following national MRLs for pome fruits were reported to the Meeting.

Country	Commodity	MRL, mg/kg
Argentina	Pome fruit	0.1
Denmark	Pome fruit	0.01* T
France	Apple	0.3
	Pear	0.3
Germany	Pome fruit	0.2
Italy	Pome fruit	0.15
Spain	Pome fruit	0.2
Sweden	Fruit	0.2

T: temporary

APPRAISAL

Methamidophos is a widely used organophosphorus insecticide with systemic properties; its residues may also occur as a metabolite of acephate. It was first evaluated in 1976 with further reviews of residue aspects in 1979, 1981, 1984, 1989, 1990, 1994 and 1996. The 1994 JMPR recommended an MRL of 0.5 mg/kg for pome fruit, based on a 21-day PHI. It was held at step 7B by the 29th (1997) Session of the CCPR. The manufacturer has submitted new residue data to support the estimation of a maximum residue level for pome fruit.

The analytical methods employed in supervised trials were based on GLC. Recoveries were >70% and the LOD in all the methods was 0.01 mg/kg.

Studies of the storage stability of residues in several commodities were included in the studies of the stability of acephate residues evaluated in 1996 but no studies of the stability of methamidophos on apples were submitted.

Trials conducted in France and Italy in 1992 on apples and pears were evaluated by the 1994 JMPR and again reviewed by the present Meeting. In three trials on apples according to French GAP (1-2 applications at 0.5 kg ai/ha, 21-day PHI) the residues were 0.01, 0.06 and 0.1 mg/kg. The residue of 0.06 mg/kg was at 28 days; the residue at day 21 was 0.04 mg/kg. Three Italian trials on apples carried out with two applications of methamidophos (Italian GAP allows one) at 0.049 kg ai/hl were evaluated against Greek GAP (1 or 2 applications at 0.045-0.06 kg ai/hl, 21-day PHI). The residues were 0.02, 0.29 and 0.33 mg/kg.

The residues in three trials on apples according to GAP in Greece in 1995/96 were 0.31, 0.4 and 0.49 mg/kg. In similar trials in Spain in 1995 the residues were somewhat lower: 0.08, 0.14 and 0.24 mg/kg at a 21-day PHI. As the application rates were higher than in Spanish GAP the results were evaluated against Greek GAP.

Several of these trials were designed to produce residue decline curves. They showed that when methamidophos was applied twice with an interval of 3 weeks most of the residues resulted from the second application.

Two trials on apples in Israel in 1970 gave residues of 0.04 and 0.23 mg/kg at a 19-day PHI. Since no relevant GAP was reported these results were not considered for the estimation of a maximum residue level.

In a trial in which apples were treated with acephate at an application rate of 1.55-1.6 kg ai/ha the residue of methamidophos at a 15-day PHI was 0.03 mg/kg.

The residues of methamidophos in apples in rank order from the 12 trials according to GAP were 0.01, 0.02, 0.06, 0.08, 0.1, 0.14, 0.24, 0.29, 0.31, 0.33, 0.4 and 0.49 mg/kg.

Pears treated with methamidophos in France in 1992 according to GAP showed residues of 0.15 and 0.21 mg/kg after 21 days.

In view of the identical use patterns on apples and pears the Meeting agreed to evaluate the combined data as applying to pome fruit.

The residues of methamidophos in apples and pears in rank order (median underlined) were 0.01, 0.02, 0.06, 0.08, 0.1, 0.14, 0.15, 0.21, 0.24, 0.29, 0.31, 0.33, 0.4 and 0.49 mg/kg.

The Meeting agreed to confirm the previously estimated maximum residue level of 0.5 mg/kg, and estimated an STMR of 0.18 mg/kg for methamidophos in pome fruit. The Meeting expressed its concern at the long period of storage of many of the samples and the lack of data on the stability of residues during storage, but noted that methamidophos was scheduled for periodic review in 2002.

RECOMMENDATIONS

On the basis of data from supervised trials the Meeting concluded that the residue level shown below is suitable for establishing a Maximum Residue Limit and the supervised trials median residue is suitable for use in dietary intake estimations.

Definition of the residue for compliance with MRLs and for estimation of dietary intake: methamidophos.

Commodity		MRL, mg/kg		PHI, days	STMR, mg/kg
CCN	Name	new	previous		
FP 0009	Pome Fruit	0.5	0.5	21	0.18

FURTHER WORK OR INFORMATION

Desirable

1. Information on methamidophos residues in processed apples.

2. Data on the storage stability of residues of methamidophos for the full duration of studies to be submitted for periodic review in 2002.

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1

MEVINPHOS (053)

EXPLANATION

Mevinphos was first evaluated toxicologically in 1965 and for residues in 1972. Since the ADI was established before 1976, it is included in the CCPR periodic review programme (ALINORM 89/24A, para 299; appendix V). The 1991 CCPR scheduled the review for 1996 JMPR on the basis of the availability of new residue and toxicological data (ALINORM 91/24A para 316; Appendix IV, para 11). The residue review was postponed to 1997 by the 1995 CCPR (ALINORM 95/24A, Appendix IV).

Studies on animal and plant metabolism and environmental fate, information on analytical methods and updated GAP, reports of supervised residue trials on vegetables, fruit, pulses and tobacco, and information on residues after storage and processing were supplied by the manufacturer.

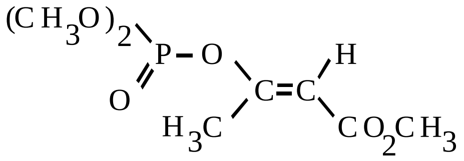
Information on national MRLs and GAP and data from residue trials were supplied variously by the governments of The Netherlands, Australia, Norway and Thailand. The Netherlands also provided information on analytical methods and residues found in food monitoring. The government of Germany informed the Meeting that the use of mevinphos is not authorized in Germany.

IDENTITY

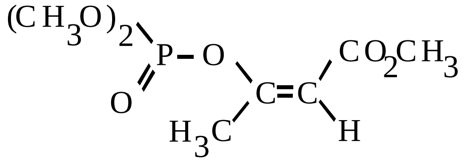
ISO common name:	mevinphos
Chemical name	
IUPAC:	2-methoxycarbonyl-1-methylvinyl dimethyl phosphate, methyl 3-(dimethoxyphosphinyloxy)but-2-enoate
CA:	methyl 3-[(dimethoxyphosphinyl)oxy]-2-butenoate
CAS No:	7786-34-7 ((*Z*)- + (*E*)- isomers) 338-45-4 ((*Z*)- isomer) 26718-65-0 ((*E*)- isomer, formerly 298-01-1)
Synonyms:	“Phosdrin” (trade name)

Structural formula:

(*E*)-mevinphos



(*Z*)-mevinphos



Molecular formula:	$C_7H_{13}O_6P$
Molecular weight:	224.1

Physical and chemical properties

Pure active ingredient

Vapour pressure:	0.0029 mm Hg at 25°C ((*E*)- isomer)		
	0.00074 mm Hg at 25°C ((*Z*)- isomer)		
Octanol/water partition coefficient:	31.6	Log P_{ow} 1.50	((*E*)- isomer)
	10.0	Log P_{ow} 1.00	((*Z*)- isomer)
Henry's law constant:	1.99 x 10-6 m3 atm/mole at 25°C		

Technical material

Composition:	(*E*)-mevinphos	63%
	(*Z*)-mevinphos	25%
	Impurities	12%
Colour:	Light yellow to orange	
Physical state:	Liquid at 20°C	
Density:	1.225 at 24.2°C	
Odour:	Little or no odour at room temperature	
Melting range:	Not applicable	
Boiling point:	98.9-103.3°C at 0.03 mm Hg	
Solubility:	Miscible in all the following	
	Water	
	Acetone	
	Benzene	
	Carbon tetrachloride	
	Chloroform	
	Ethanol	
	Methanol	
	Propan-1-ol	
	Toluene	
	Xylene	
	Insoluble in hexane	
pH:	3.2-3.5 (0.25% solution)	
Stability	Stable for 40 months at ambient temperature in sealed bottle	
	Metal and/or metal ions have no effect on the stability	

Formulations

Mevinphos is formulated as an emulsifiable concentrate (EC) or soluble concentrate (SL). The following products are currently used.

Product name:	Phosdrin IPA4
Formulation type:	SL
Solvent:	isopropyl alcohol
(*E*)- isomer content:	31.5%
(*Z*)- isomer content:	12.5%

Product name:	Phosdrin 4EC
Formulation type:	EC
Solvent:	aromatic solvent
(*E*)- isomer content:	30.4%
(*Z*)- isomer content:	12.1%

Product name:	Phosdrin 1110g/l
Formulation type:	SL
Solvent:	water
(*E*)- isomer content:	63%
(*Z*)- isomer content:	25%

METABOLISM AND ENVIRONMENTAL FATE

Animal metabolism

Studies of animal metabolism have been conducted on rats, cows, goats and hens with unlabelled, ^{32}P-labelled and ^{14}C-labelled mevinphos. The results show that mevinphos is rapidly absorbed, metabolized and excreted. Neither mevinphos nor its metabolites accumulated in the tissues.

Rats. Male and female Sprague-Dawley rats were treated orally with single doses of [*vinyl*-1-^{14}C] mevinphos, 87% (*E*)- isomer, 11% (*Z*)- isomer, at 0.15 mg/kg bw and 1.5 mg/kg bw, and multiple doses of 0.15 mg/kg bw (15 days of unlabelled followed by one day of labelled mevinphos), and intravenously with a single dose of 0.15 mg/kg bw (Reddy *et al*., 1991).

With the single low oral dose (0.15 mg/kg) the exhaled $^{14}CO_2$ averaged 77.3% of the dose in males and 78.4% in females, with 60.2% and 64.9% of the dose respectively exhaled within the first 2 hours. Only trace amounts of ^{14}C were eliminated as other volatile compounds by both male and female rats. The excretion of radioactivity in the urine was 13.6% and 14.5% by males and females respectively in 24 hours, with 12.4% and 11.8 respectively excreted within 8 hours and only minimal amounts between 8 and 24 hours. Faecal elimination of radioactivity between 0 and 24 hours was low in both males and females (1.2% and 1.4%).

In the high (1.5 mg/kg) dose rats the elimination of radioactivity in the expired air averaged 61.5% in males and 61.9% in females, with 61.4% and 61.7% of the dose exhaled as ^{14}C, most of it within 6 hours (58% by both male and females). The excretion of radioactivity in the urine was higher than in the 0.15 mg/kg group, 23.3% and 23.5% by male and female rats respectively. Most of this was eliminated within 8 hours. Small amounts of radioactivity, representing 1.3% and 1% of the administered dose in males and females respectively, were recovered from the faeces within 24 hours.

The rats treated with the series of daily oral doses of 0.15 mg/kg excreted 75% and 77.5% (males and females respectively) in the expired air, similar proportions to the single oral low-dose group. Virtually all of this (75% and 77.4%) was exhaled as $^{14}CO_2$. The excretion of radioactivity in the urine in 24 hours was 16% and 19% of the dose by male and female rats respectively, and the faecal elimination 1% and 0.7%.

After the i.v. administration of 0.15 mg/kg of [^{14}C]mevinphos the elimination of radioactivity in the exhaled air was similar to that in the single and multiple low oral dosage groups; 71.3% and 71.1%

was exhaled as $^{14}CO_2$ by males and females respectively, with trace amounts as other volatile compounds (0.07% and 0.05%). The urinary excretion of ^{14}C was also similar to that in the oral low dosage groups and averaged 16.1% and 17.4% of the dose by male and female rats respectively in 24 hours, approximately 15% of it in 8 hours.

The distribution of radioactivity in the blood and tissues after 24 hours was found to be similar in all the groups. Its total recovery from the tissues ranged from 5.4% to 7.5%. In both male and female rats the highest concentrations in all groups were found in the skin (2.4-3.0% in males and 1.9-2.2% in females) and bone (0.8-1.2% in males and 0.7-1.2% in females) The RBC and plasma levels were 0.3-0.4% in males and 0.2-0.4% in females, and the fat of both males and females contained 0.4-0.6%. Very low amounts of radioactivity were recovered from the other tissues and carcass.

Three of the four major radioactive peaks or areas separated by HPLC or TLC of urine extracts were identified as the (*E*)- isomers of mevinphos, mevinphos acid and demethyl-mevinphos. The fourth, polar, concentration of ^{14}C was not identified and may have included several components.

A representative metabolic profile from urine from the singe oral high-dose group is shown in Table 1.

Table 1. Mevinphos and its metabolites in rat urine collected 0 to 8 hours after oral administration of [^{14}C]mevinphos.

	^{14}C in urine as % of administered dose	% of total ^{14}C in urine (mean of 2 or 3 rats) found as			
		M 1	M 2	M 3	M 4
Male	22.0	19.8	25.9	24.2	13.7
Female	21.9	21.1	26.7	29.1	9.9

M1: Unidentified HPLC peak.
M2: HPLC retention matched (*E*)-demethyl-mevinphos.
M3: HPLC retention matched (*E*)-mevinphos acid.
M4: HPLC retention matched (*E*)-mevinphos.

Cows. Twelve lactating cows were dosed for 12 weeks with unlabelled mevinphos (65% (*E*)-isomer, 34% (*Z*)- isomer) at levels equivalent to 0, 1, 5 and 20 ppm in the diet on a dry matter basis by capsule (Casida *et al*., 1958). Two other lactating cows were dosed by capsule with [^{32}P]-labelled mevinphos (57.7% (*E*)-isomer, 14.9% (*Z*)- isomer, 27% impurities). One cow received a single dose of 2.0 mg/kg bw and was maintained for 1 week to study the fate of the pesticide. The second cow was dosed with 1.0 mg/kg bw per day for a week.

The anticholinesterase activities in the milk, fat, liver, kidney, muscle, heart and brain of the cows receiving up to 20 ppm mevinphos for 12 weeks corresponded to less than 0.03 mg/kg mevinphos equivalents at all dose levels throughout the dosing period.

Milk from the cow which received the single dose of [^{32}P]mevinphos contained a maximum of 0.062 mg/kg mevinphos equivalents of organosoluble radioactive material at 6 hours after administration, which decreased to below 0.007 mg/kg after 96-108 hours.

Milk from the cow dosed for 7 days with [^{32}P]mevinphos contained about 0.05 mg/kg organosoluble radioactive material from 6 hours to 7 days after the first dose.

Excretion in the faeces and urine accounted for 77% of the single dose of [^{32}P]mevinphos. Over half of this was excreted in the urine within the first 12 hours. A similar initial excretion was found with the cow dosed for 7 days.

Goats. Two lactating goats were dosed by gelatine capsules with [*vinyl*-1-^{14}C]mevinphos, 85% (*E*)- and 15% (*Z*)- isomers, for 6 successive days at a level equivalent to 18.0 or 2.9 ppm in the feed (Craine, 1992). Milk was collected twice daily, in the morning before the daily dose and in the evening, 8 hours after dosing. The repeated treatment did not affect milk production. Urine was also collected as daytime fractions (for 8 hours after dosing) and night-time fractions (8 to 24 hours after dosing).

Mevinphos was absorbed from the gastro-intestinal tract and eliminated in the urine. The patterns of urinary elimination of the radioactivity were similar for the low and high doses.

In the low-dose goat, 18.5% of the dosed radioactivity appeared in the urine during the first 8 hours, but only 2.5% in the following 16 hours. This elimination pattern was repeated through the following dosing cycles. After the 6th dose, 19.8% was excreted in the daytime urine and 3.0% in the night-time. The urinary elimination of each dose was apparently complete within 24 hours of administration. The average proportion of each dose eliminated within 24 hours was 24.3% over the 6-day period.

In the high-dose goat a higher percentage of the dose was eliminated in the urine but the elimination pattern was similar to that in the low-dose goat. After the 6th dose, 32.7% appeared in the daytime urine and 6.9% in the night-time. The average proportion of the dose eliminated within 24 hours was 38.7% over the 6-day period.

The faeces were a minor route of elimination, with average proportions of 3.38% and 2.55% of each dose eliminated by the low- and high-dose goats respectively.

Radioactivity appeared in the milk at the first (evening) collection, 8 hours after the first dose, at levels of 0.47 and 3.84 mg/kg mevinphos equivalents in the low- and high-dose milks respectively. The following morning the levels had decreased to 0.21 and 0.52 mg/kg. The day/night elimination pattern persisted and the levels of eliminated radioactivity reached a plateau after the 4th dose (Table 2).

Measurable radioactive residues were found in the blood and tissues 24 hours after the last dose was administered, and a dose relationship was evident. The highest concentrations were in the liver and kidneys. At the low dose level the residue was highest in liver at 0.646 mg/kg and in kidney at 0.382 mg/kg. At the high dose level the residue in liver was 1.873 mg/kg and in kidney was 1.897 mg/kg. Lower residues were observed in fat (0.419 mg/kg) and muscle (0.176 mg/kg).

Analysis of the milk fraction showed that the radioactivity was associated with normal endogenous components, specifically fatty acids, lactose, casein and amino acids, and the radioactivity in fractions from the liver, kidneys and muscles was associated with fatty acids, cholesterol and amino acids. The radioactivity in the fatty tissue was associated with saturated and unsaturated fatty acids, glycerol and lactic acid.

Table 2. Concentration of ^{14}C in milk of treated goats.

Treatment day	^{14}C, mg/kg as mevinphos			
	Low-dose goat		High-dose goat	
	a.m. collection	p.m. collection	a.m. collection	p.m. collection
1	--	0.47	--	3.84
2	0.21	0.65	0.52	2.48
3	0.18	0.62	1.11	4.07
4	0.27	0.82	1.25	5.09
5	0.29	0.72	0.80	4.62
6	0.22	0.73	0.88	4.73
7	0.28	--	1.05	--

Table 3. Concentration of ^{14}C in the blood and tissues of treated goats 24 hours after last dose.

Sample	**^{14}C as mevinphos, mg/kg**	
	Low-dose goat	High-dose goat
Blood	0.034	0.143
Heart	0.060	0.198
Kidney	0.128	0.636
Liver	0.187	0.826
Hindquarter muscle	0.050	0.126
Tenderloin muscle	0.040	0.095
Back fat	0.201	0.027
Omental fat	0.360	0.037

Hens. Laying hens (5 birds per group) were dosed by gelatin capsules with [*vinyl*-1-^{14}C]mevinphos, 85% (*E*)-, 15% (*Z*)- isomer, for 3 successive days at a level equivalent to 23 or 2.3 ppm in the feed (Craine, 1993).

The level of radioactivity in the excreta was fairly constant over the three-day collection period, and amounted to 23.0-29.6% and 38.5-43.1% of each daily dose for the birds in the low- and high-dose groups respectively.

Radioactivity was found in the whites of the eggs 24 hours after administration of the first dose, at 0.013 mg/kg mevinphos equivalent in the low-dose group and 0.019 mg/kg in the high-dose group, but was not detectable (<0.001 mg/kg) in the yolks at that time. The radioactivity in the whites increased with repeated dosing in both groups, and reached 0.087 and 0.876 mg/kg after the third treatment in the low- and high-dose groups respectively. Radioactivity was detected in the yolks after the second administration (0.007 and 0.017 mg/kg in the low- and high-dose groups respectively) and increased to 0.104 and 0.393 mg/kg after the third treatment.

Twenty four hours after the last dose measurable levels of ^{14}C were present in all tissues. The concentrations were highest in the liver and kidneys, with 0.646 mg/kg and 0.382 mg/kg respectively at the low dose and 1.873 mg/kg and 1.897 mg/kg respectively at the high dose. Lower residues were observed in the fat (0.419 mg/kg) and muscle (0.176 mg/kg).

Much of the radioactivity in the fat (99%), egg yolk (82%), liver (67%), kidneys (49%) and muscle (28%) could be extracted with hexane and, after saponification of the fats, was shown by HPLC to be due to incorporation into cholesterol, glycerol and long-chain fatty acids. This was confirmed by mass spectrometry. Aqueous methanol could extract additional radioactivity only from the kidneys (21%), liver

(13%) and muscle (9%). Analysis of these extracts by HPLC suggested the presence of mono- and disaccharides, with minor amounts of lactic acid and amino acids. The radioactive compounds were derivatized with BSTFA for form silyl derivatives and subjected to GC-MS which confirmed the presence of the sugars. The unextracted radioactivity could be solubilized either by protease enzymes or by hydrolysis with 6N HC1. Derivatization with butyl trifluoroacetate and analysis by HPLC suggested the incorporation of the radiolabel into amino acids, and this was confirmed by GC-MS. Neither mevinphos nor any metabolite which retained the P-O-C group was identified in any sample.

Plant metabolism

Studies have been conducted with lettuce, strawberries and turnips. The results showed that mevinphos is metabolized by two pathways in all these plants. A minor proportion is converted to mevinphos acid, whereas the major path involves the cleavage of the P-O-C group to form methyl acetoacetate. This then undergoes reduction to methyl 3-hydroxybutyrate, which was found conjugated to carbohydrates in plant tissues. The 3-hydroxybutyrate and acetoacetate can undergo hydrolysis to 3-hydroxybutyric acid and acetoacetic acid, which in turn can conjugate with carbohydrates.

Lettuce. Lettuce plants were grown in pots in the greenhouse. Six leaf lettuces were treated three times with (*vinyl*-1-^{14}C]mevinphos (82.4% (*E*)- isomer, 14.5% (*Z*)- isomer) at 0.95 kg/ha 19, 12 and 5 days before harvest. The applications were made by paint brush. Plant tops and soils were harvested 5 days after the last application (Velagaleti *et al*., 1992a). The TRR was 6.29 mg/kg mevinphos equivalents in the plant tops and 0.28 mg/kg in the soil at 0-7.6 cm depths.

One extraction with 50:50 acetonitrile/water followed by two more with 30:70 released a total of 96.7% of the TRR from the lettuce plants. The unextracted ^{14}C was not further characterized.

HPLC showed that most of the radioactivity in the extracts of the treated lettuce was due to two polar components (both 38.2% of the TRR, 2.4 mg/kg as mevinphos), (*E*)-mevinphos (6.4% of the TRR, 0.40 mg/kg), (*Z*)-mevinphos (9.8%, 0.62 mg/kg) and (*E*)-mevinphos acid (1.8%, 0.12 mg/kg). The last three components were characterized by HPLC co-chromatography with reference standards and confirmed by mass spectrometry.

Hydrolysis of the polar components and pectinase or pectinase + cellulase yielded methyl 3-hydroxybutyrate (50.1% of the TRR, 3.15 mg/kg), methyl acetoacetate (2.8%, 0.18 mg/kg), and a third component (12.7%, 0.80 mg/kg) characterized as 3-hydroxybutyric acid and/or acetoacetic acid by HPLC co-chromatography with reference standards. The esters were characterized by co-chromatography and their identities confirmed by mass spectrometry.

(*E*)-mevinphos was unstable under the conditions of enzymatic hydrolysis, but it was supposed that the identified metabolites were largely released from polar conjugates since the total radioactivity of the identified metabolites (65.6%) was almost equal to the total radioactivity of the two polar components (76.4%). The results of the chromatographic analysis of the lettuce extracts are shown in Table 4.

Table 4. Distribution of radioactivity in acetonitrile/water extracts of lettuce after application of [^{14}C]mevinphos.

Compound	Before enzyme treatment		After hydrolysis with pectinase + cellulase	
	% of TRR	mg/kg as mevinphos	% of TRR	mg/kg as mevinphos
E-mevinphos	6.4	0.40		
Z-mevinphos	9.8	0.62	9.9	0.62
E-mevinphos acid	1.8	0.12	2.1	0.13
Methyl acetoacetate			2.8	0.18
Methyl 3-hydroxybutyrate ester			50.1	3.15
Component 30 3-hydroxybutyric acid and/or acetoacetic acid[1]			12.7	0.80
Component 20	38.2	2.40		
Component 23	38.2	2.40		
Unknown			3.3	0.21
Others[2]	2.4	0.15	14.4	0.90
Total	96.8	6.09	95.2	5.99

[1]Component 30 and reference standards 3-hydroxybutyric acid and acetoacetic acid co-eluted in two different HPLC systems
[2]Scattered components and ^{14}C eluted in void volume

Strawberries. Strawberries grown in a field plot were treated three times with [*vinyl*-1-^{14}C]mevinphos (82.4% (*E*)-, 14.5% (*Z*)- isomer) at 0.95 kg/ha, 16, 9 and 2 days before harvest. The applications were made by CO_2-assisted backpack sprayer as a foliar broadcast (Velagaleti *et al*., 1992b). The harvested plants contained 2.17 mg/kg and 19.57 mg/kg mevinphos equivalent in the fruit and plant tops respectively.

More than 90% of the TRR was extracted from both fruit and plant tops by acetonitrile/water, with 3.6% and 6.6% of the TRR remaining in the fruit and tops respectively. The unextracted radioactivity was not further characterized.

HPLC analysis of the fruit (95.0% of the TRR, 2.06 mg/kg as mevinphos) demonstrated that approximately half of the extracted radioactive residue was unaltered mevinphos (34.5% of the TRR, 0.75 mg/kg as (*E*)-, and 11.1%, 0.24 mg/kg as (*Z*)-). The remaining half of the radioactivity was distributed mainly among three very polar components.

In the extract of the plant tops (95.8% of the TRR, 18.76 mg/kg), the metabolite profile was similar except that an additional polar component was present. Mevinphos represented 29% of the TRR (21.7%, 4.24 mg/kg as (*E*)-; 7.6%, 1.49 mg/kg as (*Z*)-), and 60% of the TRR was distributed among four polar components.

Pectinase hydrolysis of the conjugates present in the fruit released two major exocons, methyl 3-hydroxybutyrate (29.4% of the TRR, 0.64 mg/kg) and methyl acetoacetate (13.1%, 0.28 mg/kg), characterized by HPLC co-chromatography with reference standards. (*E*)-mevinphos acid (1.9% of the TRR, 0.04 mg/kg) was a minor hydrolysis product. Fourteen per cent of the TRR (0.31 mg/kg) was eluted as several minor polar components, none of which exceeded 3% (0.06 mg/kg). The results of hydrolysis by pectinase + cellulase were very similar to those produced by pectinase alone.

The polar components in the extracts of plant tops were also hydrolysed by pectinase, which released methyl 3-hydroxybutyrate (34.3% of the TRR, 6.7 mg/kg), methyl acetoacetate (6.6%, 1.28 mg/kg), (*E*)-mevinphos acid (2.3%, 0.44 mg/kg) and several minor components (14.8%, 2.9 mg/kg).

Pectinase did not completely hydrolyse the polar components: when the extract was treated with pectinase + cellulase one additional component which co-chromatographed with 3-hydroxybutyric acid and acetoacetic acid appeared.

The results show that the metabolic pathways of mevinphos in strawberry fruit and plant tops are essentially the same as in lettuce. The distribution of the compounds found in the fruit and tops after the application of [^{14}C]mevinphos is shown in Table 5.

Table 5. Distribution of radioactivity in acetonitrile/water extracts of strawberry fruit and plant tops after the application of [^{14}C]mevinphos.

Compound	Fruit				Plant tops			
	Before enzyme treatment		After hydrolysis with pectinase + cellulase		Before enzyme treatment		After hydrolysis with pectinase + cellulase	
	% of TRR	mg/kg[1]	% of TRR	mg/kg[1]	% of TRR	mg/kg[1]	% of TRR	mg/kg[1]
(E)-mevinphos	34.5	0.75	21.6	0.47	21.7	4.24	14.4	2.81
(Z)-mevinphos	11.1	0.24	9.6	0.21	7.6	1.49	10.0	1.95
(E)-mevinphos acid			2.0	0.04			1.8	0.35
methyl acetoacetate			14.0	0.30			9.3	1.83
methyl 3-hydroxybutyrate			29.9	0.65			35.8	7.01
Component 30, 3-hydroxybutyric acid and/or acetoacetic acid[2]							12.9	2.52
Component 18	13.3	0.29			8.9	1.74		
Component 20	4.2	0.09			3.0	0.58		
Component 23	27.1	0.59			32.9	6.43		
Component 27					15.5	3.03		
Others[3]	4.7	0.1	17.9	0.39	6.3	1.24	11.7	2.29
Total	94.9	2.06	95.0	2.06	95.8	18.76	95.9	18.77

[1]As mevinphos
[2]Component 30 and reference standards 3-hydroxybutyric acid and acetoacetic acid co-eluted in two different HPLC systems
[3]Scattered components and ^{14}C-eluted in void volume

Turnips. Field-grown turnips were treated three times with [*vinyl*-1-^{14}C]mevinphos (82.4% (*E*)-, 14.5% (*Z*)-) at 0.48 kg/ha 17, 10 and 3 days before harvest. The solution was applied directly to the foliage using laboratory sprayer units (Velagaleti *et al*., 1992c).

The turnip tubers contained 0.39 mg/kg and the tops 5.80 mg/kg mevinphos equivalent at harvest.

Acetonitrile/water extracted 89.3% of the TRR (5.18 mg/kg) from the plant tops and 7.4% (0.43 mg/kg) remained unextracted. The unextracted radioactivity was not characterized.

Acetonitrile/water extracted 70.8% of the TRR (0.28 mg/kg) from the tubers and an additional 13.5% (0.05 mg/kg) was released from the post-extraction solids by treatment with pectinase. Protease released a further 4.6% (0.02 mg/kg), bringing the total radioactivity extractable from the tubers to 88.9% of the TRR (0.35 mg/kg). Five per cent of the TRR (0.02 mg/kg) remained unextracted and was not further characterized.

HPLC analysis did not detect either (*E*)- or (*Z*)-mevinphos in the acetonitrile/water extract of the tubers, but 6.3% of the TRR (0.02 mg/kg) was accounted for by (*E*)-mevinphos acid. 3-Hydroxybutyric and acetoacetic acids and six other polar components were also detected. The radioactive material released by pectinase from the post-extraction solids showed only one HPLC peak representing 10.8% of the TRR (0.04 mg/kg), with 2.7% of the TRR (0.01 mg/kg) eluting in the void volume. The HPLC peak from the pectinase-released fraction had the same retention time as the main polar component found in the original acetonitrile/water extract.

The HPLC profile of the acetonitrile/water extract of the tubers was not significantly altered by mild acid/base or enzymatic hydrolysis, suggesting that there were no conjugates in the extracts, but harsher conditions of acid hydrolysis (1N or 5N HCl, ~100°C) caused partial hydrolysis and generated four components. This suggests that the radioactive components in the extracts may have included natural components such as proteins or carbohydrates.

In the acetonitrile/water extract of the plant tops, (*E*)-mevinphos (4.0% of the TRR, 0.23 mg/kg), (*Z*)-mevinphos (4.2%, 0.24 mg/kg) and (*E*)-mevinphos acid (2.0% of the TRR, 0.11 mg/kg) were identified by HPLC analysis. Most of the ^{14}C (76.0% of the TRR, 4.4 mg/kg) was associated with seven polar components, which were hydrolysed by pectinase or pectinase + cellulase.

Three exocons released by pectinase hydrolysis were methyl 3-hydroxybutyrate (42.8% of the TRR, 2.48 mg/kg), methyl acetoacetate (2.1%, 0.12 mg/kg) and (*E*)-mevinphos acid (5.6%, 0.32 mg/kg). Hydrolysis with pectinase + cellulase yielded several additional components, one of which was characterized as DL-3-hydroxybutyric acid and/or acetoacetic acid by co-chromatography. The other components were not identified but it was suggested that they were natural components in the turnip plant cells.

The distribution of the compounds found in turnip tubers and tops is shown in Table 6.

It can be seen that the metabolism of mevinphos in a range of plants follows the same pathway.

Table 6. Distribution of radioactivity in acetonitrile/water extracts of turnip tubers and tops after application of [^{14}C]mevinphos.

Compound	Tubers		Plant tops			
	Before enzyme treatment		Before enzyme treatment		After hydrolysis with pectinase + cellulase	
	% of TRR	mg/kg[1]	% of TRR	mg/kg[1]	% of TRR	mg/kg[1]
(*E*)-mevinphos			4.0	0.23		
(*Z*)-mevinphos			4.2	0.24	0.9	0.05
(*E*)-mevinphos acid	6.3	0.02	2.0	0.11	6.1	0.35
methyl acetoacetate					2.9	0.17
methyl 3-hydroxybutyrate					40.1	2.32
Component 30 3-hydroxybutyric acid and/or acetoacetic acid[2]	7.2	0.03	6.7	0.39	6.1	0.36
Component 17	3.0	0.01	1.4	0.08		
Component 20	11.9	0.05	13	0.75		
Component 23	28.6	0.11	36.9	2.14		
Component 26			4.5	0.26		
Component 28	3.8	0.01	10.9	0.63		
Component 33	1.4	0.01				

Compound	Tubers		Plant tops			
	Before enzyme treatment		Before enzyme treatment		After hydrolysis with pectinase + cellulase	
	% of TRR	mg/kg[1]	% of TRR	mg/kg[1]	% of TRR	mg/kg[1]
Component 43	1.1	<0.01				
Component 47			2.6	0.15		
Component 49					1.5	0.08
Component 51					1.5	0.09
Component X					8.6	0.50
Component Y					5.0	0.29
Others[3]	7.4	0.03	3.1	0.18	17.5	1.01
Total	70.8	0.28	89.3	5.18	90.0	5.22

[1]As mevinphos
[2]Component 30 and reference standards 3-hydroxybutyric acid and acetoacetic acid co-eluted in two different HPLC systems
[3]Scattered components and ^{14}C-eluted in void volume

The proposed routes of metabolism in plants are shown in Figure 1.

Environmental fate in soil

Adsorption and desorption

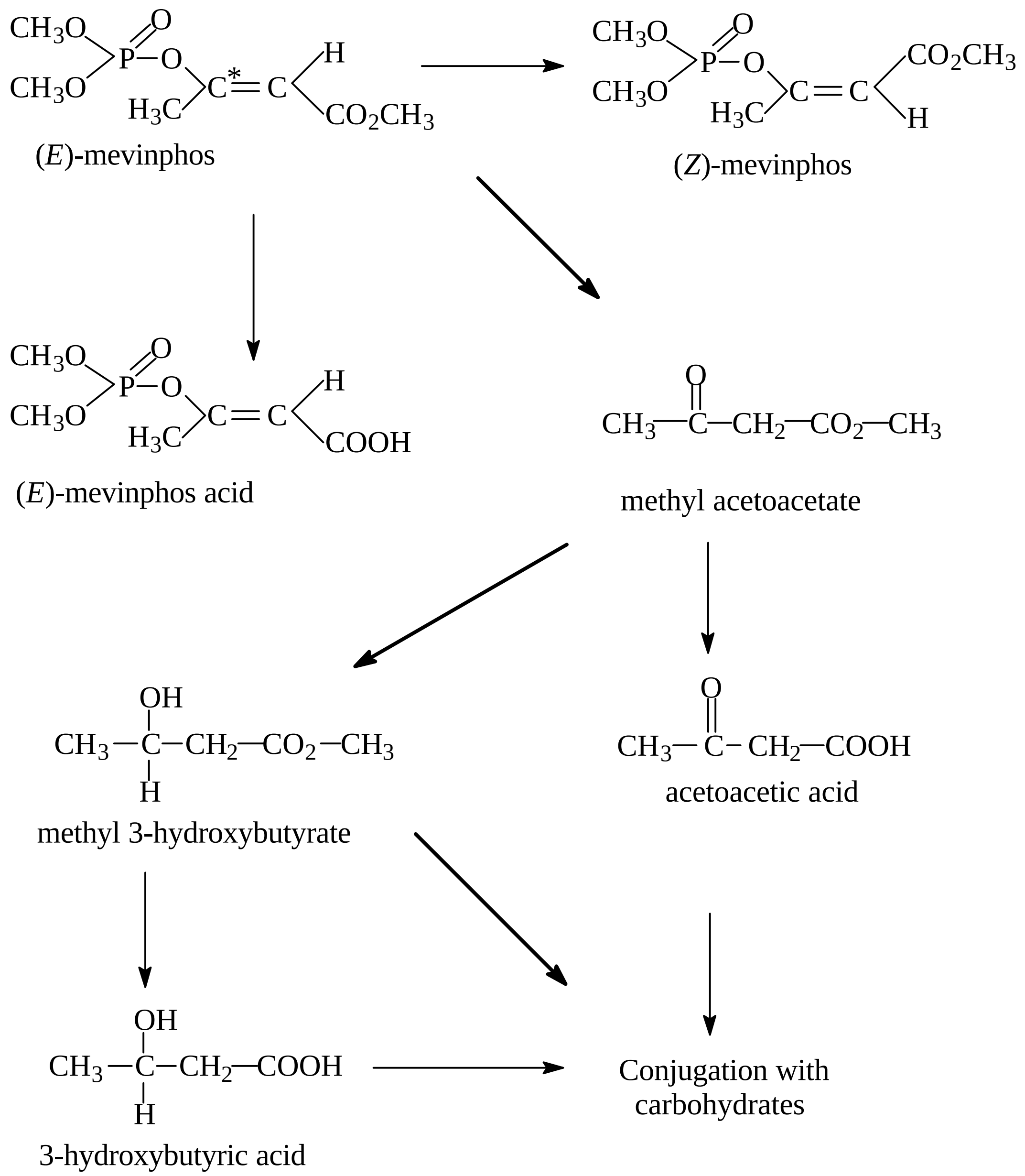
Aqueous solutions of [^{14}C]mevinphos (the proportions of (*E*)- and (*Z*)- isomers were not reported) in 0.01 M $CaCl_2$ were equilibrated with four soils and Freundlich constants were determined (Warren, 1987). The concentrations of test material in the aqueous phase were measured by liquid scintillation counting. The results are shown in Table 7.

Table 7. Soil adsorption and desorption coefficients of mevinphos.

	% Organic carbon[1]	Adsorption			Desorption		
		Kd	Koc[2]	n	Kd	Koc[2]	n
Sandy loam	0.50	0.392	78.4	1.00	1.32	264	0.949
Silt loam	1.0	0.862	86.2	1.02	1.40	140	1.03
Loam	1.5	0.607	40.5	1.02	1.16	77.3	1.03
Clay loam	2.45	1.92	78.4	1.03	3.53	144	1.05

[1]% organic carbon = % organic matter/2.0
[2]$K_{oc} = (K_d \times 100)$ / % organic carbon

Figure 1. Proposed metabolic pathways of mevinphos in plants.



Degradation under aerobic conditions

Reynolds (1994, 1995) examined degradation under aerobic conditions in the laboratory. The (*E*)- and (*Z*)-isomers of [*vinyl*-1-^{14}C]mevinphos were incubated separately with sandy loam soil at 1.1 mg/kg on a dry weight basis at 25 ± 1°C in the dark. The moisture content of the soil was adjusted to about 75% field moisture capacity initially. The radiochemical purities were >95.3% for the (*E*)- isomer and >96.9% for the (*Z*)- isomer.

Soil and alkaline trap fluid were collected intervals up to 14 days. Soil samples were extracted successively with methanol/dichloromethane and acetonitrile/acidic water. Both extracts and the bound residues were analysed by HPLC (reversed-phase for the organic extracts). Initially 99.15% of the (*E*)- and 97.12% of the (*Z*)- isomer radioactivity was extracted into methanol/dichloromethane. The percentage extracted from the soil had decreased to 2.92% and 11.50% of the total applied radioactivity for the (*E*)- and (Z)- isomers respectively after 12 hours, and 1.62 and 3.99% after 14 days.

The levels of radioactivity detected in the extracted soil increased from 1.04-1.10% initially to a maximum of 65-68% at 24 hours, then began to decrease. At the end of the experimental period (14 days), the levels were 25.5 and 46.9% of the applied radioactivity for the (*E*)- and (Z)- isomers respectively, while the evolved acidic volatiles, including $^{14}CO_2$, accounted for 66.2% and 44.6% respectively.

<u>(*E*)-mevinphos</u> accounted for 97.97% of the ^{14}C initially, but for less than 50% after 1.5 hours, for only 8.46% after 3 hours and only 1.4% after 12 hours. Up to eight degradation products were observed at various times. The main product in the organic extract after 0.75 and 1.5 hours co-eluted with the methyl acetoacetate reference standard on both reversed-phase and ion-exchange HPLC. Low levels of other metabolites were observed at later intervals, but none exceeded 4.82%. The radioactivity extracted by acetonitrile/acidic water amounted to 15.02% of the total applied after 1.5 hours, but had decreased to 2.16% after 3 hours. Characterization of the bound residues from the 1.5-hour sample showed 14.20%, 2.14% and 3.07% of the applied ^{14}C to be associated with fulvic acid, humic acid and humins respectively.

<u>(*Z*)-mevinphos</u> accounted for 94.88% of the applied ^{14}C at time 0, 55% at 3 hours and 10.23% at 12 hours. A total of six degradation products was observed. One, which was confirmed as methyl acetoacetate by both reverse-phase and ion-exchange HPLC, was detected at each sampling interval and ranged from 1.07% of the total applied radioactivity at zero time to 0.12% at 14 days. Another component which had a very short retention time increased gradually throughout the experiment and reached a maximum of 2.83% of the applied radioactivity. Low levels of other products were observed, but none exceeded 0.75%. The radioactivity extracted by acetonitrile/acidic water accounted for 3.22-4.44% of the total applied at 3 and 6 hours and 1.89% at 12 hours. In the bound residues after 3 hours 23.21-23.76% 1.41-1.48% and 3.60-3.98% of the applied ^{14}C was incorporated into fulvic acid, humic acid and humins respectively.

Mevinphos was rapidly degraded under aerobic conditions with an average half-life of 1.21 and 3.83 hours for the (*E*)- and (*Z*)- isomers respectively. Methyl acetoacetate was a major product of the (*E*)-isomer, but only a minor one of the (*Z*)- isomer. The conversion of (*E*)- to (*Z*)-mevinphos and vice versa was not a significant pathway. Both isomers of mevinphos bound quickly to the soil constituents, especially to fulvic acid. The formation of CO_2 was also very rapid and the soil-bound residues were ultimately mineralized and converted to CO_2. The distribution of radioactivity in the analytical fractions is shown in Table 8.

Table 8. Distribution of radioactivity in soil fractions at intervals after treatment with [^{14}C]-mevinphos.

Fraction	% of applied ^{14}C in fraction at									
	0	0.75 h	1.5 h	3 h	6 h	12 h	24 h	3 days	7 days	14 days
(*E*)-mevinphos										
Extracted	99.15	80.39	65.86	11.78	5.90	2.92	2.07	2.52	6.92	1.62
Unextracted	1.10	16.36	34.44	67.95	63.38	64.77	64.60	43.00	42.47	25.54
Foam plug	NA	NA	NA	NA	NA	NA	NA	0.20	0.15	NA
KOH	NA	0.50	1.96	19.11	23.30	31.08	33.73	43.94	69.11	66.23

Total	100.25	97.25	102.26	98.84	92.57	98.76	100.39	89.66	118.64	93.39
(*Z*)-mevinphos										
Extracted	97.12	88.27	70.28	56.93	36.67	11.50	6.21	3.16	3.19	3.99
Unextracted	1.04	16.34	24.43	32.40	53.37	65.32	67.93	53.24	47.27	46.89
KOH	NA	0.96	2.76	5.70	12.00	21.55	25.54	42.35	46.03	44.57
Total	98.16	105.56	97.46	95.03	102.04	98.37	99.68	98.75	96.49	95.45

Volatility from soil

The volatility of mevinphos from the surface of a sandy loam soil was examined in vaporization chambers (Lasinger, 1994). The surface of sandy loam soil was treated with [*vinyl*-1-^{14}C]mevinphos (68% (E)-, 29% (Z)-) at the rate of 0.99 kg ai/ha. The soil moisture was adjusted 75% field moisture capacity at the beginning of the study, the air flow rate was 30 ml/minute with a relative humidity of 75%, and the temperature was maintained at 25°C throughout the experimental period. The air leaving the chamber passed through a polyurethane foam plug to trap volatile material other than $^{14}CO_2$, then through an alkaline trap. The traps were sampled after 3, 6, 24, 48, 72 and 168 hours, and the soil after 168 hours. An average of 43.3% of the applied ^{14}C was recovered as volatile material after 7 days (1.1%, trapped in the polyurethane foam plug, was shown to be methyl acetoacetate and the remainder trapped in the alkaline solution to be carbon dioxide).

At the end of the incubation period the soil was extracted with a mixture of methanol and dichloromethane. The extract contained 2.5% of the applied ^{14}C (1.2% in a mixture of acetoacetic acid, *O*-demethyl-mevinphos, acetoacetic acid and 3-hydroxybutyric acid and 8 minor compounds accounting for 1.3%). The remaining 56% of the applied radioactivity was bound to the soil.

The study shows that mevinphos does not volatilize from a soil surface but becomes completely degraded with roughly half being mineralized to carbon dioxide and the remaining degradation products mainly bound to the soil.

Field dissipation

A study was carried out to examine the mobility, degradation and dissipation of mevinphos in the soil under field conditions (Leech, 1990). Six applications of mevinphos were made to a plot growing lettuce at 7-day intervals at a rate of 0.91 kg ai/ha. The soil was sandy loam with a low organic matter content. Soil samples were taken before, just after, and 2 days after each of the six treatments, then 1, 3, 7 and 14 days and 1, 2 and 4 months after the final treatment.

Residues of both isomers of mevinphos were found sporadically in the 0-15 cm layer of the soil up to 0.08 mg/kg, but were generally below the limit of determination (0.01 mg/kg). Owing to the low residues and rapid degradation of the test compound, half-lives were difficult to determine, but were apparently less than four days for both isomers.

The results also showed that the mobility of both isomers in soil is minimal. Residues of (*E*)- and (*Z*)-mevinphos were detected in the 0-15 cm layer in only a few instances and re-analysis of these samples detected no residues, indicating that the use of mevinphos in sandy soils with a low organic matter content, which is the "worst case" for potential groundwater contamination, does not present any risk. This is mainly due to the rapid degradation of both mevinphos isomers in the top 15 cm of the soil which prevents further leaching.

Residues in rotational crops

In a rotational crop study (Ryan, 1995) bare sandy loam soil was treated with a single application of [*vinyl*-1-^{14}C]mevinphos, 68% (*E*)-, 29% (*Z*)- at 0.99 kg/ha. Lettuce, sugar beet and sorghum were planted 32 days after the application. The lettuce and sugar beet were grown to maturity and harvested. The sorghum was sampled at the immature forage stage and then grown to maturity for final harvest. The harvested crops were analysed by combustion and liquid scintillation counting to determine the total radioactive residues. All samples contained <0.01 mg/kg mevinphos equivalents.

Environmental fate in water/sediment systems

Photodegradation in water

The (*E*)- and (*Z*)- isomers of [*vinyl*-1-^{14}C]mevinphos were incubated in sterile aqueous solutions, buffered at pH 5 in 0.01M sodium acetate, under artificial sunlight at 25.0 ± 1.0 °C. (Cohen, 1994a) The concentrations were 11.29 mg/l of the (*E*)- and 9.84 mg/l of the (*Z*)-, with the same solutions incubated without irradiation as controls. Samples were analysed by LSC and radio-HPLC.

The half-lives of the test compounds were determined by linear regression analysis of the log of concentration as a function of time. The results are shown in Table 9.

Table 9. Photolytic degradation half-life (days) of mevinphos.

Isomer	Irradiated	Dark control	Net rate of photolysis
(*E*)-	14.9	32.8	27.2
(*Z*)-	20.0	71.0	27.8

Both isomers isomerized. The conversion after 480 hours exposure amounted to 29.1% and 34.4% of the initial radioactivity for (*E*)- to (*Z*)- and (*Z*)- to (*E*)- respectively.

The irradiated (*E*)- isomer produced *O*-demethyl-mevinphos, an unknown, and methyl acetoacetate, which represented 13.2, 15.3 and 2.7% respectively of the initial radioactivity after 480 hours of exposure. No (*Z*)- isomer was produced in the dark control, but *O*-demethyl-mevinphos and methyl acetoacetate were identified, and represented 18.7 and 17.9% of the initial radioactivity after 480 hours.

The irradiated (*Z*)- isomer also yielded *O*-demethyl-mevinphos and the same unknown representing 11.6 and 7.3% of the initial radioactivity after 480 hours. No (*E*)- isomer was produced in the control, but *O*-demethyl-mevinphos and methyl acetoacetate were again identified, at levels equivalent to 12.6 and 0.9% of the initial radioactivity after 480 hours.

The proposed degradation pathways are shown in Figure 2.

Hydrolysis

A thirty-day study of the hydrolysis of (*E*)- and (*Z*)-mevinphos in sterile aqueous buffered solutions at pH 5, 7 and 9 was conducted at 25 ± 1°C. The rate of hydrolysis of both isomers increased with pH. The (*E*)-isomer at pH 9 produced *O*-demethyl-mevinphos, acetoacetic acid, acetone and (*E*)-mevinphos acid, whereas (*Z*)-mevinphos yielded only *O*-demethyl-mevinphos, acetoacetic acid and acetone in the same buffer. Both isomers at pH 5 and pH 7 were hydrolysed to give only *O*-demethylmevinphos as a major product. The overall hydrolysis rate of the (*E*)- isomer was found to be approximately twice that of the

(*Z*)-. The half-lives of (*E*)-mevinphos at pH 5, 7 and 9 were 50.8, 29.2 and 2.8 days respectively; and the corresponding values for the (*Z*)- isomer were 84.6, 62.7 and 7.5 days.

METHODS OF RESIDUE ANALYSIS

Analytical methods

Before 1970 residues of mevinphos were determined mainly by enzymatic methods, but GLC methods have been used since the early 1970s. Enzymatic methods could not separate the (*E*)- and (*Z*)- isomers and, since the *(E*)- isomer is a stronger inhibitor of acetylcholinesterase than the (*Z*)- and the (*Z*)- isomer is generally more persistent than the (*E*)- in or on crops, enzymatic methods do not reliably determine either the individual isomers or their sum.

<u>Enzymatic methods</u>

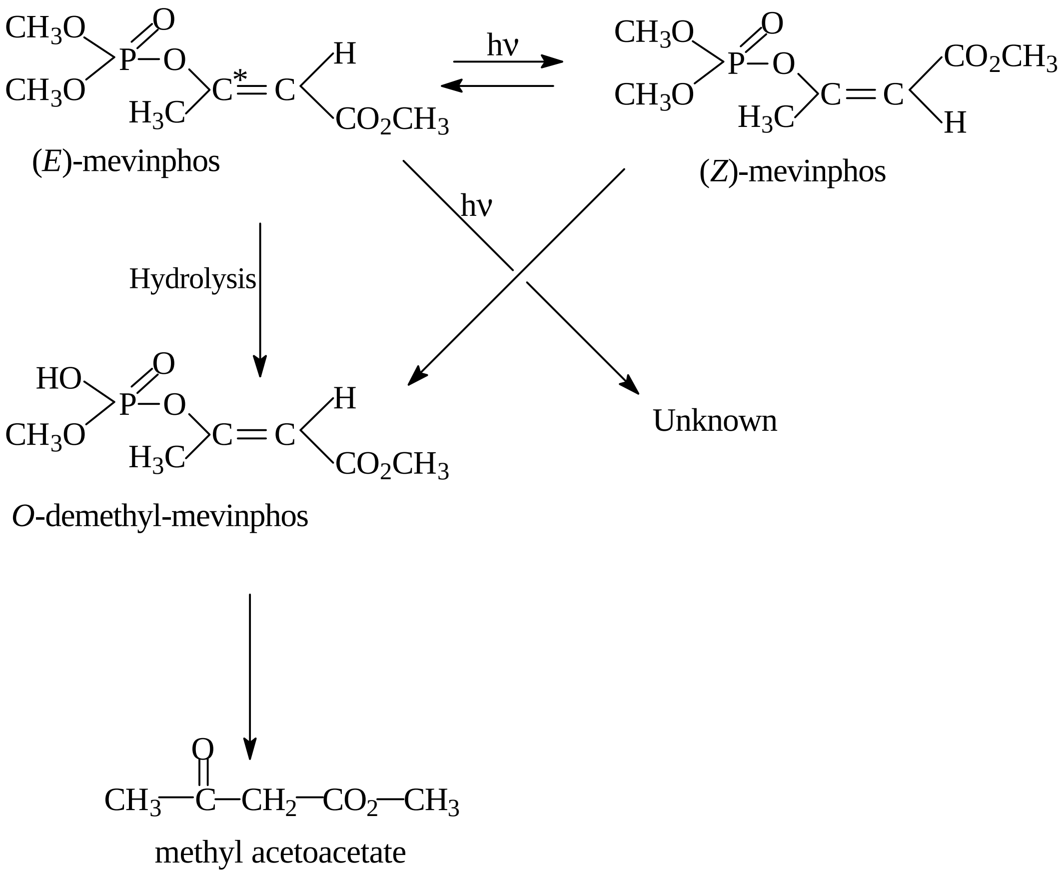
The following procedure is typical. Samples are extracted by blending with chloroform (crops) or by soxhlet extraction with petroleum ether (animal tissues), and an aliquot equivalent to 10 g of sample is concentrated to 4 ml. To this is added 8 ml of n-hexane and 0.5 ml of 6% paraffin wax in hexane, followed by 10 ml of water. After thorough mixing the organic solvent is evaporated in a stream of air, the remaining solution is shaken with 3 ml of hexane, and the organic solvent completely evaporated. The aqueous phase is diluted to give an estimated mevinphos concentration of 0.05 to 0.20 mg/l and 1.0 ml is incubated with 4-8 units of acetylcholinesterase and 1.0 ml of 0.06 M acetylcholine in buffer for 60 minutes. Incubation is stopped by adding 4.0 ml of 1 M alkaline hydroxylamine solution, then 2 ml of hydrochloric acid and 2 ml of ferric chloride reagent are added with vigorous mixing. After chilling in an ice-water bath for 5 minutes the mixture is centrifuged, the supernatant is transferred to a spectrophotometer cell, and the absorbance at 540 nm is measured with reference to distilled water. After correction for the blank the mevinphos residue is read from a standard curve.

The LOD is of the order of 0.02 mg/kg for crops and 0.1 mg/kg for animal tissues. Recoveries generally ranged from 80 to 110% at 0.1 mg/kg.

<u>GLC methods</u>

Mevinphos has been determined by GLC with flame-photometric detection sine the 1970s. Since the FPD has a specific response to phosphorus or sulfur, a wide range of crops with low fat content could be analysed without clean-up, and this has been done in most of the supervised trials. If the sample has a large amount of fat or interference is seen on chromatograms a liquid-liquid chromatographic clean-up procedure is employed. A typical procedure is as follows.

Figure 2. Proposed photodegradation pathways of mevinphos.



The chopped or ground sample (~ 100g) is macerated for several minutes with about 200 ml of chloroform or dichloromethane and 50g anhydrous sodium sulfate. If the sample has a low moisture content (e.g. grain), it is moistened with water before adding the organic solvent. After filtration, if necessary through an anhydrous sodium sulfate, the extract is injected into the gas chromatograph directly or after concentration.

If necessary, the extract is cleaned up as follows. The solvent in an aliquot of the extract equivalent to 2-3 g of sample is exchanged for n-hexane or petroleum spirit by repeatedly concentrating the solution in a stream of dry air and adding fresh solvent. This hexane extract may be suitable for injection. If not, 4 g of brickdust (Silocel C22, 60-100 mesh, containing 45% water) is packed into a chromatography column with petroleum spirit. After running off the excess petroleum spirit, 3 ml of extract is introduced and the column is eluted with 7 ml of petroleum spirit, which is discarded, then with chloroform. The first 10 ml of the chloroform fraction is collected and concentrated to 1 ml in a stream of clean air. This solution is ready for injection. In general the limit of determination was 0.01 mg/kg, with recoveries of 80-110% of both (*E*)- and (*Z*)- isomers.

In a study designed to validate typical enforcement methods for the determination of mevinphos residues in crop samples "aged" mevinphos residues derived from the study of lettuce metabolism were analysed by the methods specified in the Food and Drug Administration's Pesticide Analytical Manual (PAM II) and the Multiresidue Method (MRM).

The ^{14}C in the metabolism samples was also measured to determine the total recovery of the aged residues carried through each analytical procedure. The results showed that 16% of the total radioactivity was extracted and quantified as mevinphos by the PAM II method while only approximately 7.6% of the TRR was extracted by the MRM method. While these values appear to be low, the metabolism study showed that the mevinphos present in the metabolism samples accounted for only 16.2% of the total radioactive residue in the lettuce. The concentrations of (*E*)- and (*Z*)-mevinphos were 0.47 and 0.60 mg/kg by the PAM II method and 0.22 and 0.32 mg/kg respectively by the MRM method, while the metabolism results showed respective concentrations of 0.40 and 0.62 mg/kg. Thus the PAM II method provided a more accurate measurement of the mevinphos concentration than the MRM, although the methods showed concentrations of mevinphos of the game order, supporting their use for enforcement.

Official methods of analysis in The Netherlands

Non-fatty foods are extracted with one of several organic solvents and the residue is determined by GLC with NP or ion trap detection without clean-up. The reported LOD for lettuce was 0.05 mg/kg with recoveries of 88-94% at 0.09 and 0.43 mg/kg fortification levels with an NPD and 96% at 0.12 and 0.58 mg/kg with an ion trap.

Fatty foods are extracted with an organic solvent and cleaned up by GPC. The determination is by GLC as before. The reported LOD was 0.01-0.04 mg/kg and recoveries were 65-105% (information from the government of The Netherlands).

Stability of pesticide residues in stored analytical samples

Studies of the stability of mevinphos residues in analytical samples were carried out with the metabolism studies on lettuce, strawberries and turnips. The results, shown in Table 10, showed that both isomers of mevinphos are stable in a variety of crops under frozen conditions (~ –20 °C) for periods of about 10 months (Velagaleti *et al*., 1992a,b,c).

Lettuce harvested 5 days after of the last of three treatments with [^{14}C]mevinphos at 0.95 kg ai/ha were stored at –20 °C for 3-10 months. After storage, lettuce samples were extracted three times with acetonitrile/water, cleaned up on a C-18 reverse-phase silica cartridge column and analysed by reverse-phase HPLC.

Strawberries were harvested 2 days after the last of 3 treatments with [^{14}C]mevinphos at 0.95 kg ai/ha and stored at –20°C for 4-10.5 months. After storage, fruit and plant top samples were analysed in the same way as lettuce.

Turnips were treated three times with [^{14}C]mevinphos at 0.48 kg ai/ha, harvested 3 days after the last treatment and stored at –20°C for 2.7-9.9 months. Plant top samples were analysed in the same way as lettuce. Because no mevinphos residues were found in the tubers they were not included in the study.

Table 10. Stability of mevinphos under frozen storage (–20°C).

Storage period, months[1]	Lettuce			Strawberry fruit			Strawberry tops		Turnip tops	
	3 (0.5)	3.5 (3)	10 (2)	4 (1)	6 (4)	10.5 (1.5)	4 (4)	10.5 (1.8)	2.7 (6.1)	9.9 (1.1)
E-mevinphos	100	85	91	100	103	104	100	92	100	116
Z-mevinphos	100	91	99	100	103	98	100	97	100	87

[1]Months in parentheses are storage periods of acetonitrile/water extracts

Definition of the residue

The plant metabolism studies showed that residues of mevinphos are degraded rapidly by cleavage of the P-O-C group, with the formation of methyl acetoacetate. Mevinphos is also converted to (*E*)-mevinphos acid by a minor route but the Meeting concluded that the level of (*E*)-mevinphos acid was low in relation to that of mevinphos and it could be excluded from the definition of the residue, which should be “sum of (*E*)- and (*Z*)-mevinphos" for both enforcement and the estimation of dietary intake.

USE PATTERN

Mevinphos is a systemic and contact organophosphate insecticide and acaricide. It is used to protect a wide range of crops such as pome and stone fruit, berries and small fruit, fruiting vegetables, bulb, stem and root vegetables, pulses, nuts and beet. It is also used on ornamentals and tobacco. The registered uses on food crops are shown in Table 11 and those on ornamentals and tobacco in Table 12.

Table 11. Registered uses of mevinphos on food and feed commodities. All EC applications.

Crop[1]	Country	Application			No.	PHI, days[2]
		Method	kg ai/ha	kg ai/hl		
Apples	Austria	Spray	0.096-0.19	0.0096-0.019		14
Apples	France	Spray	0.25-0.75	0.05	4	7
Apples	Netherlands	Spray	0.11-0.16	0.011	1	7
Apples	Netherlands	Spray	0.073-0.11	0.007	3	7
Apples	Portugal	Spray		0.012-0.036		4
Apples	Switzerland	Spray	0.54	0.036		21
Apricots	Austria	Spray	0.096-0.19	0.0096-0.019		14
Apricots	France	Spray	0.25-0.75	0.05	8	7
Cherries	Austria	Spray	0.096-0.19	0.0096-0.019		14
Cherries	Netherlands	Spray	0.073-0.11	0.007	3	7
Citrus	South Africa	Spray		0.015		3
Grapes	Austria	Spray	0.096-0.19	0.0096-0.019		14
Grapes	France	Spray	0.05-0.25	0.05	3	7
Grapes	South Africa	Spray		0.019-0.023		7
Grapes	Switzerland	Spray	0.54	0.036		21
Grapes (G)	Netherlands	Spray	0.036-0.11	0.007	3	7 or 14
Peaches	Austria	Spray	0.096-0.19	0.0096-0.019		14
Peaches	France	Spray	0.25-0.75	0.05	8	7
Peaches (G)	Netherlands	Spray	0.036-0.11	0.007	3	7 or 14
Pears	Austria	Spray	0.096-0.19	0.0096-0.019		14
Pears	Netherlands	Spray	0.073-0.11	0.007	3	7
Pears	Portugal	Spray		0.012-0.036		4
Pears	Switzerland	Spray	0.54	0.036		21
Plums	Austria	Spray	0.096-0.19	0.0096-0.019		14
Plums	France	Spray	0.25-0.75	0.05	8	7
Plums	Netherlands	Spray	0.073-0.11	0.007	3	7
Plums (G)	Netherlands	Spray	0.036-0.11	0.007	3	7 or 14
Berries and small fruit (F/G)	Netherlands	Spray	0.073-0.087	0.007	3	7 (F) 7 or 14 (G)
Strawberries	France	Spray	0.35	0.088-0.3	4	7
Brassicas	Netherlands	Spray	0.015-0.11	0.007-0.011	3	7
Brassicas	South Africa	Spray	0.11	0.011		4
Brassicas	Thailand	Spray	0.36-0.48	0.036-0.048		3
Brassicas	Zimbabwe	Spray	0.19	0.019		4

Crop[1]	Country	Application			No.	PHI, days[2]
		Method	kg ai/ha	kg ai/hl		
Broccoli	Australia	Spray	0.29-1.1	0.072		2
Broccoli	Finland	Spray	0.24-0.48			4
Broccoli	Sweden	Spray	0.24-0.48			4
Brussels sprouts	Australia	Spray	0.29-1.1	0.072		2
Cabbages	Australia	Spray	0.29-1.1	0.072		2
Cabbages	Austria	Spray	0.096	0.0096		14
Cabbages	Finland	Spray	0.24-0.48			4
Cabbages	France	Spray	0.35	0.035	3	7
Cabbages	Sweden	Spray	0.24-0.48			4
Carrots	Australia	Spray	0.29-0.87	0.072		2
Carrots	Zimbabwe	Spray	0.19	0.019		4
Cauliflower	Australia	Spray	0.29-1.1	0.072		2
Celery	Australia	Spray	0.29-0.87	0.072		2
Corn salad	France	Spray	0.35		1	7
Courgettes	France	Spray	0.35	0.035	2	7
Courgettes	Netherlands	Spray	0.015-0.15	0.007-0.015	6	7
Cucumbers (G)	Belgium	pulverization or nebulization	0.12-0.36	0.012-0.018	3	3
Cucumbers (G)	Finland	Spray		0.024		4
Cucumbers	France	Spray	0.35	0.035	5	7
Cucumbers (G)	Luxembourg	pulverization or nebulization	0.12-0.36	0.012-0.018	3	3
Cucumbers	Norway	Spray		0.024		7
Cucumbers	Portugal	Spray		0.036		4
Cucumbers (G)	Sweden	Spray		0.024		3
Cucurbits	Australia	Spray	0.29-0.87	0.072		2
Cucurbits	South Africa	Spray	0.11	0.011		4
Egg plants	France	Spray	0.35	0.035		7
Egg plants	Australia	Spray	0.29-0.87	0.072		2
Fruiting vegetables (G)	Netherlands	Spray	0.073-0.22	0.007-0.015	4	3
Garden cress	France	Spray	0.35	0.088-0.3		7
Garlic	Portugal	Spray		0.036		4
Gherkins (G)	Belgium	pulverization or nebulization	0.12-0.36	0.012-0.018	3	3
Gherkins	France	Spray		0.035	2	7
Gherkins (G)	Luxembourg	pulverization or nebulization	0.12-0.36	0.012-0.018	3	3
Gherkins	Netherlands	Spray	0.015-0.15	0.007-0.015	6	3
Globe artichokes	Australia	Spray	0.29-0.87	0.072		2
Herbs (F/G)	Netherlands	Spray	0.015-0.058	0.007	3	7 (F) 7 or 14 (G)
Legume vegetables	Netherlands	Spray	0.015-0.15	0.007-0.015	3	7
Legume vegetables (G)	Netherlands	Spray	0.036-0.15	0.007-0.015	2	7 or 14
Lettuce	Australia	Spray	0.29-0.87	0.072		2
Lettuce (G)	Finland	Spray	0.24-0.48		2	~~7~~
Lettuce	France	Spray	0.35	0.035	4	7
Lettuce	South Africa	Spray	0.11	0.011		4
Lettuce	Sweden	Spray	0.24-0.48			7
Lettuce	Netherlands	Spray	0.015-0.22	0.007-0.011	3	7
Lettuce (G)	Netherlands	Spray	0.036-0.15	0.007-0.015	2	7 or 14
Lettuce	Norway	Spray		0.024		7
Lucerne	Australia	Spray	0.39			2
Melons	France	Spray	0.35	0.035	2	7
Melons	Portugal	Spray	0.36-0.72	0.036	2	4

Crop[1]	Country	Application			No.	PHI, days[2]
		Method	kg ai/ha	kg ai/hl		
Onions	Australia	Spray	0.29-0.58	0.072		2
Onions	Netherlands	Spray	0.029-0.15	0.015	6	7
Onions	Thailand	Spray	0.075-0.15	0.012-0.024		3
Parsnips	Australia	Spray	0.29-0.87	0.072		2
Peppers (G)	Belgium	pulverization or nebulization	0.12-0.36	0.012-0.018	3	7 or 14
Peppers	France	Spray	0.35	0.035		7
Peppers (G)	Luxembourg	pulverization or nebulization	0.12-0.36	0.012-0.018	3	7 or 14
Peppers	South Africa	Spray	0.094	0.019		2
Peppers (Capsicums)	Australia	Spray	0.29-0.87	0.072		2
Potatoes	Australia	Spray	0.29-0.87	0.072		2
Potatoes	Zimbabwe	Spray	0.19	0.019		4
Rhubarb	Australia	Spray	0.29-0.87	0.072		2
Root and tuber vegetables	Netherlands	Spray	0.015-0.15	0.007-0.015	6	7
Root and tuber vegetables (G)	Netherlands	Spray	0.036-0.15	0.007-0.015	2	7 or 14
Shallots	Netherlands	Spray	0.029-0.15	0.015	6	7
Spinach	Australia	Spray	0.29-0.87	0.072		2
Spinach	France	Spray	0.35	0.035	2	7
Spinach	Netherlands	Spray	0.015-0.22	0.007-0.011	3	7
Spinach	South Africa	Spray	0.11	0.011		3
Spinach (G)	Netherlands	Spray	0.034-0.11	0.007-0.011	2	7 or 14
Stem vegetables	Netherlands	Spray	0.015-0.15	0.007-0.015	3	7
Stem vegetables (G)	Netherlands	Spray	0.036-0.073	0.007	2	7 or 14
Sweet corn	Australia	Spray	0.29-1.1	0.072		2
Sweet corn	Thailand	Spray	0.06-0.12	0.012-0.024		3
Tomatoes	Australia	Spray	0.29-1.1	0.072		2
Tomatoes (G)	Belgium	Pulverization/nebulization	0.12-0.36	0.012-0.018	3	7 or14
Tomatoes (G)	Finland	Spray		0.024		3
Tomatoes	France	Spray	0.35	0.035		7
Tomatoes (G)	Luxembourg	Pulverization/nebulization	0.12-0.36	0.012-0.018	3	7 or 14
Tomatoes	Norway	Spray		0.024		7
Tomatoes	Portugal	Spray	0.36-0.96	0.036-0.048	2	4
Tomatoes	South Africa	Spray		0.019		2
Tomatoes (G)	Sweden	Spray		0.024		3
Tomatoes	Thailand	Spray	0.075-0.15	0.012-0.024		3
Tomatoes	Zimbabwe	Spray	0.14	0.029		2
Witloof	France	Spray	0.35	0.088-0.3	1	7
Beetroot	Australia	Spray	0.29-0.58	0.072		2
Beetroot	Zimbabwe	Spray	0.19	0.019		4
Beets	France	Spray	0.35	0.035	2	7
Silver beet	Australia	Spray	0.29-0.87	0.072		2
Sugar beet	Austria	Spray	0.12	0.02-0.03		14
Sugar beet	France	Spray	0.35	0.035	2	7
Fodder beet	France	Spray	0.35	0.035	2	7
Beans	Australia	Spray	0.29-0.87	0.072		2
Beans	France	Spray	0.35	0.035	2	7
Beans	South Africa	Spray	0.11	0.011		4
Beans	Zimbabwe	Spray	0.19	0.019		4
Broad beans	Australia	Spray	0.29-0.87	0.072		2

Crop[1]	Country	Application			No.	PHI, days[2]
		Method	kg ai/ha	kg ai/hl		
Peas	France	Spray	0.35	0.035	2	7
Peas	South Africa	Spray	0.11	0.011		4
Peas	Switzerland	Spray	0.096	0.024		21
Peas	Zimbabwe	Spray	0.19	0.019		4
Peas (fresh)	Netherlands	Spray	0.058-0.29	0.029	3	7
Maize	Thailand	Spray	0.06-0.12	0.012-0.024		3
Wheat	South Africa	Spray	0.06-0.12			7
Wheat	Zimbabwe	Aerial spray	0.096-0.19			7

[1]F = Field; G = Glasshouse
[2]"7 or 14" means a PHI of 7 days for the period from March to October and 14 days from November to February

Table 12. Registered uses of mevinphos on ornamentals and tobacco. All spray applications of EC formulations.

Crop	Country	Application			PHI, days
		kg ai/ha	kg ai/hl	No.	
Tobacco	South Africa	0.15			7
Cut flowers	Netherlands	0.073-0.11	0.0073-0.011		
Flowers	Austria	0.096-0.19	0.0096-0.019		14
Gladioli	Australia	0.78			2
Ornamentals	Austria	0.096-0.19	0.0096-0.019		14
Ornamentals	Finland	0.024-0.12	0.0024-0.03	5	4
Ornamentals	South Africa		0.015		
Ornamentals	Switzerland	0.48	0.024		
Pot plants	Netherlands	0.073-0.11	0.0073-0.011		
Roses	France		0.035	4	
Tree nurseries	Netherlands	0.073-0.11	0.0073-0.011		

RESIDUES RESULTING FROM SUPERVISED TRIALS

The results of supervised trial on agricultural crops are shown in Tables 13-24. All trials were reported on detailed summary sheets, but necessary information was well documented except where indicated.

The trials were with several types of formulation (e.g. 10-50% EC, 50% WP, 24% SL and technical grade active ingredient), but in view of the high solubility of mevinphos in water it is unlikely that the type of formulation will significantly affect the residue level.

The information on recoveries and limits of determination was generally sufficient for evaluation.

In the trials before 1970 residues were determined by enzymatic methods. Because the (*E*)-isomer is a stronger inhibitor of acetylcholinesterase than the (*Z*)- and the (*Z*)- isomer is more persistent than the (*E*)- the results of enzymatic analyses are likely to be lower than those obtained by GLC. The Meeting therefore agreed not to use the data from trials in which the analyses were by enzymatic methods. It was also agreed not to use data from trials in which the conditions or duration of the storage of analytical samples were not reported, because such information is essential to assess the validity of the data. The trials which were excluded on these grounds are shown shaded in the Tables.

Residues resulting from trials according to GAP are underlined.

Table 13. Residues of mevinphos in oranges. South Africa, 1972.

Form.	Application			PHI, days	Residues, mg/kg[1]			Ref.
	No.	kg ai/ha	kg ai/hl		(*E*)- isomer	(*Z*)- isomer	Total	
24%	1		0.04	0	f<0.01, p0.55	f<0.01, p0.15	f<0.02, p0.70	WKGR
EC							w0.20	0187.72
				2	f<0.01, p0.04	f<0.01, p0.04	f<0.02, p0.08	
							w0.04	
				7	f<0.01, p0.02	f<0.01, p0.02	f<0.02, p0.04	
							w0.02	

[1]f = pulp; p = peel; w = whole fruit

Table 14. Residues of mevinphos in pome fruit. All single applications.

Crop, Country, Year	Application			PHI, days	Residues, mg/kg[1]			Ref.
	Form	kg ai/ha	kg ai/hl		(E)- isomer	(Z)- isomer	Total	
Apple	10%	0.5	0.05	0	0.17 (0.13)	0.25 (0.22)	0.42 0.35)	BEGR
France	EC			3	0.16 (0.07)	0.26 (0.16)	0.42 (0.23)	0041/70
1969				7	0.07 (0.06)	0.22 (0.17)	<u>0.29</u> (0.23)	
				14	0.01 (<0.01)	0.11 (0.07)	0.12 (0.08)	
		1.0	0.10	0	0.43 (0.11)	0.39 (0.19)	0.82 (0.30)	
				3	0.36 (0.11)	0.45 (0.17)	0.81 (0.28)	
				7	0.23 (0.09)	0.26 (0.16)	0.49 (0.25)	
				14	0.04 (0.01)	0.13 (0.09)	0.17 (0.10)	
		0.5	0.05	0	0.15 (0.05)	0.11 (0.16)	0.26 (0.21)	
				3	0.12 (0.07)	0.17 (0.15)	0.29 (0.22)	
				7	0.10 (0.07)	0.18 (0.15)	<u>0.28</u> (0.22)	
				14	<0.01 (<0.01)	0.14 (0.07)	0.15 (0.08)	
		1.0	0.10	0	0.37 (0.21)	0.35 (0.28)	0.72 (0.49)	
				3	0.35 (0.16)	0.34 (0.30)	0.69 (0.46)	
				7	0.25 (0.15)	0.37 (0.23)	0.62 (0.38)	
				14	0.03 (0.03)	0.20 (0.15)	0.23 (0.18)	
Apple	TG[2]	0.5		1	0.45, 0.30	0.20, 0.15	0.65, 0.45	WKGR
UK				3	0.30, 0.10	0.15, 0.10	0.45, 0.20	0052.72
1971				7	0.15, 0.08	0.10, 0.07	<u>0.25</u>, 0.15	
				10	0.08, 0.06	0.10, 0.09	0.18, 0.15	
				14	0.10, 0.05	0.09, 0.10	0.19, 0.15	
		1.0		1	0.90, 0.70	0.35, 0.30	1.25, 1.00	
				3	0.40, 0.80	0.20, 0.35	0.60, 1.15	
				7	0.40, 0.40	0.15, 0.25	0.55, 0.65	
				10	0.30, 0.35	0.20, 0.25	0.50, 0.60	
				14	0.15, 0.15	0.15, 0.20	0.30, 0.35	
Apple	24%	0.25		0	0.15, 0.15	0.05, 0.06	0.20, 0.21	WKGR
UK	SL			2	0.06, 0.04	0.04, 0.03	0.10, 0.07	0005.73
1972				5	<0.01, <0.01	<0.01, 0.01	<0.02, 0.02	
				8	<0.01, <0.01	0.02, 0.02	<u>0.03</u>, 0.02	
				13	<0.01, 0.02	0.02, <0.01	<u>0.03</u>, 0.03	
		0.5		0	0.50, 0.30	0.15, 0.10	0.65, 0.40	
				2	0.15, 0.15	0.07, 0.08	0.22, 0.23	
				5	0.06, 0.05	0.05, 0.05	0.11, 0.10	
				8	0.04, 0.03	0.05, 0.04	<u>0.09</u>, 0.07	

Crop, Country, Year	Application			PHI, days	Residues, mg/kg[1]			Ref.
	Form	kg ai/ha	kg ai/hl		(E)- isomer	(Z)- isomer	Total	
				13	0.01, 0.02	0.04, 0.03	0.05, 0.05	
	24%	0.25		0	0.10, 0.10	0.04, 0.04	0.14, 0.14	
	EC			2	0.03, 0.03	0.03, 0.03	0.06, 0.06	
				5	<0.01, <0.01	0.02, 0.02	0.03, 0.03	
				8	<0.01, <0.01	0.02, 0.02	0.03, 0.03	
				13	<0.01, <0.01	0.02, 0.01	0.03, 0.02	
Pear	10%	0.5	0.05	0	0.10 (0.04)	0.05 (0.01)	0.15 (0.05)	BEGR
France	EC			1	0.03 (<0.01)	0.01 (<0.01)	0.04 (<0.02)	0020/70[3]
1969				3	0.02 (<0.01)	<0.01 (<0.01)	0.03 (<0.02)	
				7	<0.01 (<0.01)	<0.01 (<0.01)	<0.02 (<0.02)	
				14	<0.01 (<0.01)	<0.01 (<0.01)	<0.02 (<0.02)	
				21	<0.01 (<0.01)	<0.01 (<0.01)	<0.02 (<0.02)	
		1.0	0.10	0	0.22 (0.10)	0.09 (0.05)	0.31 (0.15)	
				1	0.12 (0.05)	0.06 (0.01)	0.18 (0.06)	
				3	0.06 (0.03)	0.03 (0.01)	0.09 (0.04)	
				7	0.03 (0.01)	0.01 (<0.01)	0.04 (0.02)	
				14	<0.01 (<0.01)	<0.01 (<0.01)	<0.02 (<0,02)	
				21	<0.01 (<0.01)	<0.01 (<0.01)	<0.02 (<0.02)	

[1]Figures in parentheses are for peeled fruits
[2]Technical grade active ingredient was used
[3]Sample storage conditions not clear

Table 15. Residues of mevinphos in stone fruit.

Crop, country, year	Application				PHI, days	Residues, mg/kg[1]			Reference
	Form.	No.	kg ai/ha	kg ai/hl		(*E*)- isomer	(*Z*)- isomer	Total	
Apricot		1	0.28		0			0.15	RES
USA					1			0.13	58-65[2,3]
1958					3			0.10	
					7			0.04	
Cherry	10%	1	0.5	0.05	1	0.55	0.25	0.80	BEGR
France	EC				3	0.20	0.10	0.30	0056/70
1970					5	0.10	0.08	0.18	
					7	0.04	0.05	0.09	
		1	1.0	0.10	1	0.70	0.35	1.05	
					3	0.30	0.15	0.45	
					5	0.10	0.06	0.16	
					7	0.05	0.05	0.10	
		1	0.5	0.05	1	1.10	0.55	1.65	
					3	0.60	0.30	0.90	
					5	0.45	0.35	0.80	
					7	0.20	0.25	0.45	
		1	1.0	0.10	1	1.80	0.85	2.65	
					3	1.60	0.70	2.30	
					5	0.95	0.65	1.60	
					7	0.35	0.60	0.95	
Cherry	10%	1	0.5	0.05	0	0.30	0.15	0.45	BEGR
France	EC				8	0.20	0.15	0.35	0018.72
1971					14	0.05	0.08	0.13	
		1	1.0	0.10	0	1.20	0.55	1.75	
					8	0.50	0.30	0.80	

Crop, country, year	Application				PHI, days	Residues, mg/kg[1]			Reference
	Form.	No.	kg ai/ha	kg ai/hl		(*E*)- isomer	(*Z*)- isomer	Total	
					14	0.25	0.25	0.50	
		1	0.5	0.05	1	0.45	0.25	0.70	
					7	0.35	0.20	0.55	
					14	0.15	0.20	0.35	
		1	1.0	0.10	1	2.10	0.90	3.00	
					7	0.65	0.35	1.00	
					14	0.25	0.25	0.50	
Cherry	50%	3		0.025	0	0.90	0.31	1.21	WKGR
Germany	EC				7	0.09	0.07	0.16	0172.74
1974					10	0.04	0.05	0.09	
					14	0.01	0.02	<u>0.03</u>	
					21	<0.01	<0.01	<0.02	
		3		0.025	0	0.29	0.16	0.45	
					7	0.01	0.02	0.03	
					10	<0.01	0.02	0.03	
					14	<0.01	0.01	<u>0.02</u>	
					21	<0.01	<0.01	<0.02	
					28	<0.01	<0.01	<0.02	
		3		0.025	7	0.08	0.06	0.14	
					10	0.09	0.08	0.17	
					14	0.01	0.03	<u>0.04</u>	
					21	<0.01	<0.01	<0.02	
Cherry	48%	3	0.24	0.024	0	0.21	0.11	0.32	BEGR
Germany	EC				4	0.12	0.09	0.21	83.016
1982					7	0.07	0.07	0.14	
					10	0.04	0.05	0.09	
Cherry	48%	3	0.24	0.024	0	0.55	0.23	0.78	BETR
Germany	EC				7	0.16	0.13	0.29	84.011
1983					14	0.02	0.07	<u>0.09</u>	
					21	<0.01	0.05	0.06	
Cherry		1	0.28		0			0.64	RES
USA					1			0.56.	58-65
1958					3			0.45	2), 3)
					7			0.31	
					14			0.11	
					21			0.10	
Peach	10%	1	0.5	0.05	0	0.51 (0.51)	0.27 (0.27)	0.78 (0.78)	BEGR
France	EC				1	0.23 (0.23)	0.14 (0.13)	0.37 (0.36)	0019/70[4]
1969					3	0.15 (0.10)	0.10 (0.04)	0.25 (0.14)	
					7	0.08 (0.06)	0.06 (0.04)	0.14 (0.10)	
					14	<0.01 (<0.01)	0.10 (0.01)	0.11 (0.02)	
					21	<0.01 (<0.01)	0.03 (0.02)	0.04 (0.03)	
		1	1.0	0.10	0	1.50 (0.71)	0.80 (0.39)	2.30 (1.10)	
					1	1.10 (0.73)	0.60 (0.37)	1.70 (1.10)	
					3	0.57 (0.28)	0.36 (0.11)	0.93 (0.39)	
					7	0.25 (0.20)	0.17 (0.15)	0.42 (0.35)	
					14	0.25 (0.19)	0.17 (0.16)	0.42 (0.35)	
					21	0.04 (<0.01)	0.05 (0.03)	0.09 (0.04)	
Peach	50%	3		0.025	0	0.60	0.21	0.81	WKGR
Germany	EC				7	0.04	0.03	0.07	0023.75
1974					10	0.01	0.02	0.03	
					14	0.01	0.01	<u>0.02</u>	

Crop, country, year	Application				PHI, days	Residues, mg/kg[1]			Reference
	Form.	No.	kg ai/ha	kg ai/hl		(*E*)- isomer	(*Z*)- isomer	Total	
					21	<0.01	<0.01	<0.02	
		3		0.025	0	0.31	0.11	0.42	
					7	0.03	0.01	0.04	
					10	0.02	0.01	0.03	
					14	0.02	0.01	0.03	
					21	<0.01	<0.01	<0.02	
		3		0.025	0	0.76	0.26	1.02	
					7	0.03	0.05	0.08	
					10	0.02	0.03	0.05	
					14	<0.01	<0.01	<0.02	
					21	<0.01	<0.01	<0.02	
Peach	15%	5	0.56	0.15	0			0.32	RES
USA	EC				1			0.09	57-45[2,3]
1957					2			0.05	
					4			0.04	
					7			0.04	

[1]Figures in parentheses are for peeled fruits
[2]Sample storage period not clear.
[3]Analyses by enzymatic method
[4]Sample storage conditions not clear

Table 16. Residues of mevinphos in small fruits and berries.

Crop, country, year	Application				PHI, days	Residues, mg/kg[1]			Ref.
	Form.	No.	kg ai/ha	kg ai/hl		(E)-isomer	(Z)-isomer	Total	
Currant	50%	3		0.025	0	1.40	0.45	1.85	BEGR
Germany	EC				7	0.07	0.03	0.10	0113.74
1974					10	0.01	<0.01	0.02	
					14	<0.01	<0.01	<0.02	
					21	<0.01	<0.01	<0.02	
		3		0.025	0	1.30	0.45	1.75	
					7	0.03	0.01	0.04	
					10	0.02	0.01	0.03	
					14	<0.01	<0.01	<0.02	
					21	<0.01	<0.01	<0.02	
Currant	50%	3		0.025	0	0.40	0.18	0.58	BEGR
Germany	EC				7	0.03	0.04	0.07	0114.74
1974					10	0.02	0.03	0.05	
					14	<0.01	<0.01	<0.02	
					21	<0.01	<0.01	<0.02	
					28	<0.01	<0.01	<0.02	
		3		0.025	0	0.45	0.09	0.54	
					7	<0.01	<0.01	<0.02	
					10	<0.01	<0.01	<0.02	
					14	<0.01	<0.01	<0.02	
					21	<0.01	<0.01	<0.02	
		3		0.025	0	0.80	0.37	1.17	
					7	0.04	0.10	0.14	
					10	0.02	0.10	0.12	
					14	<0.01	0.05	0.06	
					21	<0.01	<0.01	<0.02	

Crop, country, year	Application				PHI, days	Residues, mg/kg[1]			Ref.
	Form.	No.	kg ai/ha	kg ai/hl		(E)-isomer	(Z)-isomer	Total	
Currant	50%	3		0.025	0	2.30	1.00	3.30	BEGR
Germany	EC				7	0.10	0.04	0.14	0095.75
1975					10	0.05	0.02	0.07	
					14	<0.01	<0.01	<0.02	
					21	<0.01	<0.01	<0.02	
					25	<0.01	<0.01	<0.02	
Currant	24%	1	0.28		1	0.32	0.12	0.44	WKGR
UK	EC				3	0.19	0.10	0.29	0028.72
1971					6	0.07	0.05	0.12	
		1	0.56		1	0.32	0.12	0.44	
					3	0.43	0.18	0.61	
					6	0.08	0.05	0.13	
		1	0.28		1	0.26	0.13	0.39	
					3	0.34	0.18	0.52	
					6	0.09	0.06	0.15	
		1	0.56		1	0.60	0.22	0.82	
					3	0.34	0.21	0.55	
					6	0.12	0.08	0.20	
Grape	10%	1	0.5	0.05	0	0.45	0.33	0.78	BEGR
France	EC				1	0.12	0.08	0.20	0018.70[1]
1969					3	0.12	0.09	0.21	
					7	0.02	0.03	0.05	
					14	<0.02	<0.02	<0.04	
					21	<0.02	<0.02	<0.04	
		1	1.0	0.10	0	1.40	0.53	1.93	
					1	0.43	0.23	0.66	
					3	0.16	0.11	0.27	
					7	0.12	0.15	0.27	
					14	0.02	0.04	0.06	
					21	<0.02	0.02	0.04	
		1	0.5	0.05	0	0.25	0.12	0.37	
					1	0.05	0.03	0.08	
					3	0.05	0.03	0.08	
					7	<0.02	<0.02	<0.04	
					14	<0.02	<0.02	<0.04	
					21	<0.02	<0.02	<0.04	
		1	1.0	0.10	0	0.60	0.20	0.80	
					1	0.17	0.07	0.24	
					3	0.16	0.07	0.23	
					7	0.09	0.03	0.12	
					14	<0.02	<0.02	<0.04	
					21	<0.02	<0.02	<0.04	
Grape	24%	1	0.15		5	<0.01	<0.01	<u><0.02</u>	WKGR
France	EC								077.72
1971	2% D	2	0.48		5	<0.01	<0.01	<0.02	
Grape	24%	1		0.045	0	0.58	0.17	0.75	WKGR
South Africa	EC				3	0.14	0.07	0.21	0182.70
1970					7	0.06	0.05	0.11	
Grape	24%	1		0.045	0	0.90	0.28	1.18	BEGR
South Africa	EC				2	0.73	0.24	0.97	0092.74
1974					4	0.27	0.19	0.46	
Grape		1	0.56		0			0.20	RES
USA					1			0.06	57-36[2]
1957					2			<0.05	
					3			<0.05	

Crop, country, year	Application				PHI, days	Residues, mg/kg[1]			Ref.
	Form.	No.	kg ai/ha	kg ai/hl		(E)-isomer	(Z)-isomer	Total	
Strawberry	24%	1		0.024	1	0.13	0.05	0.18	WKGR
Portugal	EC				4	0.05	0.02	0.07	0168.71
1971					7	0.03	0.01	0.04	
		1		0.048	1	0.26	0.08	0.34	
					4	0.08	0.04	0.12	
					7	0.06	0.03	0.09	
		1		0.024	1	0.08	0.03	0.11	
					4	0.03	0.01	0.04	
					7	0.04	0.01	0.05	
		1		0.048	1	0.09	0.04	0.13	
					4	0.03	0.02	0.05	
					7	0.04	0.02	0.06	
Strawberry	10%	1	0.22		2			0.06	RES
USA	EC				4			0.06	57-25[2,3]
1957					10			<0.05	
Strawberry	EC	1	1.1		0			0.26	RES
USA					1			0.14	62-23[2]
1962					2			0.24	
(aerial application)					3			0.14	
		1	1.4		0			0.62	
					1			0.45	
					2			0.28	
					3			<0.05	

[1]Sample storage conditions not clear
[2]Analyses by enzymatic method
[3]Sample storage period not clear.

Table 17. Residues of mevinphos in brassica vegetables.

Country, crop, year	Application				PHI, days	Residues, mg/kg[1]			Reference
	Form.	No.	kg ai/ha	kg ai/hl		(E)- isomer	(Z)- isomer	Total	
Broccoli		1	1.1		1			1.6	RES
USA	EC				2			0.12	62-27[2]
1965					3			0.08	
					4			<0.01	
		1	1.7		1			2.3	
					2			1.8	
					3			0.14	
					4			<0.01	
Cauliflower	10%	1	0.25	0.025	0	0.52, 0.38	0.55, 0.40	1.07, 0.78	BEGR
France	EC				1	0.46, 0.50	0.55, 0.52	1.01, 1.02	0016/70[3]
1969					2	0.32, 0.18	0.35, 0.20	0.67, 0.38	
					4	0.04, <0.02	0.15, 0.07	0.19, 0.09	
					7	0.02, <0.02	0.05, 0.05	0.07, 0.07	
		1	0.50	0.050	0	0.80, 0.80	0.75, 0.70	1.55, 1.50	
					1	0.58, 0.60	0.55, 0.65	1.13, 1.25	
					2	0.45, 0.40	0.50, 0.42	0.95, 0.82	
					4	0.04, 0.06	0.10, 0.20	0.14, 0.26	
					7	0.02, <0.02	0.05, 0.02	0.07, 0.04	
Cauliflower	50%	3		0.025	0	0.15	0.08	0.23	BEGR

Country, crop, year	Application				PHI, days	Residues, mg/kg[1]			Reference
	Form.	No.	kg ai/ha	kg ai/hl		(E)- isomer	(Z)- isomer	Total	
Germany	EC				4	0.04	0.03	0.07	0116.74
1974					7	<0.01	<0.01	<0.02	
					10	<0.01	<0.01	<0.02	
		3		0.025	0	0.06	0.10	0.16	
					4	0.02	0.08	0.10	
					7	<0.01	0.05	0.06	
					10	<0.01	<0.01	<0.02	
		3		0.025	0	0.10	0.07	0.17	
					4	0.05	0.05	0.10	
					7	0.01	0.03	0.04	
					10	<0.01	<0.01	<0.02	
Cauliflower	48%	1	1.1		0	0.04	0.02	0.06	WKGR
USA 1972	EC				2	<0.01	<0.01	<0.02	0073.72
(aerial application)									
Brussels	15%	9		0.011	0	<0.02	<0.02	<0.04	SBGR
sprout	EC				1	<0.02	<0.02	<0.04	81.224
South Africa					2	<0.02	<0.02	<0.04	
1980					4	<0.02	<0.02	<0.04	
					8	<0.02	<0.02	<0.04	
					16	<0.02	<0.02	<0.04	
Cabbage		2		0.025	0	0.82	0.44	1.26	WKGR
Germany					4	0.01	0.02	0.03	0014.75
1975					7	0.01	0.01	0.02	
					10	<0.01	<0.01	<0.02	
					14	<0.01	<0.01	<0.02	
					21	0.01	0.01	0.02	
		2		0.025	0	0.08	0.08	0.16	
					4	<0.01	0.01	0.02	
					7	<0.01	<0.01	<0.02	
					10	<0.01	<0.01	<0.02	
					14	<0.01	<0.01	<0.02	
					21	0.01	0.02	0.03	
		2		0.025	0	0.33	0.16	0.49	
					4	0.01	0.03	0.04	
					7	<0.01	0.02	0.03	
					10	<0.01	0.02	0.03	
					21	<0.01	<0.01	<0.02	
Cabbage	48%	2	0.43	0.072	0	1.70	0.75	2.45	BEGR
Germany	EC				4	0.02	0.02	0.04	82.128
1982					7	<0.01	<0.01	<0.02	
					10	<0.01	<0.01	<0.02	
Cabbage	48%	2	0.43	0.072	0	3.00	1.30	4.30	BETR
Germany	EC				4	0.02	0.02	0.04	84.002
1983					7	<0.01	<0.01	<0.02	
					10	<0.01	<0.01	<0.02	
		2	0.43	0.072	0	1.90	0.56	2.46	
					4	0.03	0.02	0.05	
					7	<0.01	<0.01	<0.02	
					10	<0.01	<0.01	<0.02	
Cabbage	TG	1	0.28	0.05	0			0.40	WK
UK					1			0.20	138/60[2,3,5]
1960					2			0.15	
					4			0.04	
					7			0.02	

Country, crop, year	Application				PHI, days	Residues, mg/kg[1]			Reference
	Form.	No.	kg ai/ha	kg ai/hl		(E)- isomer	(Z)- isomer	Total	
		1	0.56	0.10	0			0.85	
					1			0.55	
					2			0.13	
					4			0.02	
					7			0.02	
	10%	1	0.28	0.05	0			0.51	
	EC				1			0.25	
					2			0.23	
					4			<0.01	
					7			<0.02	
		1	0.56	0.10	0			0.95	
					1			0.64	
					2			0.20	
					4			0.05	
					7			<0.02	
	24%	1	0.28	0.05	0			0.55	
	EC				1			0.15	
					2			0.07	
					4			0.05	
					7			<0.02	
		1	0.56	0.10	0			0.83	
					1			0.52	
					2			0.11	
					4			<0.01	
					7			<0.02	
Cabbage	50%	1	0.25		1	<0.01	<0.01	H<0.02	WKGR
UK	WP				2	<0.01	<0.01	H<0.02	0180.71
1970					4	<0.01	<0.01	H<0.02	
					7	<0.01	<0.01	H<0.02	
		1	0.50		1	0.01,0.45	<0.01,0.19	H0.02,L0.64	
					2	0.02,0.14	<0.01,0.08	H0.03,L0.22	
					4	<0.01	<0.01	H<0.02	
					7	<0.01	<0.01	H<0.02	
Cabbage	24%	1	0.25		0	0.25	0.15	0.40	WKGR
UK	SL				1	0.01	0.03	0.04	0006.73
1972					3	<0.01	<0.01	<0.02	
					5	<0.01	<0.01	<0.02	
		1	0.5		0	0.60	0.30	0.90	
					1	0.03	0.05	0.08	
					3	0.02	0.02	0.04	
					5	0.01	0.01	0.02	
	24%	1	0.25		0	0.25	0.15	0.40	
	EC				1	<0.01	0.03	0.04	
					3	<0.01	<0.01	<0.02	
					5	<0.01	<0.01	<0.02	
	24%	5	0.25		0	0.55	0.25	0.80	
	SL				1	0.10	0.09	0.19	
					3	0.02	0.02	0.04	
					5	0.01	0.01	0.02	
		5	0.5		0	1.45	0.60	2.05	
					1	0.10	0.10	0.20	
					3	0.08	0.06	0.14	
					5	0.03	0.03	0.06	
					8	<0.01	<0.01	<0.02	
	24%	5	0.25		0	0.30	0.20	0.50	

Country, crop, year	Application				PHI, days	Residues, mg/kg[1]			Reference
	Form.	No.	kg ai/ha	kg ai/hl		(E)- isomer	(Z)- isomer	Total	
	EC				1	0.06	0.06	0.12	
					3	0.02	0.03	0.05	
					5	0.01	0.01	0.02	
Kale	50%	3		0.025	0	1.00	0.60	1.60	BEGR
Germany	EC				4	0.04	0.08	0.12	0115.74
1974					7	0.04	0.05	0.09	
					10	0.03	0.04	0.07	
					14	<0.01	<0.01	<0.02	
		3		0.025	0	2.40	0.80	3.20	
					4	0.08	0.12	0.20	
					7	0.01	0.03	0.04	
					10	<0.01	0.03	0.04	
					14	<0.01	<0.01	<0.02	
		3		0.025	0	2.20	1.00	3.20	
					4	0.02	0.06	0.08	
					7	<0.01	0.02	0.03	
					10	<0.01	0.01	0.02	
					14	<0.01	<0.01	<0.02	
Kale		3	0.71	0.079	0	1.80	0.90	2.70	BEGR
Germany					4	0.02	0.10	0.12	0027.75
1974					7	<0.01	0.04	0.05	
					10	<0.01	0.02	0.03	
					14	<0.01	<0.01	<0.02	
		3	0.47	0.078	0	1.60	0.70	2.30	
					4	0.17	0.18	0.35	
					7	0.02	0.08	0.10	
					10	<0.01	<0.01	<0.02	
					14	<0.01	<0.01	<0.02	
		3	0.71	0.079	0	1.90	0.80	2.70	
					4	<0.01	0.05	0.06	
					7	<0.01	0.03	0.04	
					10	<0.01	0.02	0.03	
					14	<0.01	<0.01	<0.02	
Kale	48%	2	0.43	0.072	0	1.10	0.65	1.75	BEGR
Germany	EC				4	<0.01	0.07	0.08	83.017
1982					7	<0.01	0.02	0.03	
					10	<0.01	<0.01	<0.02	
Kale	48%	2	0.43	0.072	0	9.60	2.80	12.40	BETR
Germany	EC				4	0.33	0.15	0.48	84.015
1983					7	<0.01	0.03	0.04	
					10	<0.01	<0.01	<0.02	
		2	0.43	0.072	0	6.00	1.40	7.40	
					4	0.30	0.30	0.60	
					7	0.06	0.10	0.16	
					10	<0.01	<0.01	<0.02	
Chinese Kale	24%	1	0.45		0			6.40	Submitted
Thailand	EC				1			1.80	by
1987					3			<u>0.22</u>	Thailand
					5			0.08	
					7			0.12	
					10			0.19	
		1	0.89		0			19.45	
					1			3.18	
					3			0.19	
					7			0.12	

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Country, crop, year	Application				PHI, days	Residues, mg/kg[1]			Reference
	Form.	No.	kg ai/ha	kg ai/hl		(E)- isomer	(Z)- isomer	Total	
					10			0.08	

[1]H= head; L = leaf
[2]Acnalyses by enzymatic method
[3]Sample storage conditions not clear
[4]Technical grade active ingredient was used
[5]Sample storage period not clear

Table 18. Residues of mevinphos in fruiting vegetables

Crop, country, year	Application				PHI, days	Residues, mg/kg[1]			Ref.
	Form.	No.	kg ai/ha	kg ai/hl		(*E*)-	(*Z*)-	Total	
Cucumber	50%	3		0.025	0	0.05	0.03	0.08	BEGR
Germany	EC				4	<0.01	<0.01	<0.02	0018.75
1974					7	<0.01	<0.01	<0.02	
					10	<0.01	<0.01	<0.02	
		3		0.025	0	0.08	0.04	0.12	
					4	<0.01	<0.01	<0.02	
					7	<0.01	<0.01	<0.02	
					10	<0.01	<0.01	<0.02	
		3		0.025	0	0.10	0.05	0.15	
					4	<0.01	<0.01	<0.02	
					7	<0.01	<0.01	<0.02	
					10	<0.01	<0.01	<0.02	
Cucumber	24%	1		0.05	0	0.12	0.05	0.17	BEGR
Netherlands	EC				1	0.09	0.04	0.13	0015.7[2]
1970					2	0.09	0.04	0.13	
					4	0.06	0.05	0.11	
					7	0.02	0.03	0.05	
					10	<0.01	<0.01	<0.02	
		1		0.1	0	0.19	0.06	0.25	
					1	0.19	0.07	0.26	
					2	0.14	0.06	0.20	
					4	0.08	0.05	0.13	
					7	0.03	0.04	0.07	
					10	<0.01	0.02	0.03	
Cucumber	22%	1		0.011	0	<0.01	<0.01	<0.02	BEGR
Netherlands	EC				1	<0.01	<0.01	<0.02	0004.71[2]
1970					2	<0.01	<0.01	<0.02	
					4	<0.01	<0.01	<0.02	
					7	<0.01	<0.01	<0.02	
					10	<0.01	<0.01	<0.02	
		1		0.023	0	0.15	0.08	0.23	
					1	0.09	0.04	0.13	
					2	0.04	0.03	0.07	
					4	0.03	0.04	0.07	
					7	<0.01	<0.01	<0.02	
					10	<0.01	<0.01	<0.02	
Cucumber	50%	3		0.025	4	0.32	0.08	0.40	BEGR
(glasshouse)	EC				7	0.15	0.05	0.20	0003.75
Germany					11	0.15	0.05	0.20	
1974					14	0.07	0.04	0.11	
(Sep.-Oct.)		3		0.025	0	0.50	0.15	0.65	
					4	0.45	0.15	0.60	

Crop, country, year	Application				PHI, days	Residues, mg/kg[1]			Ref.
	Form.	No.	kg ai/ha	kg ai/hl		(*E*)-	(*Z*)-	Total	
					7	0.30	0.12	0.42	
					11	0.16	0.07	0.23	
					14	0.07	0.08	0.15	
		3		0.025	0	0.35	0.10	0.45	
					4	0.25	0.08	0.33	
					7	0.18	0.07	0.25	
					11	0.11	0.05	0.16	
					14	0.05	0.05	0.10	
Cucumber	48%	3	0.14	0.023	0	0.02	0.06	0.08	BEGR
(glasshouse)	EC				1	0.02	0.04	0.06	82.123
Germany					3	<0.01	0.03	<u>0.04</u>	
1982					5	<0.01	0.03	0.04	
(Jun.-Jul.)					7	<0.01	0.01	0.02	
Cucumber	48%	3	0.29	0.032	0	0.06	0.04	0.10	BETR
(glasshouse)	EC				1	0.02	0.02	0.04	84.012
Germany					4	<0.01	0.01	0.02	
1983					7	<0.01	<0.01	<0.02	
(Jun., Aug.)					10	<0.01	<0.01	<0.02	
		3	0.29	0.032	0	0.10	0.04	0.14	
					1	0.05	0.04	0.09	
					4	0.04	0.03	0.07	
					7	0.02	0.02	0.04	
					10	<0.01	<0.01	<0.02	
Melon	TG	6	1.12	0.30	0			p0.46, r3.3	RES
USA					1			p0.35, r1.8	57-17[4]
1957					3			p0.28, r0.61	
					5			p0.10, r0,21	
Melon		1	0.84		0			0.05, p<0.02	RES
USA 1958					1			0.03, p<0.02	58-21[4]
(aerial application)									4)
Melon		1	0.56		0			(<0.02) 4	RES
USA								r(<0.02) 4	58-27[2,4]
1958					1			(<0.02) 3	
								r(<0.02) 3	
Watermelon	48%	1	0.56		0			0.03, r0.12	RES
USA	EC				1			0.02, r0.08	58-46[2,4,5]
1958					3			<0.02, r0.05	
Tomato	24%	1		0.05	0	0.07	0.08	0.15	BEGR
Belgium	EC				1	0.03	0.05	0.08	0012.70[2]
1969					2	0.01	0.02	0.03	
					4	<0.01	0.02	0.03	
					7	<0.01	0.02	0.03	
					10	<0.01	<0.01	<0.02	
		1		0.10	0	0.15	0.08	0.23	
					1	0.07	0.08	0.15	
					2	0.06	0.07	0.13	
					4	0.05	0.07	0.12	
					7	0.02	0.06	0.08	

Crop, country, year	Application				PHI, days	Residues, mg/kg[1]			Ref.
	Form.	No.	kg ai/ha	kg ai/hl		(*E*)-	(*Z*)-	Total	
					10	0.02	0.06	0.08	
		1		0.05	0	0.05	0.06	0.11	
					1	0.01	0.02	0.03	
					2	<0.01	0.02	0.03	
					4	<0.01	0.02	0.03	
					7	<0.01	<0.01	<0.02	
					10	<0.01	<0.01	<0.02	
		1		0.10	0	0.11	0.09	0.20	
					1	0.03	0.05	0.08	
					2	0.03	0.06	0.09	
					4	0.02	0.06	0.08	
					7	0.02	0.04	0.06	
					10	0.02	0.0-4	0.06	
Tomato	10%	1	0.5	0.05	0	<0.02	0.02	0.04	BEGR
France	EC				0	<0.02	0.02	0.04	0017.70[2]
1969					2	<0.02	0.02	0.04	
					4	<0.02	0.02	0.04	
					7	<0.02	0.02	0.04	
		1	1.0	0.10	0	0.12	0.19	0.31	
					0	0.05	0.15	0.20	
					2	0.02	0.10	0.12	
					4	<0.02	0.08	0.10	
					7	<0.02	0.05	0.07	
Tomato	50%	2		0.025	0	0.02	0.02	0.04	WKGR
Germany	EC				4	<0.01	<0.01	<0.02	0040.75
1975					7	<0.01	<0.01	<0.02	
					10	<0.01	<0.01	<0.02	
		3		0.025	0	<0.01	<0.01	<0.02	
					4	<0.01	<0.01	<0.02	
					7	<0.01	<0.01	<0.02	
					10	<0.01	<0.01	<0.02	
		2		0.025	0	0.01	0.02	0.03	
					4	0.01	0.01	0.02	
					7	<0.01	<0.01	<0.02	
					10	<0.01	<0.01	<0.02	
Tomato	24%	1		0.012	0	0.05	0.04	0.09	BEGR
(glasshouse)	EC				1	<0.01	0.01	0.02	0002.71[2]
Belgium					2	<0.01	<0.01	<0.02	
1970					4	<0.01	<0.01	<0.02	
					7	<0.01	<0.01	<0.02	
					10	<0.01	<0.01	<0.02	
		1		0.024	0	0.20	0.10	0.30	
					1	0.03	0.02	0.05	
					2	<0.01	0.01	0.02	
					4	<0.01	<0.01	<0.02	
					7	<0.01	<0.01	<0.02	
					10	<0.01	<0.01	<0.02	
Tomato	50%	3		0.025	0	0.03	0.04	0.07	WKGR
(glasshouse)	EC				4	<0.01	<0.01	<0.02	0040.75
Germany					7	<0.01	<0.01	<0.02	
1975					10	<001	<0.01	<0.02	
					14	<0.01	<0.01	<0.02	
		3		0.025	0	0.12	0.07	0.19	
					4	0.02	0.02	0.04	
					7	0.02	0.02	0.04	

Crop, country, year	Application				PHI, days	Residues, mg/kg[1]			Ref.
	Form.	No.	kg ai/ha	kg ai/hl		(*E*)-	(*Z*)-	Total	
					10	0.01	0.02	0.03	
					14	0.01	0.01	0.02	
		3		0.025	0	0.04	0.03	0.07	
					4	0.01	0.01	0.02	
					7	0.01	0.02	0.03	
					10	<0.01	<0.01	<0.02	
					14	<0.01	<0.01	<0.02	
Tomato	48%	3	0.14	0.023	0	0.02	0.10	0.12	BEGR
(glasshouse)	EC				1	<0.01	0.04	0.05	82.129
Germany					3	<0.01	<0.01	<0.02	
1982					5	<0.01	<0.01	<0.02	
					7	<0.01	<0.01	<0.02	
Tomato	22%	1		0.011	0	0.02	0.02	0.04	BEGR
(glasshouse)	EC				1	<0.01	<0.01	<0.02	0003.71[2]
Netherlands					2	<0.01	<0.01	<0.02	
1970					4	<0.01	<0.01	<0.02	
					7	<0.01	<0.01	<0.02	
					10	<0.01	<0.01	<0.02	
		1		0.023	0	0.05	0.03	0.08	
					1	0.02	0.03	0.05	
					2	<0.01	0.02	0.03	
					4	<0.01	<0.01	<0.02	
					7	<0.01	<0.01	<0.02	
					10	<0.01	<0.01	<0.02	
Baby corn	24%	1	0.14		0			ND[b]	Thailand
Thailand	EC				1			ND	
1989					3			ND	
					5			ND	
					7			ND	
					10			ND	
		1	0.28		0			ND	
					1			ND	
					3			ND	
					5			ND	
					7			ND	
					10			ND	

[1]p = pulp; r = peel
[2]Sample storage conditions not clear
[3]Technical grade active ingredient used
[4]Analyses by enzymatic method
[5]Sample storage period not clear
[6]Level of ND was not reported

Table 19. Residues of mevinphos in lettuce.

Country, year	Application				PHI, days	Residues, mg/kg			Ref.
	Form.	No.	kg ai/ha	kg ai/hl		(*E*)- isomer	(*Z*)- isomer	Total	
Belgium	24%	1		0.05	0	6.20	3.40	9.60	BEGR
1970	EC				1	1.70	1.10	2.80	0013.70[1]
					2	0.78	0.80	1.58	
					4	0.45	0.60	1.05	
					7	0.07	0.50	0.57	
					10	0.03	0.50	0.53	
		1		0.1	0	9.80	5.00	14.80	

Country, year	Application				PHI, days	Residues, mg/kg			Ref.
	Form.	No.	kg ai/ha	kg ai/hl		(*E*)- isomer	(*Z*)- isomer	Total	
					1	3.50	2.10	5.60	
					2	2.00	1.70	3.70	
					4	1.00	1.40	2.40	
					7	0.26	1.20	1.46	
					10	0.06	0.90	0.96	
Belgium	24%	1		0.012	0	1.80	0.75	2.55	BEGR
1970	EC				1	0.60	0.65	1.25	0001.71[1]
					2	0.15	0.25	0.40	
					4	0.05	0.10	0.15	
					7	0.02	0.03	0.05	
					10	<0.01	<0.01	<0.02	
		1		0.024	0	3.70	1.80	5.50	
					1	2.00	1.20	3.20	
					2	0.30	0.30	0.60	
					4	0.15	0.45	0.60	
					7	0.03	0.07	0.10	
					10	<0.01	<0.01	<0.02	
France	10%	1	0.5		1	1.00	1.50	2.50	BEGR
1970	EC				3	0.40	0.85	1.25	0051.70
					5	0.05	0.60	0.65	
					7	<0.01	0.20	0.21	
		1	1.0		1	2.40	3.00	5.40	
					3	0.95	2.30	3.25	
					5	0.10	1.00	1.10	
					7	0.03	0.30	0.33	
		1	0.5		1	0.90	1.20	2.10	
					3	0.35	1.00	1.35	
					5	0.10	0.80	0.90	
					7	0.05	0.30	0.35	
		1	1.0		1	2.70	3.00	5.70	
					3	0.60	1.90	2.50	
					5	0.15	1.10	1.25	
					7	0.05	0.50	0.55	
Germany	50%	3		0.025	0	1.40	1.60	3.00	BEGR
1974	EC				4	0.03	0.35	0.38	0089.74
					7	0.01	0.20	0.21	
					10	<0.01	0.09	0.10	
					14	<0.01	0.04	0.05	
					18	<0.01	<0.01	<0.02	
Germany	48%	3	0.14	0.023	0	0.15	0.15	0.30	BEGR
1982	EC				4	0.02	0.03	0.05	82.127
					7	<0.01	<0.01	<0.02	
					10	<0.01	<0.01	<0.02	
		2	0.43	0.072	0	0.75	0.65	1.40	
					4	0.10	0.10	0.20	
					7	<0.01	0.05	0.06	
					10	<0.01	<0.01	<0.02	
Germany	48%	2	0.43	0.072	0	4.50	2.40	6.90	BETR
1983	EC				4	0.50	0.16	0.66	84.017
					7	<0.01	0.01	0.02	
					10	<0.01	<0.01	<0.02	
		3	0.16	0.072	0	0.93	0.57	1.50	
					4	<0.01	0.05	0.06	
					7	<0.01	<0.01	<0.02	
					10	<0.01	<0.01	<0.02	
		2	0.43	0.072	0	1.60	0.60	2.20	

Country, year	Application				PHI, days	Residues, mg/kg			Ref.
	Form.	No.	kg ai/ha	kg ai/hl		(*E*)- isomer	(*Z*)- isomer	Total	
					4	0.40	0.43	0.83	
					7	0.04	0.28	0.32	
					10	<0.01	0.05	0.06	
Netherlands	24%	1		0.05	0	1.30	0.59	1.89	BEGR
1970	EC				1	0.42	0.26	0.68	0014.70[1]
					2	0.27	0.18	0.45	
					4	0.25	0.20	0.45	
					7	0.10	0.10	0.20	
					10	0.10	0.10	0.20	
		1		0.10	0	4.40	1.80	6.20	
					1	1.50	0.79	2.29	
					2	0.68	0.47	1.15	
					4	0.50	0.40	0.90	
					7	0.39	0.36	0.75	
					10	0.28	0.31	0.59	
Spain	50%	4	0.16	0.024	2	0.26	0.21	0.47	WKGR
1971	WP								0163.71
UK	50%	1	0.25		1	0.15	0.20	0.35	WKGR
1970	WP				2	0.03	0.10	0.13	0179.71
					4	<0.01	0.08	0.09	
					7	<0.01	0.03	0.04	
		1	0.50		1	0.20	0.30	0.50	
					2	0.05	0.25	0.30	
					4	0.01	0.20	0.21	
					7	<0.01	0.03	0.04	
	24%	1	0.1	0.013	0	0.70	0.35	1.05	BEGR
Belgium	EC				1	0.50	0.35	0.85	0019.72
(glasshouse)					2	0.25	0.25	0.50	
1971					4	0.20	0.25	0.45	
(Dec.)					7	0.05	0.25	0.30	
					10	0.03	0.15	0.18	
					14	<0.01	0.15	0.16	
		1	0.2	0.025	0	1.80	0.40	2.20	
					1	0.80	0.70	1.50	
					2	0.35	0.55	0.90	
					4	0.25	0.45	0.70	
					7	0.20	0.45	0.65	
					10	0.15	0.35	0.50	
					14	<0.01	0.35	0.36	
		1	0.1	0.013	0	0.90	0.40	1.30	
					1	0.60	0.50	1.10	
					2	0.20	0.20	0.40	
					4	0.20	0.20	0.40	
					7	0.07	0.25	0.32	
					10	<0.01	0.10	0.11	
					14	<0.01	0.10	0.11	
		1	0.2	0.025	0	2.40	1.00	3.40	
					1	0.80	0.50	1.30	
					2	0.40	0.35	0.75	
					4	0.25	0.35	0.60	
					7	0.10	0.30	0.40	
					10	0.05	0.30	0.35	
					14	<0.01	0.35	0.36	
Belgium		1		0.012	0	0.85,0.67	0.35,0.35	L1.20,H1.02	BEGR
(glasshouse)					1	0.40,0.35	0.20,0.25	L0.60,H0.60	0026.71

Country, year	Application				PHI, days	Residues, mg/kg			Ref.
	Form.	No.	kg ai/ha	kg ai/hl		(*E*)- isomer	(*Z*)- isomer	Total	
1971					2	0.30,0.20	0.20,0.20	L0.50,H0.40	
(Dec.)					4	0.20,0.08	0.20,0.20	L0.40,H0.28	
					7	0.10,0.05	0.20,0.20	L0.30,H0.25	
					10	0.04,<0.01	0.20,0.20	L0.24,H0.21	
					14	0.02,<0.01	0.15,0.12	L0.17,H0.13	
		1		0.024	0	1.90,0.90	0.80,0.40	L2.70,H1.30	
					1	0.90,0.40	0.50,0.30	L1.40,H0.70	
					2	0.80,0.40	0.50,0.45	L1.30,H0.85	
					4	0.30,0.20	0.50,0.40	L0.80,H0.60	
					7	0.30,0.08	0.50,0.35	L0.80,H0.43	
					10	0.10,0.05	0.40,0.35	L0.50,H0.40	
					14	0.06,<0.01	0.35,0.28	L0.41,H0.29	
		1		0.012	0	0.47,0.47	0.28,0.25	L0.75,H0.72	
					1	0.30,0.28	0.20,0.25	L0.50,H0.53	
					2	0.22,0.24	0.18,0.29	L0.40,H0.53	
					4	0.12,0.17	0.17,0.25	L0.29,H0.42	
					7	0.07,0.03	0.15,0.15	L0.22,H0.18	
					10	0.05,0.02	0.15,0.15	L0.20,H0.17	
					14	<0.02,<0.01	0.09,0.09	L0.11,H0.10	
		1		0.024	0	2.00,1.90	0.70,0.62	L2.70,H2.52	
					1	1.10,0.76	0.55,0.45	L1.65,H1.21	
					2	0.70,0.63	0.40,0.40	L1.10,H1.03	
					4	0.65,0.52	0.45,0.40	L1.10,H0.92	
					7	0.30,0.24	0.45,0.38	L0.75,H0.62	
					10	0.18,0.10	0.45,0.38	L0.63,H0.48	
					14	0.07,0.03	0.45,0.30	L0.52,H0.33	
Germany	50%	2		0.025	0	14.00	7.00	21.00	BEGR
(glasshouse)	EC				4	0.30	2.40	2.70	0090.74
1974					7	0.05	1.00	1.05	
					14	<0.01	0.20	0.21	
					21	<0.01	<0.01	<0.02	
		2		0.025	0	7.80	4.30	12.10	
					4	0.55	1.30	1.85	
					7	0.06	0.55	0.61	
					14	<0.01	0.05	0.06	
					17	<0.01	<0.01	<0.02	
Germany	50%	3		0.025	0	1.60	1.10	2.70	WKGR
(glasshouse)	EC				4	0.08	0.43	0.51	0020.75
1974					7	0.06	0.30	0.36	
					9	0.01	0.23	0.24	
					14	0.01	0.07	0.08	
					21	<0.01	0.01	0.02	
Germany	53%	3	0.16	0.027	0	0.40	0.40	0.80	BEGR
(glasshouse)	EC				4	0.02	0.15	0.17	82.126
1982					7	<0.01	0.10	0.11	
					10	<0.01	0.03	0.04	
Netherlands	24%	1		0.012	0	0.15	0.10	0.25	BEGR
(glasshouse)	EC				1	0.03	0.06	0.09	0007.71[1]
1970					2	0.02	0.05	0.07	
(Sep.)					4	0.02	0.05	0.07	
					7	<0.01	0.05	0.06	
					10	<0.01	0.03	0.04	
		1		0.024	0	0.45	0.25	0.70	
					1	0.15	0.17	0.32	
					2	0.12	0.15	0.27	
					4	0.12	0.15	0.27	

Country, year	Application				PHI, days	Residues, mg/kg			Ref.
	Form.	No.	kg ai/ha	kg ai/hl		(*E*)- isomer	(*Z*)- isomer	Total	
					7	0.07	0.15	0.22	
					10	0.01	0.10	0.11	
Netherlands	22%	1		0.011	0	4.00	1.70	5.70	BEGR
(glasshouse)	EC				1	2.30	1.00	3.30	0025.71
1970					2	1.10	0.90	2.00	
(Dec.)					4	0.85	0.85	1.70	
(Dec.)					7	0.20	0.65	0.85	
					10	0.07	0.55	0.62	
					14	0.03	0.43	0.46	
		1		0.022	0	7.40	3.50	10.90	
					1	4.30	2.40	6.70	
					2	1.30	0.90	2.20	
					4	0.80	0.65	1.45	
					7	0.30	0.80	1.10	
					10	0.10	0.40	0.50	
					14	0.05	0.45	0.50	
		1		0.011	0	3.40	1.40	4.80	
					1	2.20	1.10	3.30	
					2	1.50	0.90	2.40	
					4	1.00	0.75	1.75	
					6	0.35	0.37	0.72	
					10	0.13	0.30	0.43	
					14	0.04	0.37	0.41	
		1		0.022	0	5.30	2.20	7.50	
					1	3.20	1.50	4.70	
					2	1.70	1.10	2.80	
					4	1.30	1.50	2.80	
					6	0.40	0.85	1.25	
					10	0.15	0.63	0.78	
					14	0.10	0.39	0.49	
	48%	3	0.56		0			1.0	RES
	EC				1			0.94	65-44[2]
USA					3			0.72	
(glasshouse)					7			0.03	
1965					10			<0.02	
		3	1.1		0			1.1	
					1			1.0	
					3			0.98	
					7			0.04	
					10			0.02	
		3	0.56		0			1.2	
					1			1.0	
					3			0.53	
					5			0.27	
					7			0.06	
					10			<0.02	
		3	1.1		0			1.4	
					1			1.1	
					3			0.90	
					5			0.54	
					7			0.08	
					10			<0.02	

[1]Sample storage conditions not clear
[2]Analyses by enzymatic method

Table 20. Residues of mevinphos in spinach.

Country, year	Application				PHI days	Residues, mg/kg			Ref.
	Form.	No.	kg ai/ha	kg ai/hl		(*E*)- isomer	(*Z*)- isomer	Total	
Germany	50%	3		0.025	0	4.70	2.10	6.80	BEGR
1974	EC				4	0.04	0.15	0.19	120.74
					7	<0.01	0.02	<u>0.03</u>	
					10	<0.01	<0.01	<0.02	
					14	<0.01	<0.01	<0.02	
					20	<0.01	<0.01	<0.02	
		3		0.025	0	2.00	1.00	3.00	
					4	0.10	0.22	0.32	
					7	0.01	0.06	<u>0.07</u>	
					10	<0.01	0.02	0.03	
					14	<0.01	<0.01	<0.02	
		3		0.025	0	2.00	1.00	3.00	
					4	0.05	0.10	0.15	
					7	0.01	0.04	<u>0.05</u>	
					10	<0.01	<0.01	<0.02	
					14	<0.01	<0.01	<0.02	
South Africa	15%	1	0.11	0.022	0	0.54	0.27	0.81	BLGR
1980	EC				1	0.31	0.18	0.49	80.048
					3	<0.01	<0.01	<0.02	
UK		1		0.013	1			0.52	WK
1958					2			0.21	98/58[1]
					3			0.10	
					4			0.05	
					7			0.01	
		1		0.025	1			0.78	
					2			0.30	
					3			0.27	
					4			0.10	
					7			0.01	
					11			0.01	
		1		0.05	1			1.70	
					2			0.78	
					3			0.40	
					4			0.18	
					7			0.04	
					11			0.01	
Belgium	24%	1		0.1	2	6.0	2.7	8.7	WKGR
1972 (Glasshouse)	EC								0074.72

[1]Sample storage conditions and period not clear. Analyses by enzymatic method

Table 21. Residues of mevinphos in root vegetables.

Crop, Country, Year	Application				PHI, days	Residues, mg/kg			Reference
	Form.	No.	kg ai/ha	kg ai/hl		(*E*)- isomer	(*Z*)- isomer	Total	
Carrot	50%	2		0.025	0	0.02	0.01	0.03	BEGR
Germany	EC				4	<0.01	<0.01	<0.02	0112.74
1974					7	<0.01	<0.01	<u><0.02</u>	
					10	<0.01	<0.01	<0.02	
					14	<0.01	<0.01	<0.02	

Crop, Country, Year	Application				PHI, days	Residues, mg/kg			Reference
	Form.	No.	kg ai/ha	kg ai/hl		(*E*)- isomer	(*Z*)- isomer	Total	
		2		0.025	0	<0.01	<0.01	<0.02	
					4	<0.01	<0.01	<0.02	
					7	<0.01	<0.01	<0.02	
					10	<0.01	<0.01	<0.02	
					14	<0.01	<0.01	<0.02	
		3		0.025	0	<0.01	0.02	0.03	
					4	<0.01	<0.01	<0.02	
					7	<0.01	<0.01	<0.02	
					10	<0.01	<0.01	<0.02	
					14	<0.01	<0.01	<0.02	
Carrot		1	0.28		0			0.05	BIO
USA					1			<0.05	56-84[1]
1956					7			<0.05	
					14			<0.05	
Celeriac	48%	3	0.16	0.027	0	0.01	0.01	0.02	BETR
Germany	EC				3	<0.01	<0.01	<0.02	84.014
1983					4	<0.01	<0.01	<0.02	
					7	<0.01	<0.01	<0.02	
		3	0.16	0.027	0	0.08	0.03	0.11	
					3	<0.01	<0.01	<0.02	
					4	<0.01	<0.01	<0.02	
					7	<0.01	<0.01	<0.02	
		3	0.16	0.027	0	0.06	0.05	0.11	
					3	0.01	<0.01	0.02	
					4	<0.01	<0.01	<0.02	
					7	<0.01	<0.01	<0.02	
Turnip		1	0.28		0			<0.05	BIO
USA					1			<0.05	56-116[1,2]
1956					3			<0.05	
					4			<0.05	
		1	0.56		0			<0.05	
					1			<0.05	
					3			<0.05	
					4			<0.05	
Potato		1	0.28		0			<0.05	BIO
USA					1			<0.05	56-84[1]
1956					7			<0.05	

[1]Analyses by enzymatic method
[2]Sample storage conditions and period not clear

Table 22. Residues of mevinphos in bulb vegetables.

Crop, country, year	Application				PHI, days	Residues, mg/kg			Ref.
	Form.	No.	kg ai/ha	kg ai/hl		(E)- isomer	(Z)- isomer	Total	
Leek	50%	3		0.025	0	0.05	0.02	0.07	BEGR
Germany	EC				4	<0.01	<0.01	<0.02	0109.74
1974					7	<0.01	<0.01	<0.02	
					10	<0.01	<0.01	<0.02	
					14	<0.01	<0.01	<0.02	
		2		0.025	0	0.01	0.02	0.03	
					4	<0.01	<0.01	<0.02	
					7	<0.01	<0.01	<0.02	
					14	<0.01	<0.01	<0.02	
					21	<0.01	<0.01	<0.02	

Crop, country, year	Application				PHI, days	Residues, mg/kg			Ref.
	Form.	No.	kg ai/ha	kg ai/hl		(E)- isomer	(Z)- isomer	Total	
		2		0.025	0	0.09	0.07	0.16	
					4	<0.01	<0.01	<0.02	
					7	<0.01	<0.01	<0.02	
					10	<0.01	<0.01	<0.02	
					14	<0.01	<0.01	<0.02	
Leek	48%	3	0.14	0.023	0	0.85	0.35	1.20	BEGR
Germany	EC				4	0.01	<0.01	0.02	83.004
1982					7	<0.01	<0.01	<0.02	
					14	<0.01	<0.01	<0.02	
Leek	48%	3	0.16	0.027	0	0.01	0.02	0.03	BETR
Germany	EC				4	<0.01	<0.01	<0.02	84.013
1983					7	<0.01	<0.01	<0.02	
					10	<0.01	<0.01	<0.02	
Onion		1	0.28		0			0.40	BIO
USA					1			0.15	56-84[1]
1956					7			<0.05	
					14			<0.05	

[1]Analyses by enzymatic method

Table 23. Residues of mevinphos in pulses.

Crop, country, year	Application				PHI, days	Residues, mg/kg[1]			Ref.
	Form.	No.	kg ai/ha	kg ai/hl		(E)- isomer	(Z)- isomer	Total	
Bean	50%	3		0.025	0	0.10	0.19	0.29	BEGR
Germany	EC				4	<0.01	0.06	0.07	0110.74
1974					7	<0.01	0.02	0.03	
					10	<0.01	<0.01	<0.02	
					14	<0.01	<0.01	<0.02	
		3		0.025	0	0.02	0.08	0.10	
					4	<0.01	0.02	0.03	
					7	<0.01	<0.01	<0.02	
					10	<0.01	<0.01	<0.02	
					14	<0.01	<0.01	<0.02	
		3		0.025	0	0.19	0.18	0.37	
					4	<0.01	0.01	0.02	
					7	<0.01	<0.01	<0.02	
					10	<0.01	<0.01	<0.02	
					14	<0.01	<0.01	<0.02	
French Bean	TG[2]	1	0.28	0.05	0			0.02	WK
UK					1			0.02	137/60[3]
1960					2			<0.01	
		1	0.56	0.10	0			0.05	
					1			0.02	
					2			<0.01	
	10%	1	0.28	0.05	0			0.09	
	EC				1			0.02	
					2			<0.01	
		1	0.56	0.10	0			0.09	
					1			0.02	
					2			<0.01	
	24%	1	0.28	0.05	0			0.04	
	EC				1			0.01	
					2			<0.01	
		1	0.56	0.10	0			0.08	

Crop, country, year	Application				PHI, days	Residues, mg/kg[1]			Ref.
	Form.	No.	kg ai/ha	kg ai/hl		(E)- isomer	(Z)- isomer	Total	
					1			0.02	
					2			<0.01	
French Bean	50%	1	0.25		1	<0.01	0.02	0.03	WKGR
UK	WP				2	<0.01	<0.01	<0.02	0181.71
1971					4	<0.01	0.02	0.03	
(Aug.)					7	<0.01	<0.01	<u><0.02</u>	
		1	0.5		1	<0.01	0.03	0.04	
					2	<0.01	0.02	0.03	
					4	<0.01	<0.01	<0.02	
					7	<0.01	<0.01	<u><0.02</u>	
Bean	50%	3		0.025	0	<0.01	0.03	0.04	BEGR
(glasshouse)	EC				4	<0.01	<0.01	<0.02	0111.74
Germany					7	<0.01	<0.01	<u><0.02</u>	
1974					10	<0.01	<0.01	<0.02	
(Jul.)					14	<0.01	<0.01	<0.02	
		3		0.025	0	0.03	0.07	0.10	
					4	<0.01	0.03	0.04	
					7	<0.01	<0.01	<u><0.02</u>	
					9	<0.01	<0.01	<0.02	
					14	<0.01	<0.01	<0.02	
		3		0.025	0	0.07	0.13	0.20	
					4	<0.01	0.08	0.09	
					7	<0.01	0.04	0.05	
					11	<0.01	<0.01	<0.02	
					14	<0.01	<0.01	<0.02	
Green Bean	48%	3	0.14	0.023	0	0.07	0.09	0.16	BEGR
(glasshouse)	EC				3	<0.01	0.05	0.06	82.121
Germany					5	<0.01	0.04	0.05	
1982					7	<0.01	0.02	0.03	
(Jun.)					10	<0.01	<0.01	<0.02	
Pea	24%	2	0.17	0.015	2			<0.01	WKTR
South Africa	EC				4			<0.01	0034/69[3]
1969									
Pea		1		0.013	0			<0.02, (<0.02)	WK
UK					1			<0.02, (<0.02)	50/57[3]
1957					2			<0.02, (<0.02)	
		1		0.025	0			<0.02, (0.12)	
					1			<0.02, (<0.02)	
					2			<0.02, (<0.02)	
		1		0.05	0			<0.02, (0.34)	
					1			<0.02, (<0.02)	
					2			<0.02, (<0.02)	
Soya bean	24%	1	0.36	0.18	15	<0.01	<0.01	<0.02	BLGR
Brazil	EC				34	<0.01	<0.01	<0.02	79.096
1979					52	<0.01	<0.01	<0.02	
Peanut	24%	1	0.24		7	<0.01	<0.01	<0.02	BEGR
Brazil	EC				14	<0.01	<0.01	<0.02	80.170

Crop, country, year	Application				PHI, days	Residues, mg/kg[1]			Ref.
	Form.	No.	kg ai/ha	kg ai/hl		(E)- isomer	(Z)- isomer	Total	
1980					28	<0.01	<0.01	<0.02	
Peanut	24%	1	0.14		10			<0.02	WKTR
South Africa	EC	1	0.28		1			<0.02	0034/69[3]
1969					10			<0.02	

[1]Figures in the parentheses residues for peas with pod
[2]Technical grade active ingredient used
[3]Sample storage conditions and period not clear. Analyses by enzymatic method

Table 24. Residues of mevinphos in sugar beet.

Country, year	Application				PHI, days	Residues, mg/kg[1]			Ref.
	Form.	No.	Kg ai/ha	Kg ai/hl		(E)- isomer	(Z)- isomer	Total	
France		1	0.25		91	<0.01, (<0.01)	<0.01, (<0.01)		0106.75
1975		1	0.24		91	<0.01, (<0.01)	<0.01, (<0.01)	<0.02, (<0.02)	
		2	0.25		98	<0.01, (<0.01)	<0.01, (<0.01)	<0.02, (<0.02)	
		2	0.35		98	<0.01, (<0.01)	<0.01, (<0.01)	<0.02, (<0.02)	
Germany	50%	3		0.025	0	<0.01, (0.02)	<0.01, (0.09)	<0.02, (0.11)	WKGR
	EC				4	<0.01, (<0.01)	<0.01, (0.03)	<0.02, (0.04)	0017.75
1974					7	<0.01, (<0.01)	<0.01, (<0.01)	<0.02, (<0.02)	
					14	<0.01, (<0.01)	<0.01, (0.03)	<0.02, (0.04)	
		3		0.025	0	<0.01, (0.06)	<0.01, (0.19)	<0.02, (0.25)	
					4	<0.01, (<0.01)	<0.01, (0.03)	<0.02, (0.04)	
					7	<0.01, (<0.01)	<0.01, (0.02)	<0.02, (0.03)	
					14	<0.01, (<0.01)	<0.01, (<0.01)	<0.02, (<0.02)	

[1]Figures in parentheses are for leaves

FATE OF RESIDUES IN STORAGE AND PROCESSING

Storing, washing and cooking

Data on residues in red cabbage, broccoli, lettuce and spinach are shown in Table 25. Residues in peaches and strawberries after 2-3 days storage and in peaches after canning are shown in Table 26.

The effects of washing on residues in broccoli and cauliflower are shown in Table 27. The residues were determined by an enzymatic method.

The effects of peeling and cooking on residues in apples are shown in Table 28.

Table 25. Residues of mevinphos in vegetables after storing, washing and/or cooking. All single applications.

Crop	Red cabbage	Broccoli	Lettuce (glasshouse) Belgium (two sites) 1971		Spinach
Country	USA	USA			Belgium
Year	1972	1972			1972
Application	1.1 kg/ha	1.1 kg/ha	0.1 kg/ha	0.2 kg/ha	0.1 kg/hl
PHI	3 days	8 hours	14 days	14 days	2 days
Formulation	48% EC	48% EC	24% EC	24% EC	24% EC
Reference	WKGR 0063.72	WKGR 0072.72	BEGR 0019.72	BEGR 0019.72	WKGR 0074.72
Storage period, days	Residues (sum of (E)- and (Z)-mevinphos, mg/kg)				
0		0.27	0.11, 0.16	0.36, 0.36	8.70
0 (boiled)[1]					2.30
1	0.2		0.11, 0.16	0.36, 0.36	
2		0.09	0.06, 0.11	0.26, 0.31	5.20
2 (boiled)[1]		0.03			1.55
3	0.11				
3 (washed)[2]	0.03				
3 (boiled)[1]	<0.02				
4			0.06, 0.06	0.26, 0.21	2.70
4 (boiled)[1]					0.81
5-6	0.06	0.03			1.14
5-6 (washed)[2]	0.04				
5-6 (boiled)[1]	<0.02				0.36
7	0.03				
10	0.02	<0.02			

[1]Boiled for 20 minutes in salted water
[2]Washed under running water for 4 minutes

Table 26. Residues of mevinphos in peaches and strawberries after storing or canning. Single applications.

Crop	Peaches	Strawberries	
Country	South Africa	Portugal (two sites)	
Year	1972	1971	
Application	0.05 kg/hl	0.024 kg/hl	0.048 kg/hl
PHI	2 days	1 day	1 day
Formulation	24% EC	24% EC	24% EC
Reference	WKGR.0084.72	WKGR.0168.71	WKGR.0168.71
Storage period, days	Residues (sum of (E)- and (Z)-mevinphos, mg/kg)		
0	2.9	0.18, 0.11	0.34, 0.13
0 canned[1]	0.06		
2		0.16, 0.04	0.25, 0.08
3	2.7		

[1]The peel was removed by immersion for 20 seconds in boiling 2% sodium hydroxide. After removal of the stones, the peeled fruit were cut into portions and packed into cans. Sugar solution was added and the cans were cured for 10 minuets in a steam box. After sealing, the cans were heated at 100 C for 20 minutes.

Table 27. Residues of mevinphos in broccoli after washing and cauliflower. All single applications.

Crop	Broccoli	Broccoli	Cauliflower	Cauliflower
Country	USA	USA	USA	USA
Year	1965	1965	1962	1962
Application	1.1 kg/ha	1.7 kg/ha	1.1 kg/ha	1.7 kg/ha
PHI	1 day	1 day	0 day	0 day
Formulation	EC	EC	EC	EC
Reference	RES.62-27	RES.62-27	RES.62-26	RES.62-26
Initial residues, mg/kg	1.6	2.3	0.74	1.0
Residues after washing, mg/kg	0.72	0.86	0.52	0.08

[1]Analyses by enzymatic method. Details of washing were not reported

Table 28. Residues of mevinphos in apples after peeling and cooking, UK, 1971. Application: 1.0 kg technical grade ai/ha. Ref. WKGR. 0052-72.

PHI, days	Residues, mg/kg		
	3	7	10
Raw apples	1.15	0.65	0.60
Peeled apples	0.82	0.49	0.57
Peeled and cooked apples[1]	0.59	0.26	0.43

[1]Boiled for 10 minutes in water

Processing

Grapes. Data on the effects of processing to juice and raisins were submitted. In the field trial, a single application at 4.8 kg/ha was made and the grapes were harvested after five days. Processing into juice, wet and dry pomace, raisins and raisin waste followed commercial practice as far as possible.

To produce juice the grapes were washed with a high pressure spray for 30 seconds and then pressed with racks and cloths to give fresh juice. This method did not crush the stems or seeds. The pulp after pressing (wet pomace), consisting of seeds, skins and stems, was then dried in a bin air drier and the dry pomace fed into the pressmill for uniform milling.

Grapes for raisins were air-dried at 54-66°C for about 65 hours. After drying the raisins were de-stemmed by hand, instead of with a rotary screen and air flow as in industrial practice, and spray-washed for 15 seconds to remove any residual soil. The accumulated waste consisting of stems, stem caps and debris was fed into the pressmill to give uniform material. The results are shown in Table 29.

Table 29. Residues of mevinphos in processed fractions of grapes. USA, 1993.

Application, ref	Sample	Residues, mg/kg			Processing factor
		(*E*)- isomer	(*Z*)- isomer	Total	
1 x 4.8 kg/ha	Fresh grapes	0.067	0.092	0.159	
5 days PHI	Fresh juice	0.058	0.080	0.138	0.87
SARS-93-16	Wet pomace	0.089	0.094	0.183	1.15
	Dry pomace	0.022	0.031	0.053	0.33
	Raisins	<0.022	0.017	0.039	0.25
	Raisin waste	1.91	1.62	3.53	22.2

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

The government of The Netherlands reported monitoring data on residues of mevinphos in several crops to the Meeting. The results are summarized in Table 30.

Table 30. Monitoring data on mevinphos residues in several crops in The Netherlands in 1995.

Commodity	No. of samples	No. with residues[1]	% with residues	Mean residues, mg/kg[2]
Oranges	336	2	0.60	<0.05
Strawberries	1009	19	1.88	<0.05
Blackberries	57	3	5.26	<0.05
Lettuce	888	13	1.46	<0.05
Endive	343	6	1.75	<0.05
Parsley	142	1	0.70	<0.05
Other leafy vegetables	63	3	4.76	<0.05
Celery	76	3	3.95	<0.05
Other stem vegetables	172	6	3.49	<0.05

[1]LOD = 0.05 mg/kg
[2]For samples without residues, half the LOD is taken for the calculation of the mean

NATIONAL MAXIMUM RESIDUE LIMITS

The residue is defined as the sum of (*E*)- and (*Z*)-mevinphos in all the listed countries.

Country	Crop	MRL
Australia	Cotton seed	0.1
Australia	Edible offal (mammalian)	0.05*
Australia	Eggs	0.05*
Australia	Meat (mammalian)	0.05*
Australia	Milk	0.05*
Australia	Pome fruits	0.25
Australia	Poultry meat	0.05*
Australia	Poultry, Edible offal of	0.05*
Australia	Stone fruit	0.25
Australia	Vegetables	0.25
Austria	Apples	0.2
Austria	Apricots	0.2
Austria	Cabbages	0.1
Austria	Cherries	0.5
Austria	Grapes	0.1
Austria	Peaches	0.5
Austria	Pears	0.2
Austria	Plums	0.5
Austria	Sugar beet	0.05
Belgium	Apples	0.2
Belgium	Cucumbers	0.1
Belgium	Pears	0.2
Belgium	Peas	0.1
Belgium	Peppers	0.1
Belgium	Potato	0.1
Belgium	Tomatoes	0.1

Country	Crop	MRL
France	Almonds	0.5
France	Apples	0.2
France	Apricots	0.2
France	Beans	0.1
France	Beets	0.5
France	Cabbages	0.5
France	Corn salad	0.5
France	Courgettes	0.1
France	Cucumbers	0.1
France	Egg plants	0.1
France	Fodder beets	0.5
France	Garden cress	0.5
France	Gherkins	0.1
France	Grapes	0.1
France	Hazelnuts	0.1
France	Lettuce	0.5
France	Melons	0.1
France	Peaches	0.5
France	Peas	0.1
France	Peppers	0.1
France	Plums	0.5
France	Spinach	0.5
France	Strawberries	0.1
France	Tomatoes	0.1
Luxembourg	Apples	0.2
Luxembourg	Cucumbers	0.1
Luxembourg	Pears	0.2
Luxembourg	Peas	0.1
Luxembourg	Peppers	0.1
Luxembourg	Potato	0.1
Luxembourg	Tomatoes	0.1
Netherlands	Apricots	0.2
Netherlands	Citrus fruit	0.2
Netherlands	Fruit except citrus and stone fruit	0.1
Netherlands	Leafy vegetables	0.1
Netherlands	Pome fruits	0.2
Netherlands	Potato	0.1
Netherlands	Stone fruit except apricots	0.5
Netherlands	Vegetables	0.1
Portugal	Cucumbers	0.1
Portugal	Garlic	0.1
Portugal	Melons	0.1
Portugal	Tomatoes	0.1
Switzerland	Apples	0.3
Switzerland	Grapes	0.3
Switzerland	Pears	0.3
Switzerland	Peas	0.3

*at or about the LOD

APPRAISAL

Mevinphos is a systemic and contact organophosphate insecticide and acaricide used to protect a wide range of crops. It was first evaluated toxicologically in 1965 and for residues in 1972. Since the ADI was established before 1976, it is included in the CCPR periodic review programme (ALINORM 89/24A, para 299; Appendix V). The 1991 CCPR scheduled the review for 1996 (ALINORM 91/24A, para 316; Appendix IV, para 11), but the residue review was postponed to 1997 in 1995 (ALINORM 95/24A, Appendix IV).

The Meeting received information on animal and plant metabolism, environmental fate, analytical methods, updated GAP, supervised residue trials on fruits and vegetables, and residues after storage and processing.

Animal metabolism. The absorption, distribution, metabolism and excretion of mevinphos has been studied in rats, cows, goats and hens. Mevinphos is rapidly absorbed, metabolized and excreted.

Rats. Within 24 hours after oral and intravenous administration approximately 76% of the administered [^{14}C]mevinphos was eliminated as $^{14}CO_2$ and the cumulative excretion in exhaled air and urine accounted for 94% of the total dose in both sexes. Four radioactive peaks were observed in the urine: (*E*)-mevinphos, (*E*)-mevinphos acid, demethyl-(*E*)-mevinphos and unknown.

Cows. Lactating dairy cows were dosed by capsule with unlabelled or ^{32}P-labelled mevinphos at levels up to the equivalent of 20 ppm in the diet for 12 weeks. The milk, fat, liver, kidney, muscle, heart and brain contained less than 0.03 mg/kg mevinphos-equivalent anticholinestease activity at all dose levels throughout the dosing period. The level of organosoluble radioactivity in milk from a cow which received a single dose of [^{32}P]mevinphos (2.0 mg/kg bw) reached a maximum of 0.062 mg/kg mevinphos equivalents 6 hours after administration and decreased to below 0.007 mg/kg at 96-108 hours. The corresponding level in milk from a cow dosed for 7 successive days with [^{32}P]mevinphos at 1.0 mg/kg bw reached approximately 0.05 mg/kg after 6 h and maintained this level for the remainder of the 7 days. Elimination in the faeces and urine accounted for 77% of the [^{32}P] in the single dose and over half of this was excreted in the urine within the first 12 hours. A similar initial excretion was found with the cow dosed for 7 days.

Goats. Lactating dairy goats were treated with [^{14}C]mevinphos for 6 successive days at the equivalent of 18.0 or 2.9 ppm in the diet. Mevinphos was absorbed from the gastrointestinal tract and eliminated in the urine, mainly in the first 8 hours and decreasingly in the next 16 hours. The patterns of urinary elimination were similar for the low and high doses, and the urinary excretion of both doses was apparently complete within 24 hours. The average proportion of the dose excreted within 24 hours was 24.3% of the low dose and 38.7% of the high dose over the 6-day dosing period.

The faeces were a minor route of elimination with averages of 3.38% and 2.55% of the low and high doses respectively within 24 hours. Radioactivity appeared in the first milk collection, which was 8 hours after the first administration, at levels of 0.47 and 3.84 mg/kg mevinphos equivalent from the low and high doses respectively. By the following morning the levels had decreased to 0.21 and 0.52 mg/kg. The daytime/night-time elimination pattern persisted with the repeated dosing and the levels of eliminated radioactivity in the milk seemed to reach plateaus after the 4th dose. The analysis of milk and tissue fractions showed that the radioactivity was associated with normal endogenous components.

Hens. Laying hens were treated by capsule with [^{14}C]mevinphos for 3 successive days at 23 or 2.3 ppm on a daily feed intake basis. The level of radioactivity in the excreta was fairly constant over the three-day collection period and ranged from 23.0% to 29.6% and 38.5% to 43.1% of the daily dose for the birds in the low and high dose groups respectively. The radioactivity in the egg whites accumulated with repeated dosing in both groups, and reached 0.087 and 0.876 mg/kg after the third treatment in the low and high groups respectively. Radioactivity in the egg yolks was detected after the second administration in both dose groups and increased to 0.104 and 0.393 mg/kg in the low and high groups respectively after the third treatment. The analysis of egg yolk and tissue fractions showed that radioactivity was again associated with normal endogenous components.

Plant metabolism. Studies with lettuce, strawberries and turnips showed that mevinphos is metabolized via two pathways in all three plants. A small proportion of mevinphos is converted to mevinphos acid, while the major path involves cleavage of the P-O-C linkage leading to the formation of methyl acetoacetate. Methyl acetoacetate then undergoes reduction to generate methyl (*R,S*)-3-hydroxybutyrate, which was found conjugated to carbohydrates in plant tissues. The hydroxybutyrate and acetoacetate can undergo hydrolysis to 3-hydroxybutyric acid and acetoacetic acid respectively, which in turn can be conjugated with carbohydrates.

The fate of the phosphorus moiety was not determined because mevinphos was labelled on the carbon attached to the P-O group, but dimethyl phosphate would presumably be formed when the phosphate ester bond is broken.

Degradation in soil. Studies showed that under aerobic conditions mevinphos is degraded rapidly through the formation of methyl acetoacetate, finally to CO_2 or fragments which bind to fulvic acid, humic acid or humins.

Field dissipation studies to examine the mobility, degradation and dissipation of mevinphos in soil under field conditions showed that the mobility of both mevinphos isomers is minimal. Essentially no residues were detected in any soil layer one day to two months after application, indicating that the use of mevinphos in sandy soils with low organic matter content, which is the "worst case" for potential groundwater contamination, dose not present any risk. This is mainly due to the rapid degradation of both mevinphos isomers in the top six inches of the soil which prevents any further leaching.

Rotational crop studies were conducted to determine the uptake and nature of the residues in plants following the application of [^{14}C]mevinphos to the soil. Thirty two days after application, lettuce, sugar beet and sorghum were planted and harvested at maturity. All samples contained <0.01 mg/kg mevinphos equivalents.

The trials were carried out with several types of formulation (e.g. 10-50% EC, 50% WP, 24% SL and technical grade active ingredient), but the high solubility of this compound in water makes it unlikely that the type of formulation will significantly affect the residue levels in crops.

In the trials before the 1970s the residues were determined by enzymatic methods which relied on the inhibition of acetylcholinesterase activity and showed only the total inhibitory activity. Since the (*E*)-isomer is a stronger inhibitor than the (*Z*)-, and the proportion of the two isomers can be changed by their different dissipation rates in or on crops, the total inhibitory activity does not determine either the individual isomers or their sum. On the other hand, the GLC methods employed in the supervised trials after the 1960s can determine the residues of the two isomers separately, and generally have determination limits of 0.01 mg/kg and recoveries of 80-110% for both the (*E*)- and (*Z*)- isomers.

The Meeting agreed not to evaluate the results obtained by enzymatic methods because they do not determine the correct sum of the two isomers and have other limitations. Such methods are also unsuitable for the regulatory determination of mevinphos residues.

The Meeting also decided not to evaluate the results from trials where the duration or conditions of storage of analytical samples were not reported, because the data on the stability of stored analytical samples show that storage conditions are very important for the preservation of the residues of this pesticide and there is no guarantee that residues would be stable for a 10-month period.

However the studies of the stability of mevinphos residues in analytical samples of lettuce, strawberries and turnips which accompanied the plant metabolism studies showed that mevinphos residues were stable in these crops under frozen conditions (~ -20EC) for at least 10 months.

Since the residues of mevinphos in most crops disappear quickly after application the number of applications will not have a significant effect on the residues at the time of harvest. The number of applications was therefore generally disregarded when trial conditions were compared with GAP.

The plant metabolism studies showed that residual mevinphos would be degraded rapidly through the cleavage of the P-O-C linkage, leading to the formation of methyl acetoacetate, and by a minor pathway to (*E*)-mevinphos acid. Since the level of (*E*)-mevinphos acid is low in relation to the parent compound however, the Meeting considered that it could be excluded from the definition of the residue.

The Meeting concluded that the present definition of the residue (sum of (*E*)-mevinphos and (*Z*)-mevinphos) should be retained both for enforcement and the estimation of dietary intake.

Oranges. Only one supervised trial, carried out in South Africa in 1972, was reported. The trial application (0.04 kg ai/hl) was not comparable with GAP in South Africa (0.015 kg ai/hl, 3 days PHI), and no other information on GAP for citrus fruits was submitted.

There were insufficient data to estimate a maximum residue level for oranges or citrus fruit, and the Meeting recommended the withdrawal of the CXL for citrus fruits (0.2 mg/kg).

Apples and pears. Four supervised trials on apples were carried out in France (1969) and five in the UK (1971 and 1972). Two UK trials at an application rate of 0.25 kg ai/ha complied with French GAP (0.25-0.75 kg ai/ha, 0.05 kg ai/hl, 7 days PHI) and were comparable with Austrian GAP (0.096-0.19 kg ai/ha, 0.0096-0.019 kg ai/hl, 14 days PHI). Two other UK and two French trials at application rates of 0.5 kg ai/ha were also according to French GAP. The other trials were at a higher rate than any GAP reported to the Meeting.

Two supervised trials on pears were carried out in France in 1969, but critical information such as sample storage conditions was not reported.

The Meeting could not estimate a maximum residue level for apples or pears, as there were too few appropriate trials, and recommended the withdrawal of the existing CXLs for apple (0.5 mg/kg) and pear (0.2 mg/kg).

Apricots. Only one supervised trial carried out in the USA in 1958 was reported, with no comparable GAP. The trial samples were analysed by an enzymatic method, and the sample storage period was not reported. The Meeting could not estimate a maximum residue level and recommended the withdrawal of the existing CXL for apricot (0.2 mg/kg).

Cherries. Fourteen supervised trials were carried out in France (4 in 1970 and 4 in 1971), Germany (3 in 1974, 1 in 1982 and 1 in 1983) and the USA (1 in 1958).

The three German trials in 1974 with application at 0.025 kg ai/hl and one in 1983 at 0.24 kg ai/ha and 0.024 kg ai/hl could be compared with Austrian GAP (0.096-0.19 kg ai/ha, 0.0096-0.019 kg ai/hl, 14 days PHI). The 1982 trial was at the same rate as in 1983, but sampling was only up to 10 days whereas the GAP PHI is 14 days. The Meeting concluded that in view of the rapid decline of this compound on crops a PHI of 10 days was not comparable with the GAP PHI. The residues in the other trials were <0.02-0.09 mg/kg at PHIs of 14-28 days.

The other nine trials were not comparable with any reported GAP. In the US trial the samples were analysed by an enzymatic method and the sample storage period was not reported.

The Meeting could not estimate a maximum residue level from the few adequate trials, and recommended the withdrawal of the CXL for cherries (1 mg/kg).

Peaches. Two supervised trials in France in 1969 could not be evaluated because critical information on such items as sample storage conditions was not reported. Three supervised trials in Germany (1974) at an application rate of 0.025 kg ai/hl were comparable with Austrian GAP (0.0096-0.019 kg ai/hl, 14 days PHI). The residues were <0.02-0.03 mg/kg at 14-21 days. A US trial in 1957 was not comparable with any reported GAP, the samples were analysed by an enzymatic method and the sample storage period was not reported.

There were too few satisfactory trials to estimate a maximum residue level and the Meeting recommended the withdrawal of the CXL for peach (0.5 mg/kg).

Currants. Ten supervised trials were carried out in Germany (1974 and 1975) and the UK (1971) but no information on comparable GAP was reported. The Meeting could not estimate a maximum residue level.

Grapes. Four French trials in 1969 could not be evaluated because sample storage conditions were not reported.

One French trial in 1971 with an application rate of 0.15 kg ai/ha complied with French GAP (0.05-0.25 kg ai/ha, 0.05 kg ai/hl, 7 days PHI). The residue was <0.02 mg/kg at 5 days. Two South African trials and one US trial were not comparable with any reported GAP and the US samples were analysed by an enzymatic method.

The Meeting could not estimate a maximum residue level and recommended the withdrawal of the CXL for grapes (0.5 mg/kg).

Strawberries. Seven supervised trials were carried out in Portugal (1971) and the USA (1957 and 1962), but no comparable GAP was reported. The US samples were analysed by an enzymatic method, and the sample storage period was not reported in the 1957 trial.

The Meeting recommended the withdrawal of the CXL for strawberry (1 mg/kg).

Broccoli. Two supervised trials were carried out in the USA in 1965 but no comparable GAP was reported and the analyses were by an enzymatic method.

The Meeting recommended the withdrawal of the CXL for broccoli (1 mg/kg).

Cauliflower. Eight supervised trials were carried out in France (1969), Germany (1974), and the USA (1972) but no comparable GAP was reported. Sample storage conditions were not reported in the French trials.

The Meeting could not estimate a maximum residue level and recommended the withdrawal of the CXL for cauliflower (1 mg/kg).

Brussels sprouts. Only one supervised trial, which was carried out in South Africa in 1980, was reported. This conformed to South African GAP (Brassica vegetables: 0.11 kg ai/ha, 0.011 kg ai/hl, 4 days PHI), with residues of <0.04 mg/kg at 4-16 days, but was inadequate to estimate a maximum residue level.

The Meeting recommended the withdrawal of the CXL for Brussels sprouts (1 mg/kg).

Head cabbages. Six supervised trials were carried out in Germany (1975, 1982 and 1983), and 14 in the UK (1960, 1970 and 1972).

In the six UK trials in 1960 the samples were analysed by an enzymatic method.

The conditions in three German trials in 1975 with applications at 0.025 kg ai/hl were comparable with French GAP (0.35 kg ai/ha, 0.035 kg ai/hl, 7 days PHI). The highest residues at 7-21 days were 0.02 and 0.03 (2) mg/kg.

Three German trials in 1982 and 1983 were at application rates of 0.43 kg ai/ha and 0.072 kg ai/hl. This is higher than GAP in Austria (0.096 kg ai/ha, 0.0096 kg ai/hl, 14 days PHI), France (0.35 kg ai/ha, 0.035 kg ai/hl, 7 days PHI) and The Netherlands (Brassica vegetables: 0.015-0.11 kg ai/ha, 0.007-0.011 kg ai/hl, 7 days PHI), but the results could be used for residue evaluation because all three residues were below the limit of determination of 0.02 mg/kg at 7-10 days at, effectively, maximum GAP conditions.

The application rate of 0.25 kg ai/ha in five UK trials in 1970 and 1972 was comparable to the GAP rate in France, but in four of them samples were taken only up to 5 days and residues were detected in two. The other three results could be used because the residues were all below the LOD of 0.02 mg/kg.

In three other UK trials in 1970 and 1972 the application rate of 0.5 kg ai/ha was higher than GAP in Austria, France, and The Netherlands, but two results could be used for evaluation because the residues were <0.02 mg/kg at 7-8 days, below the limit of determination.

The Meeting could use six German and five UK trials to estimate a maximum residue level and an STMR. The residues from the eleven trials in rank order were <0.02 (8), 0.02 and 0.03 (2) mg/kg.

The Meeting estimated a maximum residue level of 0.05 mg/kg, to replace the existing CXL of 1 mg/kg, and an STMR level of 0.02 mg/kg for head cabbages.

Kale. Nine supervised trials were carried out in Germany in 1974, 1982 and 1983 but no comparable GAP was reported, so the Meeting could not estimate a maximum residue level and recommended the withdrawal of the existing CXL (1 mg/kg).

Chinese kale. Two supervised trials were reported by the government of Thailand and one was in accord with the national GAP (Brassica vegetables: 0.36-0.48 kg ai/ha, 0.036-0.048 kg ai/hl, 3 days PHI). One trial was not enough to estimate a maximum residue level.

Cucumbers. Three outdoor German trials at 0.025 kg ai/hl were comparable with French GAP (0.35 kg ai/ha, 0.035 kg ai/hl, 7 days PHI) and all the residues were <0.02 mg/kg.

Four outdoor trials in The Netherlands were not comparable with any reported GAP and sample storage conditions were not reported.

Three German glasshouse trials in 1974 at 0.025 kg ai/hl and two in 1983 at 0.29 kg ai/ha and 0.032 kg ai/hl did not accord with any reported GAP. One German glasshouse trial in 1982 (0.14 kg ai/ha, 0.023 kg ai/hl) complied with GAP in The Netherlands (Fruiting vegetables in glasshouse: 0.073-0.22 kg ai/ha, 0.007-0.015 kg ai/hl, 3 days PHI). The residues were 0.02-0.04 mg/kg at 3-7 days.

There were too few trials to estimate a maximum residue level and the Meeting recommended the withdrawal of the CXL for cucumber (0.2 mg/kg).

Melons and watermelons. Three supervised trials on melons and one on watermelons were reported but no information on comparable GAP was available. The samples were analysed by an enzymatic method and two trials lacked information on the conditions or duration of sample storage.

No maximum residue level could be estimated and the Meeting recommended the withdrawal of the CXL for melons except watermelon (0.05 mg/kg).

Tomatoes. Four outdoor trials were carried out in Belgium (1969), two in France (1969) and three in Germany (1975). Two glasshouse trials were conducted in Belgium (1970), four in Germany (1975 and 1982) and two in The Netherlands (1970).

The three German outdoor trials (0.025 kg ai/hl) could be compared to French GAP (0.35 kg ai/ha, 0.035 kg ai/hl, 7 days PHI); all three residues were <0.02 mg/kg at 7-10 days. In the other six outdoor trials the sample storage conditions were not reported.

Three of the four German glasshouse trials were not comparable with any reported GAP. The other (0.14 kg ai/ha, 0.023 kg ai/hl) corresponded to GAP in The Netherlands (Fruiting vegetables in glasshouse: 0.073-0.22 kg ai/ha, 0.007-0.015 kg/l, 3 days PHI) and Belgium and Luxembourg (0.12-0.36 kg ai/ha, 0.012-0.018 kg ai/hl, 7-14 days PHI). The residues were <0.02 mg/kg at 3-7 days.

The Meeting could not estimate a maximum residue level owing to the small number of trials, and recommended withdrawal of the existing CXL for tomato (0.2 mg/kg).

Baby corn. Two supervised trials were reported by the government of Thailand and one, with application at 0.14 kg ai/ha, was close to the national GAP (Sweet corn: 0.06-0.12 kg ai/ha, 0.012-0.024 kg ai/hl, 3 days PHI). No residue was detectable. One trial was not enough to estimate a maximum residue level.

Lettuce. Nineteen outdoor trials were carried out in Belgium (4), France (4), Germany (6), The Netherlands (2), Spain (1) and the UK (2), and 22 glasshouse trials in Belgium (8), Germany (4), The Netherlands (6), and the USA (4).

There was no information on whether the crops were head or leaf lettuce, and the sample storage conditions were not reported in the Belgian or Dutch outdoor trials or in two of the Dutch glasshouse trials. In the US trials (in 1965) the samples were analysed by an enzymatic method.

The manufacturer informed the Meeting that data on new trials to cover both head and leaf lettuce would be submitted to a future Meeting.

The Meeting could not estimate a maximum residue level because essential information was not available and recommended the withdrawal of the CXL for head lettuce (0.5 mg/kg).

Spinach. Three supervised trials were carried out in Germany (1974), three in the UK (1958) and one each in Belgium (1972 under glass) and South Africa (1980).

The Belgian trial was at an application rate of 0.1 kg ai/hl, but no comparable GAP was reported. The samples in the UK trials were analysed by an enzymatic method. The trial in South Africa at an application rate of 0.11 kg ai/ha with 0.022 kg ai/hl did not comply with South African GAP (0.11 kg ai/ha, 0.011 kg ai/hl, 3 days PHI).

In the German trials the application rate of 0.025 kg ai/hl was comparable to French GAP (0.35 kg ai/ha, 0.035 kg ai/hl, 7 days PHI). The residues at 7 days were 0.03, 0.05 and 0.07 mg/kg.

The Meeting could not estimate a maximum residue level as there were too few trials, and recommended the withdrawal of the CXL for spinach (0.5 mg/kg).

Carrots, celeriac, potatoes and turnips. Three German trials on carrots were at an application concentration of 0.025 kg ai/hl. This is higher than GAP in The Netherlands (root and tuber vegetables: 0.015-0.15 kg ai/ha, 0.007-0.015 kg ai/hl, 7 days PHI), but the results could be used for residue evaluation because all three residues were below the limit of determination of 0.02 mg/kg at 7-14 days. A single US trial was not comparable to any reported GAP and the residues were determined by an enzymatic method.

Three supervised trials were carried out on celeriac in Germany in 1983 at an application rate of 0.16 kg ai/ha at 0.027 kg ai/hl. The spray concentration was higher than GAP in The Netherlands but again all three residues were <0.02 mg/kg at 7 days.

Supervised trials were carried out on turnips and potatoes in the USA in 1956 but no comparable GAP was reported and the residues were determined by an enzymatic method.

The Meeting could not estimate maximum residue levels for carrots, celeriac, potatoes or turnips with so few trials and recommended the withdrawal of the existing CXLs for carrot, potato and garden turnip (all 0.1 mg/kg).

Leeks. Five supervised trials were carried out in Germany (1974, 1982 and 1983) at application rates of 0.025 kg ai/hl or 0.14-0.16 kg ai/ha with 0.023-0.027 kg ai/hl. These rates are higher than GAP in The Netherlands for stem vegetables (including leeks) of 0.015-0.15 kg ai/ha, 0.007-0.015 kg ai/hl, 7 days PHI, but all five residues were below the limit of determination, <0.02 mg/kg, at 7-14 days.

The Meeting estimated a maximum residue level of 0.02* mg/kg and an STMR of 0.02 mg/kg.

Bulb onions. Only one supervised trial, carried out in the USA in 1956, was reported. There was no comparable GAP, and residues were determined by an enzymatic method.

The Meeting could not estimate a maximum residue level and recommended the withdrawal of the CXL for bulb onions (0.1 mg/kg).

Common beans. Eleven outdoor trials were carried out in Germany (3 in 1974) and the UK (6 in 1960 and 2 in 1971) and four glasshouse trials in Germany in 1974 and 1982, but the samples in the 1960 UK trials were analysed by an enzymatic method.

The three outdoor trials in Germany in 1974 were at an application concentration of 0.025 kg ai/hl. This was higher than GAP in The Netherlands (Legume vegetables: 0.015-0.15 kg ai/ha, 0.007-

0.015 kg ai/hl, 7 days PHI) but two of the trials could be used for residue evaluation because all the residues at 7-14 days were <0.02 mg/kg, below the limit of determination.

The two outdoor trials in the UK in 1971 were at application rates of 0.25 and 0.5 kg ai/ha. The lower rate was comparable to French GAP (0.35 kg ai/ha, 0.035 kg ai/hl, 7 days PHI) but exceeded GAP in The Netherlands, and the higher rate exceeded GAP in both countries. The results of both trials could again be evaluated however because the residues were below the limit of determination, <0.02 mg/kg, at 2-7 days. This also applied to two of the three glasshouse trials in Germany in 1974 where the application concentration of 0.025 kg ai/hl was higher than GAP in The Netherlands (Legume vegetables in glasshouse: 0.036-0.15 kg ai/ha, 0.007-0.015 kg ai/hl, 7 days PHI for July).

A glasshouse trial in Germany in 1982 was at a rate of 0.14 kg ai/ha with 0.023 kg ai/hl. The kg ai/ha rate accorded with GAP in The Netherlands and the residue at 7 days was 0.03 mg/kg.

The Meeting agreed to combine the residue data from the outdoor and glasshouse trials because no difference was observed in the residue populations. The residues from the seven relevant trials in rank order were <0.02 (6) and 0.03 mg/kg.

The Meeting estimated a maximum residue level of 0.05 mg/kg and an STMR of 0.02 mg/kg for common beans. The maximum residue level is recommended as an MRL to replace the existing CXL (0.1 mg/kg).

Peas. Four supervised trials were carried out in South Africa (1969) and the UK (1957), but all the samples were analysed by enzymatic methods.

The Meeting could not estimate a maximum residue level and recommended the withdrawal of the CXL for peas (0.1 mg/kg).

Soya beans and peanuts. A supervised trial on soya beans in Brazil and two trials on peanuts in Brazil and South Africa were reported to the Meeting, but relevant GAP was not available.

The Meeting could not estimate maximum residue levels.

Sugar beet. Five supervised trials were carried out in France in 1975 at application rates of 0.24, 0.25 and 0.35 kg ai/ha, but the PHIs (91-98 days) were not comparable with any reported GAP.

In two supervised trials in Germany the application concentration of 0.025 kg ai/hl complied with Austrian GAP (0.12 kg ai/ha, 0.02-0.03 kg ai/hl, 14 days PHI). The residues at 7-14 days were <0.02 mg/kg (4 results) in the roots and <0.02 (2), 0.03 and 0.04 mg/kg in the leaves.

The Meeting could not estimate a maximum residue level for sugar beet or for sugar beet leaves or tops.

Storage

Storage studies were carried out on peaches, strawberries, red cabbage, broccoli, lettuce and spinach at ambient temperature.

Because the number of trials was limited the decline profile of mevinphos residues in or on crops was not clear, but the data showed that the residues declined quickly except in peaches. Half-lives of 29, 3.7-3.9, 3.4, 1.3, 3.4-6.3 and 2.2 days were calculated for peaches, strawberries, red cabbage, broccoli, lettuce and spinach respectively on the assumption that the logarithm of the residue value decreased linearly with time.

Processing

Household. Boiling, washing, and/or peeling studies were carried out on red cabbage, broccoli, cauliflower, spinach and apples but the samples from two of the three broccoli trials and the two cauliflower trials were analysed by enzymatic methods and were not evaluated.

The processing factors were 0.27 and 0.67 for washing cabbage, 0.18 for boiling cabbage, 0.33 for boiling broccoli, 0.26-0.32 for boiling spinach, 0.71-0.95 for peeling apples and 0.53-0.75 for cooking peeled apples.

Industrial. Processing factors for grapes were 0.87 to fresh juice, 1.15 to wet pomace, 0.33 to dry pomace, 0.25 to raisins and 22.2 to raisin waste. Evidently more mevinphos than moisture is lost during drying.

RECOMMENDATIONS

On the basis of the data on residues from supervised trials, the Meeting concluded that the residues listed below are suitable for establishing MRLs and STMR levels. The Meeting could not confirm many current CXLs and recommended their withdrawal. The recommended withdrawals are also indicated below.

Definition of the residue for compliance with MRLs and for estimation of dietary intake: Sum of (*E*)- and (*Z*)-mevinphos.

Commodity		Maximum residue level, mg/kg		PHI, days	Estimated STMR, for dietary intake estimation
CCN	Name	New	Previous		(mg/kg)
FP 0226	Apple	W	0.5		
FS 0240	Apricot	W	0.2		
VB 0400	Broccoli	W	1.0		
VB 0402	Brussels sprouts	W	1.0		
VB 0041	Cabbages, Head	0.05	1.0	7	0.02
VR 0577	Carrot	W	0.1		
VB 0404	Cauliflower	W	1.0		
FS 0013	Cherries	W	1.0		
FC 0001	Citrus fruits	W	0.2		
VP 0526	Common bean (pods and/or immature seeds)	0.05	0.1	7	0.02
VC 0424	Cucumber	W	0.2		
FB 0269	Grapes	W	0.5		
VA 0384	Leek	0.02*		7	0.02
VL 0480	Kale	W	1.0		
VL 0482	Lettuce, Head	W	0.5		
VC 0046	Melons, except watermelon	W	0.05		
VA 0385	Onion, Bulb	W	0.1		
FS 0247	Peach	W	0.5		
FP 0230	Pear	W	0.2		
VP 0063	Peas (pods and succulent = immature seeds)	W	0.1		
VR 0589	Potato	W	0.1		

Commodity		Maximum residue level, mg/kg		PHI, days	Estimated STMR, for dietary intake estimation
CCN	Name	New	Previous		(mg/kg)
VL 0502	Spinach	W	0.5		
FB 0275	Strawberry	W	1.0		
VO 0448	Tomato	W	0.2		
VR 0506	Turnip, Garden	W	0.1		

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MYCLOBUTANIL (181)

EXPLANATION

Myclobutanil first evaluated at the 1992 JMPR and maximum residue levels were estimated for fruits and animal commodities. Draft MRLs for apricots, cherries, peaches and plums are at Step 6.

At the 28th (1996) CCPR (ALINORM 97/24), the EU was in disagreement with residue evaluation on stone fruits.

The manufacturer proposed the reconsideration of MRLs for stone fruits in the light of new GAP in the USA. Updated information on new GAP for myclobutanil in the USA and the EU was available to the Meeting.

The manufacturer submitted new residue data on apricots, bananas, citrus fruit, blackcurrants, strawberries, tomatoes, and hops for evaluation.Myclobutanil is used as a post-harvest treatment on citrus and there is as proposed use on bananas to prevent losses during shipment.

METHODS OF RESIDUE ANALYSIS

Analytical methods

Analytical methods were developed to determine the parent compound and its main metabolites, α-(3-oxobutyl)-α-(4-chlorophenyl)-1*H*–1,2,4-triazole-1-propranenitrile (RH-9089, the keto derivative), α-(3-hydroxybutyl)-α-(4-chlorophenyl)-1*H*–1,2,4-triazole-1-propanenitrile (RH-9090) the hydroxy derivative, and the conjugate of RH-9090 (Stavinski *et al*.,1988a).

Residues are soxhlet-extracted overnight with 0.5 N HCl in methanol; this converts the RH-9090 conjugate to RH-9090. RH-9089 is reduced to RH9090 with sodium borohydride ($NaBH_4$). The extract is partially purified by partitioning successively with petroleum ether and methylene chloride, and further cleaned up by chromatography on columns of Chelex 100-Fe^{+++} and Florisil or silica gel. Myclobutanil and RH-9090 fractions are eluted separately by adjusting the polarity of the eluent. The two fractions are separately analysed by GLC with NP or EC detection. The recoveries of myclobutanil and RH-9090 were 92±17% and 83±20% respectively.

This method was also used to determine the parent compound and the same metabolites in bananas with an LOD of 0.01 mg/kg and recoveries of 90% for myclobutanil and 85% for RH-9090.

A simplified analytical method (Brackett, 1984), methanol extraction, partitioning first with hexane and then between hexane and methylene chloride, and clean-up on a Florisil column (silica gel or LC 18 Cartridge) and detection by GLC ECD or NPD, was used in many trials and reported in 1992 (Brackett,1984).

Another analytical method was reported for the analysis of hops. Samples are extracted with acetone/water and partitioned with hexane saturated with sodium chloride. The aqueous layer is extracted twice with dichloromethane. After evaporation of the solvent the reisude is dissolved in cyclohexane/ethyl acetate and cleaned up by gel permeation. The residues are determined by GC-MS, with an LOD of 0.2 mg/kg for both myclobutanil and RH-9090. The reported recoveries were 72% for myclobutanil and 81% for RH-9090 (Nat. Hop Assoc.1995).

It was reported that myclobutanil could be determined by the multi-residue method in The Netherlands "Analytical Methods for Pesticide Residues in Foodstuffs" 6th. edition (1996). The reported LOD in various types of samples ranged from 0.01 to 0.05 mg/kg, with a recovery of 107%.

Stability of pesticide residues in stored analytical samples

The stability of myclobutanil was studied in stored samples of soils, apples, grapes and tomatoes.

Soil samples fortified at 1 mg/kg with myclobutanil and stored at -15°C were analysed after 0, 7, 14, 42, 83, 183, 365 days and one and two years. There was no significant decrease with time. After two years recoveries were about 82% (Brackett *et al.*, 1985).

Apples. Untreated apple samples were homogenized, fortified at 0.1 mg/kg with [^{14}C]-myclobutanil, and stored at -15°C for two years. The average recovery was 95% ± 8% (Deakyne *et al.*, 1986a).

Grapes fortified at 0.1 mg/kg and stored at -15°C showed an average recovery of 98% over a two-year period (Deakyne *et al.*, 1986b).

Cucurbits. In a study to determine the effect of long-term frozen storage on myclobutanil and RH-9090 in cucurbits samples were fortified at 0.5 mg/kg and stored at -10°C for three years. There was no effect on the total residue during this period (Bartra, 1995a).

Tomatoes. Samples of tomatoes were spiked with 1 mg/kg each of myclobutanil and RH-9090 and stored frozen in the dark at approximately -10°C. At specified intervals from 0 to 36 months, samples of stored and freshly fortified tomatoes were analysed. After all intervals, the average recoveries of both myclobutanil and RH-9090 from the fresh fortifications were similar to those from the stored samples. Recoveries ranged from 77.8% to 100% for myclobutanil and from 63.1% to 109% for RH-9090 (Bartra, 1997a).

Almond kernels and hulls. Samples of almond kernels and hulls were spiked with 1 mg/kg of myclobutanil and RH-9090. After two years of storage at -10°C there was no decrease of either compound (Bartra,1997b).

Definition of the residue

The results of supervised trials indicated that the parent compound is the major component of the residue. Where residues of the parent compound are significant those of the main metabolites are much lower. The previous definition of the residue as the parent compound is therefore considered appropriate for monitoring compliance with MRLs and for the estimation of dietary intake.

Definition of the residue (for compliance with MRLs and for the estimation of dietary intake): myclobutanil.

USE PATTERN

Information on GAP for the use of myclobutanil was given in the 1992 monograph. New information on GAP for stone fruit in the USA and the EU is shown in Table 1.

Myclobutanil formulated as an emulsifiable liquid concentrate or a wettable powder can be used to protect against crown rot complex in bananas at a concentration of 200 to 400 mg ai/l in water. It may be applied to banana hands by spraying, and in some locations by dipping or passing the fruit through a recirculating cascade system.

Myclobutanil formulated as an EC or wax emulsion in water can be used on citrus fruit as a post-harvest treatment to control Penicillium spp.

Table 1. Registered uses of myclobutanil.

Crops	Country	Form.	Application				PHI, days
			Method	kg ai/ha	kg ai/hl	No.	
Apricot	France	EC 125g/l	F		0.00625	3-6	7
	France	60 WP	F		0.006	3-6	7
	Greece	EC 125g/l	F	0.055-0.125	0.0037-0.005	3	15
	Hungary	EC	F	0.025-0.0375	0.0025	>2	24
	Romania	EC	F	0.062	0.0062	>2	10
	Spain	EC 75 g/l	F	0.09	0.006	1-2	21
		WP 8 g/kg	F	<0.084	0.0056	1-4	21
	Switzerland	WP 35 g/kg	F	0.05-0.078	0.005	>4	21
	USA	40 WP	F	0.07-0.165 max.total 1.22	0.0038-0.006	7-17	0
Blackcurrants	France	EC	F	0.075		5-6	14
		WP	F	0.075		5-6	14
	Eire	SC	F	0.09		4-6	14
	Switzerland	EC	F	0.084-0.165	0.007-0.011	>4	28
	Switzerland	WP	F	0.06-0.135	0.005-0.009	>4	28
	UK	SC	F	0.09		4-6	14
Cherries	France	EC	F		0.0075	2-4[1]	
	Germany	WP	F	0.135	0.009	>3	21
	Hungary	EC	F	0.025-0.0375	0.0025	>2	14-24
	Romania	EC	F	0.062	0.0062	>2	10
	Switzerland	WP	F	0.05-0.078		>4	21
	USA	WP	F	0.07-0.165 max. 1.45	0.0038-0.006	<9	0
Citrus fruit	Spain	SC	post-harvest drench or spray		0.05	1	0
	Spain	WWE 3g/l	post-harvest spray	0.01 kg ai /t of fruit	0.003	1	0
Hops	UK	SC	F	0.045-0.135	0.0045	6	10
Nectarines	Greece	EC	F	0.055-0.125	0.0037-0.005	3	15
	Hungary	EC	F	0.025-0.0375	0.0025	>2	24
	Romania	EC	F	0.062	0.0062	>2	10
	Spain	EC	F	0.1125	0.075	1-4	15
	Switzerland	WP	F	0.05-0.078	0.005	>4	21
	Tunisia	EC	F	0.1125	0.075	1-3	10
	USA	WP	F	0.07-0.165 max.1.45	0.0038-0.006	>9	0
	Yugoslavia	EC	F	0.0336-0.072	0.0048-0.006	4	35
Peaches	France	EC	F	0.075	0.00625-0.0075	3-6	10
	France	WP	F		0.0045	3-6	[1]
	Greece	EC	F	0.0555-0.125	0.0037-0.005	3	15
	Hungary	EC	F	0.025-0.0375	0.0025	>2	14
	Italy	SC	F	0.075-0.093	0.005-0.0062	3-4	15
	Italy	WP	F	0.042-0.073	0.0035-0.0052	3	15
	Israel	EC	F	0.09	0.006	1-2	14
	Portugal	EC	F	0.0375	0.00375	1-2	21

Crops	Country	Form.	Application				PHI, days
			Method	kg ai/ha	kg ai/hl	No.	
	Spain	EC	F	0.09-0.1125	0.006-0.0075	1-4	15
	Spain	WP	F	0.084	0.0056	1-4	15
	Switzerland	WP	F	0.05-0.078	0.005	>4	21
	Tunisia	EC	F	0.1125	0.0075	1-3	10
	USA	WP	F	0.07-0.165 max. 1.45/season	0.0038-0.006	<9	0
	Yugoslavia	SC	F	0.0336-0.0576	0.0048	4	35
Plums	France	WP	F		0.0045-0.0075	2-6	[1]
	France	EC	F		0.0075	2-4	[1]
	Greece	EC	F	0.055-0.1125	0.0037-0.005	3	15
	Hungary	EC	F	0.025	0.0025	up to 2	14
	Israel	EC	F	<0.12	0.006-0.012	2-3	14 (b)
	Romania	EC	F	0.062	0.0062	up to 2	10
	Switzerland	WP	F	0.05-0.078	0.005	up to 4	21
	Tunisia	EC	F	<0.1125	0.0075	1-3	10
Plums /Prunes	USA	40WP	F	0.07-0.165 max. 1.22	0.0038-0.006	7	0
Strawberries	Belgium	12EC	F	0.0625		3-6	1
	Eire	6SC	F	0.09		4- 6	3
	France	12EC	F	0.0625		4-6	3
		60WP	F	0.06		4-6	3
		7.5 EC	F	0.0375		2	3
	Germany	60WP	F	0.045-0.12	0.0045-0.006	up to 6	7
	Italy	12EC	F	0.05	0.005	3-4	7
	Spain	12EC	F	0.075	0.0075	1-4	5
	Spain	8WP	F	0.048	0.0048	1-4	5
	Spain	7.5 EC	F	0.06	0.006	1-2	7
	Switzerland	35WP	F	0.06-0.135	0.005-0.009	up to 4	
		EC	F	0.084-0.165	0.007-0.011	up to 4	28
	UK	6SC		0.09		4-6	3
Tomatoes	Belgium	60 WP	fg	<0.075	0.0075	3-6	3
	Belgium	125 g/l EC	fg/F	<0.075	0.0075	3-6	3
	Canada	40 WP	fg/F	0.136	0.0136	1-2	14
	Italy	12EC	fg/F	0.05-0.075	0.005-0.0075	3-4	7
	Israel	12EC	F	0.120		2-3	3
	Morocco	12EC	fg/F	0.62	0.006	1-3	7
	Spain	12EC	fg/F	<0.112	0.0075	1-6	3
		8WP	fg/F	<0.072	0.0048	1-6	5
	USA	40WP	f/F	0.07 max. 0.4 /season			0 or 5[3]

F: foliar
fg: field or glasshouse
[1] No PHI on label
[2] Also post-harvest
[3] For tomatoes for processing

RESIDUES RESULTING FROM SUPERVISED TRIALS

Residues of both myclobutanil and its main metabilites were determined most of the trials. In the Tables residues of "RH-9090" are total residues of RH-9089, RH-9090 and its conjugates, determined and expressed as RH-9090.

Stone fruits

Supervised field trials carried out on stone fruits in France, Italy, Spain and the USA were reported in the 1992 monograph. Residues were measured in the edible portions of the fruits and the proportional weights of the edible portions were not given. The 1992 Meeting estimated maximum residue levels in accordance with the Codex commodity descriptions by taking into account the average percentage weights of the stones of the individual commodities.

Apricots. The 1992 monograph reported 10 trials in France and one in Italy; the metabolites were determined only in the Italian trial. Residues following treatments at 0.075 to 0.36 kg ai/ha were 0.04 to 0.17 mg/kg in samples taken 7-10 days after the last treatment.

US GAP reported to the present Meeting allows a maximum application of 1.22 kg ai/ha per season with a PHI of 0 days. Five trials were conducted at five locations in the USA according to GAP (7 applications at 0.167 kg ai/ha, 0 day PHI), with different crop varieties (Bartra *et al.*,1993). The plot size ranged from 4 to 15 trees. Ground applications were made with an air-blast sprayer or hand-gun. Apricot samples were analysed for myclobutanil and the metabolites by GLC with an LOD of 0.01 mg/kg. The sampling-to-analysis interval (SAI) ranged from 173 to 207 days. Myclobutanil residues in the edible portions ranged from 0.11 to 0.62 mg/kg and total residues from 0.13 to 0.7 mg/kg.

The results are shown in Table 2.

Table 2. Myclobutanil residues in apricots from supervised trials. The underlined residues are from treatments according to Gap and were used to estimate maximum residue levels.

Country, State, year	Form.	Application			PHI days	Residues mg/kg			Ref.
		No.	kg ai/ha	Total kg ai/ha		Myclo-butanil	RH-9090	Total	
France, 1987	SC	6	0.06	0.36	8	<u>0.04</u>			F60-03-87
France, 1987	SC	6	0.06	0.36	7	<u>0.08</u>			F60-03-87
France, 1987	12EC	6	0.062	0.372	7	<u>0.06</u>			F60-01-87
France, 1987	12EC	6	0.075	0.45	7	<u>0.01</u>			F60-01-87
France, 1987	12EC	6	0.0625	0.375	8	<u>0.04</u>			F60-03-87
France, 1987	12EC	6	0.075	0.45	8	<u>0.04</u>			F60-03-87
France, 1986	12EC	1	0.075	0.075	0	0.4			F33-01-86
					5	0.4			
					10	0.17			
					14	0.1			
					21	0.05			
					28	0.02			
France	SC	7	0.06	0.42	14	0.04			F60-02-87
Italy	12EC	5	0.05, 0.075, 0.1, 0.15	0.3	18	0.02	<0.01	0.02	4148808
Italy	12EC	5	0.05, 0.075.1, 0.15	0.6	18	0.04	<0.01	0.04	
USA, CA 1992	40WP	7	0.167	1.17	0	<u>0.17</u>	<u>0.08</u>	<u>0.25</u>	Bartra *et al.*, 1993
USA, CA 1992	40WP	7	0.167	1.17	0	<u>0.23</u>	<u>0.06</u>	<u>0.29</u>	

Country, State, year	Form.	Application			PHI days	Residues mg/kg			Ref.
		No.	kg ai/ha	Total kg ai/ha		Myclo-butanil	RH-9090	Total	
USA, CA 1992	40WP	7	0.167	1.17	0	0.11	0.02	0.13	
USA, CA 1992	40WP	7	0.167	1.17	0	0.12	0.02	0.14	
USA, WA 1992	40WP	7	0.167	1.17	0	0.62	0.08	0.7	

Cherries. Data from residue decline trials in Germany were evaluated in 1992. Since four of these trials were according to German GAP reported to the present Meeting they were re-evaluated, with the results shown Table 3.

The 1992 monograph reported several trials on five varieties of fruit carried out on seven US States, where the total amounts applied were below the maximum permitted (1.45 kg ai/ha per season). The results were evaluated according to the 1992 GAP PHI of 7 days (Spina 1991a). The results of those trials which can be evaluated against the current US GAP PHI of 0 days are shown in Table 3.

In five other trials at four locations in the USA in 1987 (Spina, 1991b), five or six ground applications were made with an air-blast sprayer or a hand-gun, the total amounts applied being below the maximum permitted. The SAIs in these trials were 1501-1590 days. The total residues in the edible portions ranged from 0.22 to 2.05 mg/kg.

Table 3. Myclobutanil residues in cherries from supervised trials. The underlined residues are from treatments according to GAP and were used to estimate maximum residue levels.

Country, State, year	Application				PHI, days	Residues mg/kg			Ref.
	Form	No.	kg ai/ha	Total kg ai/ha		Myclo-butanil	RH-9090	Total	
Germany, 1987	6WP	3	0.09 0.135	0.315	0	0.94	0.02	0.96	DEU 87F20021
					7	0.17	0.03	0.2	
					14	0.06	0.02	0.08	
					21	0.02	<0.01	0.02	
					21	0.006	<0.01	0.006	juice
Germany, 1987	6WP	3	0.09-0.135	0.315	0	0.36	0.01	0.37	(DEU 87F20041)
					7	0.12	0.02	0.14	
					14	0.05	0.01	0.06	
					21	0.02	<0.01	0.02	
					21	0.02	<0.01	0.02	juice
Germany 1987	6WP	3	0.09 0.135	0.315	0	0.46	<0.01	0.46	(DEU 87F20111)
					14	0.01	<0.01	0.01	
					21	<0.01	<0.01	<0.01	
Germany 1987	6WP	3	0.09 0.09 0.135	0.315	0	0.19	0.02	0.21	DEU 87F20131
					7	0.02	<0.01	0.02	
					14	0.02	<0.01	0.02	
					21	0.02	<0.01	0.02	
USA, CA 1987	60DF	5	0.21	1.05	0	0.68	0.11	0.79	Spina 1991a
					7	0.44	0.23	0.67	
					15	0.27	0.2	0.47	
					21	0.16	0.46	0.62	

Country, State, year	Application				PHI, days	Residues mg/kg			Ref.
	Form	No.	kg ai/ha	Total kg ai/ha		Myclo-butanil	RH-9090	Total	
USA, CA 1987	60DF	5	0.21	1.05	7	0.41	0.19	0.6	Spina 1991a
					15	0.22	0.27	0.49	
					21	0.23	0.39	0.62	
USA, CA, 1987	60DF	5	0.21	1.05	0	0.92	0.13	1.05	Spina 1991a
					7	0.45	0.06	0.51	
					15	0.39	0.11	0.50	
					21	0.02	0.01	0.03	
USA, MI,1984	40W	5	0.425	2.13	0	2.64	0.44	3.08	
USA, WI, 1984	40W	6	0.421	2.53	9	0.56	0.39	0.95	
USA WI, 1984	40W	7	0.421	2.95	1	1.0	0.48	1.48	
USA OR, 1987	60DF	5	0.21	1.05	0	0.28	0.04	0.32	Spina 1991a
					7	0.19	0.04	0.23	
					14	0.10	0.05	0.15	
					21	0.03	0.03	0.06	
USA MI, 1987	60DF	6	0.21	1.26	7	0.92	0.60	1.52	
					14	0.61	0.73	1.34	
USA MI, 1987	60DF	6	0.21	1.26	7	0.84	0.58	1.42	
					14	0.35	0.60	0.95	
USA WI, 1987	60DF	6	0.21	1.26	8	0.38	0.43	0.81	
					14	0.19	0.50	0.69	
USA CA, 1987	60DF	5	0.211	1.06	0	0.85	0.22	1.07	Spina, 1991b
USA OR, 1987	60DF	5	0.211	1.06	0	0.20	0.02	0.22	
USA MI, 1987	60DF	6	0.211	1.39	0	1.44	0.61	2.05	
USA MI, 1987	60DF	6	0.211	1.39	0	1.04	0.57	1.61	
USA WI, 1987	60DF	6	0.211	1.39	0	1.12	0.4	1.52	

Peaches. Several field trials carried out in the USA, France and Spain were evaluated in 1992. Details of some of them that can be re-evaluated according to current GAP are given in Table 4.

The Meeting also received data from field trials in 1987 on seven varieties at seven locations in the USA, where samples were taken at a 0-day PHI. Total applications up to 1.5 kg ai/ha approximated GAP (maximum application per season 1.45 kg ai/ha). Residues were determined in the edible portions of the fruit. The SAIs were 1486-1492 days (Ding,1991a). The results are shown in Table 4.

Table 4. Myclobutanil residues in peaches from supervised trials. The underlined residues are from treatments according to GAP and were used to estimate maximum residue levels.

Country, Location, Year	Application				PHI, days	Residues, mg/kg			Ref.
	Form.	No.	kg ai/ha	Total kg ai/ha		Myclobut	Rh-9090	Total	
France 1986	EC	1	0.075	0.075	0	0.01			F31-02-86
					5	0.05			
					10	0.03			
					14	0.03			
					21	0.02			
					28	0.02			
Spain 1985	6WP	5	0.081	0.41	20	0.02			491-85-18
Spain 1987	12EC	3	0.10	0.30	20	0.03			491-87-14
USA, AR 1987	60DF	9	0.211	1.90	0	0.33	0.02	0.35	Ding,
USA, CA 1987	60DF	7	0.211	1.48	0	0.66	0.11	0.76	1991a
USA, NC 1987	60DF	10	0.211	2.11	0	0.84	0.28	1.12	

Country, Location, Year	Application				PHI, days	Residues, mg/kg			Ref.
	Form.	No.	kg ai/ha	Total kg ai/ha		Myclobut	Rh-9090	Total	
USA, PA 1987	60DF	8	0.22	1.78	0	1.22	0.31	1.53	
USA, VA 1987	60DF	9*	0.209	1.81	0	0.85	0.21	1.06	
USA, CA 1987	60DF	9	0.211	1.90	0	0.38	0.17	0.55	
USA, MO 1987	60DF	11	0.211	2.32	0	0.62	0.19	0.81	
USA, CA 1987	60DF	7	0.21	1.47	0	0.74	0.17	0.91	
					7	0.36	0.24	0.6	
					14	0.24	0.22	0.46	
					20	0.11	0.11	0.22	
USA, CA 1987	60DF	7	0.21	1.49	0	0.75	0.07	0.82	Stavinski *et al.*,
					7	0.28	0.06	0.34	1987b
					14	0.23	0.06	0.29	
					20	0.11	0.07	0.18	
USA, AR 1987	60DF	9	0.21	1.89	0	0.34	0.03	0.37	Stavinski *et al.*,
					7	0.18	0.04	0.22	1987a
					14	0.1	0.04	0.14	
USA, CA 1987	60DF	7	0.21	1.44	0	0.75	0.07	0.82	Stavinski *et al.*,
					7	0.28	0.06	0.34	1987a
					14	0.23	0.06	0.29	

Plums and prunes. The 1992 JMPR evaluated field trials on plums in three US States. In five trials the parent ane metabolites were determined in both fresh and dried fruit. Data from supervised trials in Italy reported to the 1992 JMPR were re-evalutated against other current southern European GAP.

The Meeting also received summarized data from field trials in 1987 on four varieties of fruit at five locations in the USA, where samples were taken at a 0-day PHI. In three of these trials the total application was much higher than permitted per season according to US labels (1.22 kg ai/ha). The application season for these trials was from the end of February to July, with PHIs of 8 to 30 days. The SAIs ranged from 1474 to 1573 days (Ding, 1991b). The residues of the parent compound ranged from 0.09 to 1.12 mg/kg and the total residues from 0.1 to 1.44 mg/kg. The results of all the evaluated trials are shown in Table 5.

Table 5. Myclobutanil residues in plums from supervised trials. The underlined residues are from treatments according to GAP and were used to estimate maximum residue levels.

Location, Year	Application				PHI, days	Residues, mg/kg			Ref.
	Form.	No.	kg ai/ha	Total, kg ai/ha		Myclobut	RH-9090	Total	
Italy, 1988	12EC	4	0.05-0.075	0.25	14	0.01	0.03	0.04	41 48808
Italy, 1988	12EC	4	0.1-0.15	0.5	14	0.07	0.03	0.1	
USA, CA 1987	60DF	9	0.21	1.92	0	0.11	0.04	0.15	Stavinski *et al.*, 1988b
					6	0.07	0.03	0.1	
					14	0.06	0.03	0.09	
USA, CA 1987	60DF	7	0.211	1.48	0	0.09	0.01	0.1	Ding, 1991b
USA, CA 1987	60DF	9[2]	0.211	1.90	0	0.25	0.11	0.36	
USA, WA 1987	60DF	8[3]	0.211	1.69	0	0.28	0.12	0.4	
USA, MI 1987	60DF	6[4]	0.211	1.39	0	0.59	0.14	0.73	
USA, PA 1987	60DF	12[5]	0.200	2.67	0	1.12	0.32	1.44	
USA, CA 1987	40W	6	0.21	1.28	14 (Ff)	0.41	0.07	0.48	Stavinski *et al.*, 1990
					14 (Df)	0.49	0.22	0.71	
USA, CA 1989	40W	6	0.21	1.28	14 (Ff)	0.12	0.01	0.13	
					14 (Df)	0.53	0.10	0.63	

Location, Year	Application				PHI, days	Residues, mg/kg			Ref.
	Form.	No.	kg ai/ha	Total, kg ai/ha		Myclobut	RH-9090	Total	
USA, CA 1989	40W	6	0.21	1.28	14 (Ff)	0.14	<0.01	0.14	
					14 (Df)	0.74	0.22	0.96	
USA, ID 1989	40W	6	0.21	1.28	14 (Ff)	0.05	<0.01	0.05	
					14 (Df)	0.22	0.02	0.24	
USA, OR	40W	6	0.21	1.28	14 (Ff)	0.09	0.04	0.13	
					14 (Df)	0.35	0.34	0.69	

Ff: fresh fruit. Df: dried fruit
[1]Application intervals 8 to 36 days from February to June, the two last applications at 8 and 14 day intervals
[2]Applications from April to August at 13-16 day intervals
[3]Applications from April to August at 7-42 day intervals, the two last applications at 31-42 day intervals
[4]Applications from April to September at 7-14 day intervals, the two last applications at 14 and 6 day intervals

Bananas. Four trials in the USA (California) were according to the proposed use in the banana packing stations in Central America. They were designed to simulate the normal treatment procedures used by commercial growers before shipment and distribution. All treatments consisted of one application at 200, 400 or 800 mg ai/l with a back-pack or hand-gun sprayer. Washed green banana hands were dipped or sprayed and allowed to air-dry for one hour before packing and storage in a cold room at 13°C.

Samples of bananas were taken at 0, 7, 14 and 21 days after application were removed from cold storage, gasified for 24 hours with ethylene and stored for 3 days to simulate supermarket shelf storage. A minimum of 16 random individual whole fruit samples from 4 hands per trial were then sampled, separated into peel and pulp, chopped and stored frozen (-10°C) until analysis. The SAIs were 8-14 days (Zogorski and Ding, 1992). The results are shown in Table 6. Because the highest rate consistent with the proposed GAP is 400 mg ai/l, the data from trials carried out at 800 mg ai/l were omitted from the Table.

Four other trials were conducted in Hawaii, where myclobutanil (CE or WP) was used at 200, 400 or 800 mg ai/l. Whole fruit were weighed before separation into pulp and peel, the weights of which were also recorded (Bartra and Zogorski, 1993). At day 0 residues in the pulp were between 0.01 and 0.05 mg/kg, but increased slightly with storage. The hands lost weight during storage, by an average of 7.26%, mainly from the peel. Residues in the whole fruit were 0.49-0.61 mg/kg from 200 mg ai/l and 0.6-1.36 mg/kg from 400 mg ai/l. The results are shown in Table 6.

In three trials at three different sites in Costa Rica bananas were treated with myclobutanil 2EC at 0, 100, 200 and 400 mg ai/l. Samples of pulp and peel taken 7, 14 and 21 days after treatment were analysed and the residues in the whole fruit calculated (Bartra,1994). The residue in the peel were 0.46-0.65 mg/kg from 100 mg ai/l, 1.0-1.6 mg/kg from 200 mg ai/l and 2.7-3.9 mg/kg from 400 mg ai/l. The residues in the whole fruit were 0.5-0.69 mg/kg from 200 mg ai/l and 1.3-1.7 mg/kg from 400 mg ai/l. Because the lowest rate consistent with the proposed GAP is 200 mg ai/l the data from the trial at 100 mg ai/l are not shown in Table6.

Table 6. Myclobutanil residues in bananas from supervised trials with treatments according to proposed GAP. Singly underlined residues would be used to estimate maxium residue levels, and doubly underlined to estimate STMRs if the GAP is confirmed.

Country, State, Year	Application			Storage period, days	Sample	Residue, mg/kg			Ref.
	Form	mg ai/l	Method			Myclob.	HD-9090	Total	
USA, CA 1991	2EC	200	spray	0	peel	0.36	0.01	0.37	Zogorski 1992

Country, State, Year	Application			Storage period, days	Sample	Residue, mg/kg			Ref.
	Form	mg ai/l	Method			Myclob.	HD-9090	Total	
					pulp	nd	nd	nd	
Trial 1				7	peel pulp	0.29 <0.01	<0.01 nd	0.3 <0.01	
				14	peel pulp	0.33 0.02	<0.01 nd	0.34 0.02	
				21	peel pulp	* 0.02	0.02 nd	* 0.02	
		400	spray	0	peel pulp	0.36 <0.01	nd nd	0.36 <0.01	
				7	peel pulp	0.51 0.02	0.01 nd	0.52 0.02	
				14	peel pulp	0.34 0.02	0.02 <0.01	0.36 0.03	
				21	peel pulp	0.82 0.03	0.02 <0.01	0.84 0.03	
USA, CA, 1991	2EC	200	hand dip	0	peel pulp	1.08 nd	<0.01 nd	1.09 nd	
Trial 2				7	peel pulp	1.14 0.03	0.08 0.01	1.22 0.04	
				14	peel pulp	1.1 0.03	0.05 <0.01	1.15 0.03	
				21	peel pulp	1.58 0.07	0.03 <0.01	1.6 0.08	
	2EC	400	dip	0	peel pulp	1.64 nd	<0.01 nd	1.64 nd	
				7	peel pulp	2.06 0.03	0.07 0.01	2.13 0.04	
				14	peel pulp	2.34 0.05	0.07 0.01	2.4 0.06	
				21	peel pulp	2.54 *	0.05 <0.01	2.6 *	
USA CA, 1991	2EC	200	spray	0	peel pulp	0.13 nd	nd nd	0.13 nd	
Trial 3				7	peel pulp	0.2 0.02	0.01 nd	0.21 0.02	
				14	peel pulp	0.41 *	0.01 nd	0.42 *	
				21	peel pulp	0.36 nd	0.01 nd	0.37 nd	
		400	spray	0	peel pulp	0.28 nd	<0.01 nd	0.28 nd	
				7	peel pulp	0.56 0.02	0.02 <0.01	0.58 0.02	
				14	peel pulp	0.59 0.03	0.03 0.01	0.6 0.04	
				21	peel pulp	0.72 0.02	<0.01 nd	0.72 0.02	
USA, CA 1991	2EC	200	spray	0	peel pulp	0.03 nd	<0.01 nd	0.03 nd	
Trial 4				7	peel pulp	0.02 nd	nd nd	0.02 nd	
				14	peel pulp	0.03 *	nd nd	0.03 *	
				21	peel pulp	0.02 nd	nd nd	0.02 nd	
		200	dip	0	peel pulp	0.67 nd	<0.01 nd	0.67 nd	

Country, State, Year	Application			Storage period, days	Sample	Residue, mg/kg			Ref.
	Form	mg ai/l	Method			Myclob.	HD-9090	Total	
				7	peel pulp	0.93 0.03	0.01 <0.01	0.94 0.03	
				14	peel pulp	1.12 0.03	0.02 nd	1.14 0.03	
				21	peel pulp	0.99 0.03	0.03 nd	1.02 0.3	
	2EC	400	spray	0	peel pulp	nd nd	nd nd	nd nd	
				7	peel pulp	<0.01 0.02	nd nd	<0.01 0.02	
				14	peel pulp	0.06 0.01	nd nd	0.06 0.01	
				21	peel pulp	0.13 <0.01	nd nd	0.13 <0.01	
	2EC	400	dip	0	peel pulp	1.32 0.01	0.06 nd	1.38 0.01	
				7	peel pulp	1.71 0.01	0.03 0.01	1.74 0.02	
				14	peel pulp	1.6 0.04	0.03 0.01	1.63 0.05	
				21	peel pulp	1.43 0.05	0.04 <0.01	1.47 0.05	
Hawaii, Kurtistown, 1992	2EC	200	spray	0	peel pulp whole	1.02 0.01	nd nd	1.02 0.01 0.48	Bartra and Zogomski 1993
				7	peel pulp whole	1.14 0.09	0.03 nd	1.17 0.09 0.54	
				14	peel pulp whole	1.14 0.1	0.04 nd	1.18 0.1 0.55	
				21	peel pulp whole	1.28 0.09	0.04 0.05	1.32 0.14 0.55	
				28	peel pulp whole	1.16 0.07	0.04 0.04	1.2 0.11 0.49	
	2EC	400	spray	0	peel pulp whole	1.93 0.03	0.01 nd	1.94 0.03 0.89	
				7	peel pulp whole	2.21 0.19	0.06 0.01	2.27 0.2 1.07	
				14	peel pulp whole	1.76 0.19	0.06 0.02	1.82 0.21 0.85	
				21	peel pulp whole	2.25 0.22	0.08 0.06	2.33 0.28 1.06	
				28	peel pulp whole	1.99 0.16	0.08 0.07	2.07 0.23 0.86	
Hawaii, Hilo 1992	2EC	200	dip	0	peel pulp whole	1.34 0.02	nd nd	1.34 0.02 0.61	
				14	peel pulp	1.14 0.11	0.04 nd	1.18 0.11	

Country, State, Year	Application			Storage period, days	Sample	Residue, mg/kg			Ref.
	Form	mg ai/l	Method			Myclob.	HD-9090	Total	
					whole			0.54	
				28	peel pulp whole	0.98 0.17	0.08 0.02	1.06 0.19 0.49	
	2EC	400	dip	0	peel pulp whole	2.04 0.03	0.01 nd	2.05 0.03 0.9	
				14	peel pulp whole	1.94 0.21	0.06 0.01	2.0 0.22 0.93	
				28	peel pulp whole	1.53 0.19	0.09 0.08	1.62 0.27 0.69	
	2EC	400	spray	0	peel pulp whole	2.56 0.05	0.01 nd	2.57 0.05 1.13	
				14	peel pulp whole	2.27 0.25	0.07 0.01	2.34 0.26 1.06	
				28	peel pulp whole	3.65 0.27	0.12 0.05	3.77 0.32 1.36	
	40WP	400	spray	0	peel pulp whole	1.32 0.02	nd nd	1.32 0.02 0.62	
				14	peel pulp whole	1.38 0.2	0.06 0.01	1.44 0.21 0.69	
				28	peel pulp whole	1.48 0.18	0.07 0.04	1.56 0.22 0.69	
	40WP	400	dip	0	peel pulp whole	1.58 0.03	0.01 nd	1.59 0.03 0.71	
				14	peel pulp whole	1.27 0.17	0.05 0.03	1.32 0.2 0.6	
				28	peel pulp whole	1.6 0.12	0.07 0.02	1.67 0.14 0.64	
Hawaii Hilo, 1992	2EC	400	spray	0	peel pulp whole	2.26 0.02	nd nd	2.26 0.02 1.03	
				7	peel pulp whole	2.72 0.1	0.03 nd	2.75 0.1 1.24	
				14	peel pulp whole	2.08 0.12	0.05 0.01	2.13 0.13 0.97	
				21	peel pulp whole	2.84 0.17	0.06 0.01	2.9 0.18 1.24	
				28	peel pulp whole	2.58 0.15	0.07 0.02	2.65 0.17 1.07	
	40WP	400	spray	0	peel pulp whole	1.56 0.02	nd nd	1.56 0.02 0.73	

Country, State, Year	Application			Storage period, days	Sample	Residue, mg/kg			Ref.
	Form	mg ai/l	Method			Myclob.	HD-9090	Total	
				14	peel pulp whole	1.45 0.19	0.05 0.01	1.5 0.2 0.75	
				28	peel pulp whole	1.67 0.1	0.05 0.02	1.72 0.12 0.68	
Hawaii Hilo 1992	2EC	400	spray	0	peel pulp whole	2.72 0.04	0.01 nd	2.73 0.04 1.25	
				14	peel pulp whole	2.26 0.22	0.07 0.02	2.32 0.24 1.03	
				28	peel pulp whole	3.24 0.28	0.11 0.03	3.35 0.31 1.31	
	40WP	400	spray	0	peel pulp whole	1.8 0.03	nd nd	1.8 0.03 0.82	
				14	peel pulp whole	2.33 0.21	0.06 0.03	2.39 0.24 1.02	
				28	peel pulp whole	2.67 0.41	0.08 0.04	2.75 0.45 1.14	
Costa Rica 1993 2	2EC	200	spray	7	peel pulp whole	1.11 0.1	0.02 nd	1.13 0.1 0.22	Bartra 1994
				14	peel pulp whole	1.2 0.1	0.03 nd	1.23 0.1 0.3	
				21	peel pulp whole	1.32 0.14	0.05 0.01	1.37 0.15 0.25	
	2EC	400	spray	7	peel pulp whole	3.26 0.23	0.04 nd	3.3 0.23 1.52	
				14	peel pulp whole	3.62 0.31	0.06 0.02	3.7 0.33 1.64	
				21	peel pulp whole	3.77 0.35	0.08 0.02	3.85 0.37 1.64	
	2EC	200	spray	7	peel pulp whole	1.17 0.09	0.02 <0.01	1.19 0.09 0.56	
				14	peel pulp whole	1.07 0.11	0.01 nd	1.08 0.11 0.49	
				21	peel pulp whole	1.57 0.15	0.03 0.01	1.6 0.16 0.69	
		400	spray	7	peel pulp whole	2.95 0.17	0.03 nd	2.98 0.17 1.35	
				14	peel pulp whole	2.81 0.29	0.06 0.01	2.87 0.3 1.3	
				21	peel	2.91	0.06	2.97	

Country, State, Year	Application			Storage period, days	Sample	Residue, mg/kg			Ref.
	Form	mg ai/l	Method			Myclob.	HD-9090	Total	
					pulp whole	0.26	0.02	0.28 1.26	
Costa Rica St.1 1993	2EC	200	spray	7	peel pulp whole	0.99 0.12	0.02 nd	1.01 0.12 0.5	
				14	peel pulp whole	1.33 0.12	0.03 nd	1.36 0.12 0.61	
				21	peel pulp whole	1.22 0.13	0.06 nd	1.28 0.13 0.55	
	2EC	400	spray	7	peel pulp whole	2.7 0.31	0.04 nd	2.74 0.31 1.33	
				14	peel pulp whole	2.86 0.29	0.05 0.02	2.91 0.31 1.33	
				21	peel pulp whole	3.83 0.39	0.09 0.02	3.92 0.41 1.68	

*lack of residue data

Citrus fruit

GAP exists in Spain for post-harvest spraying with a water wax emulsion or spraying or drenching with an emulsifiable concentrate, to control Penicillium. Summary reports of trials in Spain on mandarins and oranges were submitted to the Meeting, but lacked critical analytical data such as LODs, recoveries and chromatograms.

Mandarins. Twelve trials were conducted using 3 g/l water wax emulsion or 120 g/l emulsifiable concentrate formulations as drenches. Only myclobutanil was determined.

Five trials were with a water wax emulsion.Residues of myclobutanil in the whole fruit were 2.6-2.9 mg/kg at day 0 (Elf. Atochem, 1996c).

Seven trials were carried out with the EC formulation applied at 0.05 kg ai/hl. In two of them whole fruit were separated into peel, pulp and juice fractions. Residues of myclobutanil in whole fruit at day 0 ranged from 0.94 to 2 mg/kg (Elf Atochem, 1996b).

Table 7. Myclobutanil residues in mandarins from supervised trials in Spain after one post-harvest treatment. Underlined residues are from treatments according to GAP. All drench applications (Elf Atochem, 1996b,c).

Trial no./Year	Application			Sample	Days after treatment	Myclobutanil, mg/kg
	Form.	kg ai/t fruit	kg ai/hl			
5/95	EC		0.05	whole[1]	0	1.15
					30	1.40
					45	1.0
				peel	0	2.75
					30	3.40
					45	2.0
				pulp	0	nd

Trial no./Year	Application			Sample	Days after treatment	Myclobutanil, mg/kg
	Form.	kg ai/t fruit	kg ai/hl			
					30	nd
					45	nd
				juice	0	0.1
					30	nd
					45	nd
6/1995	EC		0.05	whole	0	<u>0.94</u>
					30	1.5
					45	1.1
				peel	0	3.35
					30	3.8
					45	3.1
				pulp	0	nd
					30	nd
					45	nd
				juice	0	0.09
					30	nd
					45	nd
5/1994	EC		0.05	whole fruit	0	<u>2.0</u>
					7	1.7
					14	1.1
7/1994	EC		0.05	whole fruit	0	<u>1.56</u>
10/1994	EC		0.05	whole fruit	0	<u>1.33</u>
					7	1.1
					14	0.57
11/1994	EC		0.05	whole	0	<u>1.5</u>
					7	1.2
					14	1.1
12/1994	EC		0.05	whole	0	<u>1.7</u>
					7	1.2
					14	1.0
1994	WWE	0.003		whole	0	<u>2.6</u>
					7	2.35
					14	2.26
1994	WWE	0.003		whole	0	<u>2.8</u>
					7	2.54
					14	2.36
1994	WWE	0.003		whole	0	<u>2.75</u>
					7	2.18
					14	2.4
1994	WWE	0.003		whole	0	<u>2.72</u>
					7	2.53
					14	2.51
1994	WWE	0.003		whole	0	<u>2.9</u>

<u>Oranges</u>. Fifteen trials were carried out in 1994 and 1995. Five were on Valencia late oranges with a water wax emulsion at 0.003 kg ai/tonne of fruit. Whole fruits were separated into peel, pulp and juice fractions which were analysed for myclobutanil only. Residues were found only in the peel (Elf. Atochem, 1996a).

Other trials in 1994 on different varieties of orange were with the water wax emulsion or EC formulation according to Spanish GAP. Fruit treated with the wax emulsion were analysed only at day 0, but those treated with the EC were analysed at intervals. Only whole fruit were analysed and metabolites were not determined (Elf Atochem, 1996c).

The residues of myclobutanil in all the trials ranged from 0.87 to 2.66 mg/kg. The results are shown in Table 8.

Table 8. Myclobutanil residues in oranges from supervised trials in Spain after one post-harvest treatment. The underlined residues are from treatments according to GAP. All spray applications (Elf Atochem, 1996a,c).

Year, Trial no.	Form.	Application		Sample	PHI, days	Myclobutanil, mg/kg
		kg ai/t fruit	kg ai/hl			
1995 9/95	WWE	0.003		whole fruit	0	2.3
					30	1.99
					60	2.6
					90	1.88
				peel	0	7.1
					30	5.03
					60	7.1
					90	6.0
				pulp	0	nd
					30	nd
					60	nd
					90	nd
				juice	0	nd
					30	nd
					60	nd
					90	nd
1995 10/95	WWE	0.003		whole fruit	0	0.87
					30	1.04
					60	1.66
					90	0.74
				peel	0	1.77
					30	3.2
					60	4.6
					90	0.74
				pulp	0	nd
					30	nd
					60	nd
					90	nd
				juice	0	nd
					30	nd
					60	nd
					90	nd
1995 11/95	WWE	0.003		whole fruit	0	1.47
					30	1.44
					60	0.8
					90	1.24
				peel	0	3.8
					30	5.5
					60	3.0
					90	3.8
				pulp	0	nd
					30	nd

Year, Trial no.	Form.	Application		Sample	PHI, days	Myclobutanil, mg/kg
		kg ai/t fruit	kg ai/hl			
					60	nd
					90	nd
				juice	0	nd
					30	nd
					60	nd
					90	nd
1995	WWE	0.003		Whole fruit	0	2.6
12/95					30	2.6
					60	2.0
					90	1.8
				peel	0	6.6
					30	6.5
					60	6.1
					90	2.18
				pulp	0	nd
					30	nd
					60	nd
					90	nd
				juice	0	nd
					30	nd
					60	nd
					90	nd
1995	WWE	0.003		whole fruit	0	1.23
13/95					30	1.6
					60	1.98
					90	1.9
				peel	0	3.22
					30	5.0
					60	7.8
					90	4.8
				pulp	0	nd
					30	nd
					60	nd
					90	nd
				juice	0	nd
					30	nd
					60	nd
					90	nd
14/94	WWE	0.003		whole fruit	0	2.63
15/94	WWE	0.003		Whole fruit	0	2.36
20/94	WWE	0.003		whole fruit	0	2.43
22/94	WWE	0.003		whole fruit	0	2.66
1995 7/95	EC		0.05	whole fruit	0	1.3
					30	1.6
					45	1.0
				peel	0	4.22
					30	3.0
					45	3.4
				pulp	0	nd
					30	nd
					45	nd
				juice	0	0.1
					30	nd
					45	nd
1995	EC		0.05	whole fruit	0	1.06

Year, Trial no.	Form.	Application		Sample	PHI, days	Myclobutanil, mg/kg
		kg ai/t fruit	kg ai/hl			
					30	1.16
					45	1.1
				peel	0	3.5
					30	3.4
					45	3.3
				pulp	0	nd
					30	nd
					45	nd
				juice	0	0.7
					30	nd
					45	nd
1994 6/94	EC		0.05	whole fruit	0	1.8
					7	1.7
					14	0.7
1994 8/94	EC		0.05	whole fruit	0	1.36
1994	EC		0.05	whole fruit	0	1.49
9/94					7	1.37
					14	0.69
1994	EC		0.05	whole fruit	0	1.53
13/94					7	1.2
					14	1.1

Berries

Residue trials on blackcurrants and strawberries were carried out in France, Italy, Spain and the UK.

Blackcurrants. Eighteen field trials in the UK and one in France between 1990 and 1995 were reported. Four of the trials in 1995 were on commercial crops at a range of sites in the UK. Three formulations were used to determine whether the formulation type or concentration had any bearing on residue levels. Myclobutanil 60 g/l SC, 6 WP or 200 g/l EW was applied at the GAP rate of 0.09 kg ai/ha at 11-14 day intervals, using a Krist Mistblower. Three trials in 1991 were residue decline trials, also at 0.09 kg ai/ha. The plots were 5 m rows and myclobutanil was applied with a motor knapsack sprayer at 6- to 20-day intervals. Samples were stored at -20°C and the SAI was 6-10 months.

In all the UK trials the samples were analysed for the parent compound and metabolites as RH-9090. The LOD for both compounds was 0.01 mg/kg and recoveries were >80%.

Residues of myclobutanil in blackcurrant samples harvested at 13-17 days PHI in approximate accordance with UK GAP ranged from 0.04 to 0.43 mg/kg and those of RH-9090 were between 0.02 and 0.19 mg/kg. Total residues were 0.08-0.47 mg/kg.

In the trial in France blackcurrants were treated with myclobutanil 12 EC at 0.062 kg ai/ha. Samples were taken 7, 21, and 35 days after the last treatment.

The results are given in Table 9.

Table 9. Myclobutanil residues in blackcurrants. Underlined residues are from trials according to GAP and were used to estimate maximum residue levels.

Country, Location, Year	Application				PHI, days	Residues, mg/kg			Ref.
	Form	No	kg ai/hl	kg ai/ha		Myclob.	RH-9090	Total	
England, 1991	6 SC	5	0.0045	0.09	0	0.72	0.67	1.39	Murray, 1993b
					7	0.26	0.66	0.92	
					13	0.07	0.19	0.26	
					20	0.13	0.29	0.42	
			0.009	0.18	20	0.1	0.1	0.2	
England, 1991	6SC	5	0.0045	0.09	0	0.57	0.16	0.73	
					8	0.31	0.64	0.95	
					17	0.08	0.14	0.22	
					21	0.2	0.09	0.29	
			0.009	0.18	21	0.22	0.20	0.42	
England, 1991	6SC	5	0.0045	0.09	0	0.52	<0.01	0.54	
					7	0.11	0.03	0.14	
					13	0.04	0.04	0.08	
					20	0.16	0.05	0.21	
England, 1990	6SC	6	0.006	0.12	12	0.57	0.22	0.79	Murray, 1993a
				Control	12	0.01	0.03	0.04	
England, 1990	6SC	6	0.006	0.12	16	0.46	0.13	0.59	
				Control		0.02	0.06	0.08	
England, 1990	6SC	6	0.006	0.12	16	0.21	0.11	0.32	
				Control		0.03	0.13	0.16	
England, Staffordshire, 1995	6SC	6		0.09	14	0.35	0.04	0.39	Agrisearch, 1996a
England, Worcestshire	6SC	6		0.09	14	0.3	0.05	0.35	
England, Kent 1995	6SC	6		0.09	14	0.19	0.02	0.21	
England, Kent 1995	6SC	6		0.09	14	0.24	0.04	0.28	
England, Staffordshire 1995	6WP	6		0.09	14	0.42	0.04	0.46	Agrisearch, 1996b
England, Worcs. 1995	6WP	6		0.09	14	0.29	0.05	0.34	
England, Kent 1995	6WP	6		0.09	14	0.31	0.03	0.34	
	6WP	6		0.09	14	0.26	0.05	0.31	
England, Staffordshire 1995	20EW	6		0.09	14	0.43	0.04	0.47	Agrisearch 1996c
England, Worcestshire 1995	20EW	6		0.09	14	0.3	0.07	0.37	
England, Kent 1995	20EW	6		0.09	14	0.24	0.04	0.28	
England, Kent 1995	20EW	6		0.09	14	0.26	0.05	0.31	
France 1992	12EC	3		0.0625	7	0.08			Anadiag, 1994a
					21	0.07			
					35	0.04			

Strawberries (Table 10). Several residue trials in the UK between 1990 and 1996 were reported. Two were carried out in Kent in 1990 to determine the rate of decline of myclobutanil and its metabolites determined as RH-9090. Myclobutanil SC was applied at 10- to 23-day intervals. Samples were stored frozen at -18°C for 11 months before analysis (Murray,1993c).

Four other trials were conducted at 4 sites in the UK, two in England and two in Scotland. Myclobutanil was applied five times at 11-14-day intervals (Murray, 1994).

Another four sets of three field trials were on commercial crops at a range sites in England. Each set consisted of an untreated plot and three plots (2 x 10 m rows) treated with different formulations. The treated plots were sprayed six times, with the first spray applied approximately 12 weeks before the expected harvest and the following five application at 9-16 day intervals. The SAI was 154 days (Agrisearch, 1996d-f).

Two other trials were conducted in England in 1996 to determine myclobutanil residues in strawberries, strawberry jam and strawberry preserve (Huntingdon, 1997a).

Myclobutanil residues in strawberries in the UK trials which approximated GAP ranged from 0.08 to 0.69 mg/kg.

Summary data from supervised trials in France, Italy and Spain during 1988 and 1989 were reported to the Meeting.

Other residue decline trials were conducted in 1992 and 1993 in Italy and Spain at 0.07-0.09 kg ai/ha. Samples from the Spanish trials were analysed only for myclobutanil. Residues of RH-9090 in the Italian trial after 7 days were below the LOD.

Table 10. Residues in strawberries from supervised trials. Underlined residues are from trials according to GAP and are used to estimate maximum residue levels.

Country, Year, Trial	Application				PHI, days	Residues, mg/kg		Ref.
	Form	No.	kg ai/hl	kg ai/ha		Myclo-butanil	RH-9090	
England 1990	6SC	6		0.09	0	1.21	0.07	Murray, 1993c
					5	<u>0.36</u>	<u><0.01</u>	
					8	0.11	<0.01	
					15	0.07	<0.01	
England 1990	6SC	6		0.09	0	0.25	0.08	Murray 1993c
					7	0.23	<0.01	
					14	0.03	0.06	
England 1993	6SC	5		0.09	0	0.36	<0.01	Murray 1994
					3	<u>0.48</u>	<u><0.01</u>	
					7	0.12	<0.01	
					14	0.02	<0.01	
				0.18	0	1.01	<0.01	
					7	0.2	<0.01	
England 1993	6SC	5		0.09	0	0.48	<0.01	Murray, 1994
					3	<u>0.69</u>	<u><0.01</u>	
					7	0.28	<0.01	
					14	0.03	<0.01	
Scotland 1993	6SC	5		0.09	0	0.38	<0.01	Murray 1994
					3	<u>0.24</u>	<u><0.01</u>	
					7	0.09	<0.01	

Country, Year, Trial	Application				PHI, days	Residues, mg/kg		Ref.
	Form	No.	kg ai/hl	kg ai/ha		Myclo-butanil	RH-9090	
					14	0.04	<0.01	
Scotland 1993	6SC	5		0.09	0	0.83	<0.01	Murray 1994
					3	0.5	<0.01	
					7	0.27	<0.01	
					14	0.06	<0.01	
England 1995 RH/1	6SC	6		0.09	3	0.2	<0.01	Agrisearch 1996f
England 1995 RH/2	6SC	6		0.09	3	0.19	<0.01	
England 1995 RH/3	6SC	6		0.09	3	0.22	<0.01	
England 1995 RH/4	6SC	6		0.09	3	0.19	<0.01	
England 1995 RH/1	6WP	6		0.09	3	0.17	<0.01	Agrisearch 1996e
England 1995 RH/2	6WP	6		0.09	3	0.2	<0.01	
England 1995 RH/3	6WP	6		0.09	3	0.15	<0.01	
England 1995 RH/4	6WP	6		0.09	3	0.18	<0.01	
England 1995 RH/1	20EW	6		0.09	3	0.18	<0.01	Agrisearch 1996d
England 1995 RH/2	20EW	6		0.09	3	0.19	<0.01	
England 1995 RH/3	20EW	6		0.09	3	0.1	<0.01	
England 1995 RH/4	20EW	6		0.09	3	0.19	<0.01	
England 1996	20EW	6	0.05	0.076	0	0.12	<0.01	Huntingdon, 1997a
					3	0.08	<0.01	
England 1996	20EW	6	0.05	0.082	0	0.15	<0.01	
					3	0.08	<0.01	
France 1989	EC	8		0.0625	0	0.06		Procida, 1989
					4	0.04		
					7	0.03		
					14	0.02		
					28	<0.01		
Italy 1988	EC	3	0.005	0.0375	7	0.09	<0.01	Pessina, 1990
		3	0.01	0.075	7	0.11	<0.01	
Italy 1993	EC	3	0.0062	0.067	0	0.07	0.01	Pessina 1995
					3	0.07	<0.01	
					7	0.05	<0.01	
Spain 1988	EC	5	0.0062	0.093	3	0.12		Jousseaume 1988
					7	0.07		
					14	0.04		
		5	0.0075	0.112	3	0.15		
					7	0.08		
					14	0.05		
Spain 1992	EC	2	0.011	0.0875	0	0.2		Anadiag. 1993a
					3	0.15		

Country, Year, Trial	Application				PHI, days	Residues, mg/kg		Ref.
	Form	No.	kg ai/hl	kg ai/ha		Myclo-butanil	RH-9090	
					7	0.08		
					13	0.08		
		1	0.011	0.0875	0	0.2		
					3	0.07		
					7	0.12		
					13	0.03		

Tomatoes. In nine trials on greenhouse tomatoes in Belgium in 1995, three formulations of myclobutanil (EC, SC and WP) were applied according to GAP at 0.0075 kg ai/hl to the point of run-off. Residues of myclobutanil and its metabolites were determined by GLC with an ECD. The LOD was 0.01 mg/kg for myclobutanil and 0.02 mg/kg for RH-9090. Samples were taken 3 and 7 days after the last treatment (Phytopharmacie, 1996a-c). Residues of myclobutanil were 0.05-0.15 mg/kg; RH-9090 was not detected.

Only summary data from two field trials carried out in France in 1990 were submitted to the Meeting. Eight application of myclobutanil EC at 0.075 kg ai/ha were made at 10-12 day intervals. The metabolites were not determined (Herisse, 1990,a,b). In four other trials in Southern France in 1996 to determine myclobutanil and its metabolites in whole tomatoes, juice, preserve and purée tomatoes were treated with myclobutanil 240 g/l EC or 200 g/l EW. The first applications were made 11 weeks before harvest and the other five at 10-14 day intervals. The SAIs were 320 to 348 days.

In a field trial in Italy (1993) to determine residue decline on industrial tomatoes, myclobutanil EC was sprayed three times to run-off at 0.00625 kg ai/hl at 7-day intervals and samples were collected at 0, 7 and 14 days after the last treatment. No RH-9090 was found in any of the analysed samples (LOD 0.01 mg/kg). The SAI was 16 months. Other trials in Italy in 1996 on two varieties were with 6 applications of myclobutanil 240 g/l EC or 200 g/l SC: only 2-4 applications are approved according to Italian GAP. The first application was made 11 weeks before harvest and the other five at 10-14 day intervals. The SAIs were 320 to 348 days.

Two trials were carried out in Morocco in 1992 and six (three field and three glasshouse) in 1993. In 1993 1, 3 or 4 applications of myclobutanil EC were made at 0.00625 kg ai/hl. The SAI ranged from 57 to 104 days. RH-9090 was determined in 1993 but not found above the LOD.

Fourteen residue trials were conducted in Spain between 1984 and 1993, five of them to measure residue decline.The trials in 1984-1986 were outdoor and only myclobutanil was determined. The trials in 1993 were indoors and RH-9090 was also determined. Residues of myclobutanil in samples of whole fruit from trials complying with GAP ranged from 0.03 to 0.24 mg/kg.

Reported US GAP indicates that myclobutanil 40 WP can be applied by ground or aerial spray to unripe fruit at an application rate of 0.07 kg ai/ha with a maximum of 0.4 kg ai/ha per season and an application interval of 11-21 days. Application may be made up to the day of harvest for the fresh market and with a 5-day PHI for varieties used in processing. A total of 21 trials was reported from California, Indiana, Ohio, Michigan, South Carolina, New Jersey and Florida, where myclobutanil was applied to different varieties of tomato. The fruit were harvested at day 0, and in some cases also at 5, 10 and 15 days after the last treatment, and analysed for the parent compound and total metabolites. Residues of the parent compound ranged from 0.01 to 0.22 mg/kg at day 0. Residues of metabolite were detected in only two trials which complied with GAP as a low proportion of the parent compound.

The results are shown in Table 11.

Table 11. Myclobutanil residues in tomatoes (whole fruit) from supervised trials. The underlined residues are from treatments according to GAP and were used to estimate maximum residue levels.

Country, Year, Trial no.	Application				PHI, days	Residues, mg/kg		Ref.
	Form.	No	kg ai/hl	kg ai/ha		Myclobutanil	RH-9090	
Belgium 1995 9131/1	WP	4 g	0.0075		3 7	0.09 0.1	nd nd	Phyto-pharmacie, 1996c
Belgium 1995 9131/2	WP	4 g	0.0075		3 7	0.16 0.11	nd nd	
Belgium 1995 9131/3	WP	4 g	0.0075		3 7	0.06 0.14	nd nd	
Belgium 1995 9131/1	EC	4 g	0.0075		3 7	0.08 0.09	nd nd	Phyto-pharmacie, 1996a
Belgium 1995 9131/2	EC	4 g	0.0075		3 7	0.15 0.08	nd nd	
Belgium 1995 9131/3	EC	4 g	0.0075		3 7	0.05 0.08	nd nd	
Belgium 1995 9131/1	EW	4 g	0.0075		3 7	0.11 0.12	nd nd	Phyto-pharmacie, 1996 b
Belgium 1995 9131/2	EW	4 g	0.0075		3 7	0.15 0.1	nd nd	
Belgium 1995 9131/3	EW	4 g	0.0075		3 7	0.08 0.12	nd nd	
France 1990	EC	8	0.015	0.075	0	0.13		Herisse, 1990a
					2	0.12		
					4	0.02		
					7	0.02		
France 1990	EC	8	0.015	0.075	0	0.06		Herisse, 1990b.
					2	0.09		
					4	0.09		
					7	0.03		
France, Aucamville 1996	SC	6	0.0075	<0.107	0	0.07	nd	Huntingdon, 1997d
					3	0.04	nd	
					7	0.02	nd	
France, Bressoles 1996	SC	6	0.0075	<0.12	0	0.11	<0.01	
					3	0.05	nd	
					7	0.01	nd	
France, Aucamville 1996	EC	6	0.0075	<0.107	0	0.05	nd	Huntingdon, 1997c
					3	0.02	nd	
					7	0.03	nd	
France, Bressols 1996	EC	6	0.0075	<0.12	0	0.05	nd	
					3	0.03	nd	
					7	0.03	nd	
Italy 1993	EC	3	0.00625	0.0641	0	0.08	<0.01	Pessina, 1996
					7	0.02	<0.01	
					14	0.03	<0.01	
Italy 1996	EC	6	0.0075	<0.114	0	0.24	<0.01	Huntingdon, 1996c
					3	0.18	<0.01	
					7	0.05	<0.01	
Italy 1996	EC	6	0.0075	<0.113	0	0.24	<0.01	
					3	0.18	<0.01	

Country, Year, Trial no.	Application				PHI, days	Residues, mg/kg		Ref.
	Form.	No	kg ai/hl	kg ai/ha		Myclobutanil	RH-9090	
					7	0.06	<0.01	
Italy 1996	SC	6	0.0075	<0.112	0	0.22	<0.01	Huntingdon, 1997d
					3	0.18	<0.01	
					7	0.08	<0.01	
Italy 1996	SC	6	0.0075	<0.112	0	0.24	<0.01	
					3	0.18	<0.01	
					7	0.06	<0.01	
Morocco 1992	EC	4 f	0.00625	0.0625	0	0.1		Anadiag, 1993b
					3	0.04		
					5	0.05		
					7	0.04		
Morocco 1992	EC	4	0.00625		0	0.04		Anadiag, 1993c
					3	0.06		
					5	<0.02		
					7	<0.02		
Morocco 1993	EC	1 f	0.00625	0.094	0	0.06	<0.01	Anadiag, 1994d
	EC	3 f	0.00625	0.094	7	0.09	<0.01	
	EC	4 f	0.00625	0.094	0	0.04	<0.01	
					3	0.02	<0.01	
					7	0.02	<0.01	
					14	0.01	<0.01	
Morocco 1993	EC	1 g	0.00625	0.094	0	0.03	<0.01	Anadiag, 1994e
	EC	3 g	0.00625	0.094	7	0.06	<0.01	
	EC	4 g	0.00625	0.094	0	0.11	<0.01	
					3	0.05	<0.01	
					7	0.04	<0.01	
					14	0.03	<0.01	
Spain 1984	EC	6	0.009	0.08	6	0.04		Jousseaume, 1986c
Spain Barcelona 1985	EC	8 f	0.008	0.240	0	0.06		Jousseaume, 1995
					3	0.04		
					5	0.03		
					8	0.02		
					15	0.02		
					22	0.02		
	EC	5	0.008	0.240	8	0.06		
		3	0.008	0.240	8	0.03		
		2	0.008	0.240	15	0.03		
Spain Barcelona 1986	EC	5 f	0.008	0.02	0	0.1		Jousseaume, 1986a
					2	0.15		
					4	0.07		
					8	0.1		
					16	0.05		
	EC	4 f	0.16	0.04	0	0.25		
					2	0.3		
					4	0.4		
					8	0.1		

Country, Year, Trial no.	Application				PHI, days	Residues, mg/kg		Ref.
	Form.	No	kg ai/hl	kg ai/ha		Myclobuta nil	RH-9090	
Spain Barcelona 1986	EC	1 f	0.012	0.08	3	<0.01		Jousseaume, 1986b
					5	0.01		
					7	<0.01		
					9	<0.01		
					12	<0.01		
	EC	1	0.024	0.16	5	0.03		
					9	0.01		
					12	<0.01		
Spain 1993	EC	1 g	0.012	0.131	0	0.03	<0.01	Anadiag, 1994c
					7	0.02	0.02	
		2 g	0.012	0.131	7	0.02	<0.01	
		3 g	0.012	0.131	0	0.02	<0.01	
					4	0.03	<0.01	
					7	0.03	<0.01	
					14	0.04	0.02	
Spain 1993	EC	3 g	0.0083	0.131	0	0.16	0.01	Anadiag, 1994b
					3	0.24	0.02	
					7	0.16	0.03	
					14	0.04	0.02	
		2	0.0083	0.131	8	0.23	<0.01	
USA, CA 1989	WP	4 f		0.067	0	0.05	<0.01	Stavinski and Filchner, 1991
					5	0.03	<0.01	
USA, CA 1989	WP	4 f		0.07	0	0.07	<0.01	
					5	0.07	<0.01	
USA, CA 1989	WP	4 f		0.07	0	0.05	<0.01	
USA, IN 1989	WP	5 f		0.067	0	0.05	<0.01	
					5	0.04	<0.01	
USA, OH 1989	WP	5 f		0.067	0	0.05	<0.01	
					5	0.05	<0.01	
USA, MI 1989	WP	4 f		0.067	0	0.08	<0.01	
					5	0.03	<0.01	
USA, SC 1989	WP	4 f		0.067	0	0.08	<0.01	
					5	0.04	<0.01	
USA, NJ 1989	WP	5 f		0.07	0	0.03	<0.01	
					5	0.03	<0.01	
USA, FL 1990	WP	4 f		0.093	0	0.22	0.03	Ding & Zogorski, 1992
USA, CA 1993	WP	4 f		0.07	0	0.01	<0.01	Bartra 1995b
					5	0.01	<0.01	
					10	0.01	<0.01	
	WP	4		0.14	0	0.02	<0.01	
USA, NJ 1993	WP	4 f		0.07	0	0.05	0.01	

Country, Year, Trial no.	Application				PHI, days	Residues, mg/kg		Ref.
	Form.	No	kg ai/hl	kg ai/ha		Myclobutanil	RH-9090	
	WP	4 f		0.14	0	0.1	0.02	
USA, CA 1993	WP	4 f		0.07	0	0.03	<0.01	
	WP	4 f		0.14	0	0.08	0.02	
USA, OH 1993	WP	5 f		0.07	0	0.02	<0.01	
	WP	5 f		0.14	0	0.07	0.02	
USA, FL 1993	WP	4 f		0.07	0	0.02	<0.01	
					5	0.01	<0.01	
					10	0.01	0.01	
	WP	4 f		0.14	0	0.07	0.02	
USA, FL 1993	WP	5 f		0.07	0	0.02	<0.01	
	WP	5 f		0.14	0	0.04	<0.01	

f: field; g: glasshouse

Hops. In six trials in England reported to the Meeting myclobutanil EW was sprayed six times at 0.0045 kg ai/hl to the point of run-off. Residues of myclobutanil in samples of dried cones from trials complying with GAP, taken at 7-13 days PHI, ranged from 0.3 to 1.2 mg/kg. The residues of RH-9090 were all below the LOD (0.2 mg/kg). The results are shown in Table 12.

Table 12. Myclobutanil residues in hops (dried cones) from supervised trials in England (Nat. Hop Assoc., 1995).

Year	Application				PHI, days	Residues, mg/kg	
	Form	No	kg ai/hl	kg ai/ha		Myclobutanil	RH-9090
1991	EW	6	0.0045	0.101	7	0.5	<0.2
1991	EW	6	0.0045	0.101	13	0.3	<0.2
1992	EW	6	0.0045	0.103	8	1.2	<0.2
1992	EW	6	0.0045	0.103	9	0.27	<0.2
1991	EW	6	0.0045	0.103	3	1.8	<0.2
1991	EW	6	0.0045	0.09	4	1.6	<0.2

FATE OF RESIDUES IN STORAGE AND PROCESSING

In storage

No information was available.

In processing

Processing studies on tomatoes in France and the USA and on blackcurrants in the UK were reported to the Meeting.

<u>Tomatoes</u> treated at two levels were processed into various products simulating commercial practice as closely as possible in the USA (Stavinski *et al*, 1991). Samples harvested 5 days after the last of 4 applications at 0.067 and 0.14 kg ai/ha were washed and processed into canned whole tomatoes, juice, purée, wet pomace, dry pomace and paste.

Four processing studies were conducted in France in 1996. Tomatoes treated 6 times with myclobutanil 240 g/l EC or 200 g/l EW at 0.107 and 0.12 kg ai/ha and harvested 3 days after the last treatment were processed into juice, preserve and purée (Huntingdon, 1997c,d).

The results are shown in Table 13.

Table 13. Residues of myclobutanil in tomatoes and their processed products in the USA (Stavinski *et al.*, 1991) and France (Huntingdon, 1997c, d).

Application, kg ai/ha	PHI, days	Sample	Residues, mg/kg			Processing factor (total residue)
			Myclobutanil	RH-9090	Total	
USA						
0.282	5	unwashed fruit	0.03	nd	0.03	-
(0.07x4)		washed fruit	0.02	nd	0.02	0.67
		canned fruit	0.01	0.01	0.02	0.67
		wet pomace	0.25	nd	0.25	8.3
		dry pomace	0.43	nd	0.43	14.3
		juice	0.02	nd	0.02	0.67
		purée	0.02	0.02	0.04	1.3
		paste	0.08	0.03	0.11	3.7
0.56	5	unwashed fruit	0.04	0.02	0.06	-
(0.14 x 4)		washed fruit	0.06	0.02	0.08	1.3
		canned fruit	0.02	0.03	0.05	0.83
		wet pomace	0.026	0.006	0.03	0.5
		dry pomace	1.03	0.01	1.04	17.3
		juice	0.04	0.02	0.06	1.0
		purée	0.05	0.02	0.07	1.17
		paste	0.17	0.08	0.25	4.2
France						
0.107 x 6	3	whole fruit	0.02	nd	0.02	-
		juice	0.02	nd	0.02	1.0
		preserve	<0.01	nd	<0.01	<0.5
		purée	0.06	nd	0.06	3.0
0.12 x 6	3	whole fruit	0.03	nd	0.03	-
		juice	0.01	nd	0.01	0.33
		preserve	<0.01	nd	<0.01	<0.33
		purée	0.06	nd	0.06	2.0
0.107 x 6	3	whole fruit	0.04	nd	0.04	-
		juice	0.01	nd	0.01	0.25
		preserve	0.01	nd	0.01	0.25
		purée	0.04	nd	0.04	1.0
0.12 x 6	3	whole fruit	0.05	nd	0.05	-
		juice	0.01	nd	0.01	0.2
		preserve	<0.01	nd	<0.01	<0.2
		purée	0.05	nd	0.05	1.0

There was no concentration in the juice, preserve or canned tomatoes. Tomato purée showed a mean concentration factor of about 1.6. Only the paste and dry pomace consistently showed high concentration factors, largely due to the dehydration. In the US trials washing produced a slight reduction of the residue from the lower treatment rate and a slight increase at the higher rate, but the residues were too low for this to be significant.

<u>Blackcurrant</u> samples from three trials in England were processed into juice, and in one of the trials into canned fruit. Residues in the juice were lower than in the whole fruit, as were residues of the parent compound in the canned fruit. Residues of RH-9090 were higher than those of myclobutanil in both the juice and canned fruit owing to degradation of the parent compound during processing. The results are given in Table 14.

Table 14. Residues of myclobutanil in fresh and processed blackcurrants in England (Murray, 1993a).

Application			PHI, days	Sample	Residues, mg/kg		
Form.	kg ai/hl	No.			Myclobutanil	RH-9090	Total
6SC	0.006	6	12	whole fruit	0.57	0.22	0.79
				juice	<0.01	0.15	0.15
6SC	0.006	6	16	whole fruit	0.46	0.13	0.59
				juice	0.04	0.2	0.24
				canned fruit	0.09	0.5	0.59
6SC	0.006	6	16	whole fruit	0.21	0.11	0.32
				juice	0.07	0.14	0.21

<u>Strawberries</u>. Two trials were carried out in the UK in 1996 to determine the effect on residues of myclobutanil of processing strawberries treated with myclobutanil 20EW during field trials to preserve and jam. Samples for processing were taken at a 3-day PHI. The results are shown in Table15.

Table 15. Residues of myclobutanil in fresh and processed strawberries in England (Huntingdon, 1997a).

Application			PHI, days	Sample	Residues, mg/kg		Processing factor
Form.	kg ai/hl	No.			Mycobutanil	RH-9090	
20EW	0.05	6	3	whole fruit	0.08	<0.01	-
				jam	0.04	<0.01	0.5
				preserve	0.06	<0.01	0.75
20EW	0.05	6	3	whole fruit	0.08	<0.01	-
				jam	0.04	<0.01	0.5
				preserve	0.07	<0.01	0.88

RESIDUES IN FOOD COMMERCE OR AT CONSUMPTION

No information was provided.

NATIONAL MAXIMUM RESIDUE LIMITS

The following national MRLs were reported.

Country	Commodity	MRL, mg/kg
Belgium	Strawberry	0.5
	Tomato	0.5
France	Apricot	0.3
	Cherry	0.3
	Peach	0.3
	Plums	0.3
	Blackcurrants	1

Country	Commodity	MRL, mg/kg
	Strawberry	0.3
Germany	Strawberry	0.2
	Tomato	0.2
Italy	Peach	0.2
	Strawberry	0.2
Spain	Mandarin	5
	Orange	5
	Stone fruit	0.5
	Strawberry	2
	Tomato	1
Switzerland	Stone fruit	0.2
	Strawberry	0.2
USA	Stone fruit (except cherry)	2
	Cherry	5
	Prune (dried)	8
	Tomato	0.3[1]
	Tomato (paste)	1.2[1]
	Tomato purée	0.6[1]
Yugoslavia	Peaches	0.2

[1] Temporary

APPRAISAL

Myclobutanil is a systemic, foliar-applied fungicide. It was first reviewed by the 1992 JMPR. The MRLs recommended for stone fruits are now adopted as CXLs. At the 28th Session of the CCPR the EC delegation questioned the residue evaluation for stone fruits. The manufacturer provided information on GAP and data on residues for a review of use patterns and a reconsideration of maximum residue levels.

The Meeting received updated information on GAP in EC countries and the USA. The manufacturer provided reports of five supervised trials carried out in the USA on apricots in 1991, and data on residues at a 0-day PHI in cherries, peaches and plums.

The manufacturer also requested the evaluation of data on residues in bananas, blackcurrants, citrus, hops, strawberries and tomatoes. The manufacturer provided data on residues in sweet peppers, but this information was received too late for evaluation.

The analytical methods for determining the residues of myclobutanil and its metabolites in fruits were as described in the 1992 JMPR evaluation.

Studies of the stability of residues in stored analytical samples of soil, apples, grapes, tomatoes, cucurbits and almond meat and hulls were reported to the Meeting. Residues of myclobutanil and its metabolite hydroxy-myclobutanil (RH-9090) were found to be stable in frozen conditions (-15°C) in soil, apples and grapes for at least two years, in cucurbits and tomatoes (at -10°C) for three years and in almond meat and hulls at -10°C for at least two years. It can be concluded that residues of myclobutanil in stored samples are stable in frozen conditions.

Myclobutanil is available as 125 and 240 g/l emulsifiable concentrate, 60 g/l suspension concentrate, 200 g/l emulsion oil in water and 40% wettable powder.

Supervised trials

In all the trials on stone fruits reported to the Meeting the residues were determined in the edible portion of the fruits and the proportional weights of the stones were not given. The average percentage weights of the stones in each of the fruits were reported by the manufacturer and these averages were used to estimate maximum residue levels in the whole fruits.

Apricots. The results of field trials in France, Italy and the USA were provided. The trials from Italy were not considered for the estimation of a maximum residue level as no relevant GAP was reported. Residues from six trials according to GAP in France ranged from 0.01 to 0.08 mg/kg; the residues of the metabolite were not determined. In five trials according to US GAP (7-17 applications at 0.07 to 0.165 kg ai/ha, 0-day PHI), the total residues found were between 0.13 and 0.7 mg/kg in the edible fruit. Residues of the metabolite were between 13 and 26%, and in one trial 46%, of the parent compound. In summary the myclobutanil residues in apricots from trials complying with GAP were 0.01, 0.04, 0.04, 0.04, 0.06 and 0.08 mg/kg in France and 0.11, 0.12, 0.17, 0.23 and 0.62 mg/kg in the USA.

Cherries. Supervised trials on cherries carried out in Germany and the USA which were evaluated by the 1992 JMPR were re-evaluated at the light of new GAP. Total residues (myclobutanil + metabolites) were determined. Myclobutanil residues from four residue decline trials in Germany according to GAP (3 applications at 0.135 kg ai/ha, 21 days PHI) were <0.01 and 0.02 (3) mg/kg. Three of the US trials reported in 1992 which included a 0-day PHI were reviewed. Five other trials reported to the present Meeting complied with US GAP (<9 applications at 0.07-0.16 kg ai/ha, with a maximum of 1.45 kg ai/ha per season, 0-day PHI). The residues of myclobutanil *per se* in the US trials in rank order were 0.2, 0.28, 0.68, 0.85, 0.92, 1.04, 1.12 and 1.44 mg/kg. In two of the trials metabolite residues reached more than 40% of those of the parent compound.

Peaches. Several trials carried out in France, Spain and the USA were reported in 1992. A trial in France was not conducted according to GAP but it could be evaluated against the Spanish use pattern. Residues from two Spanish trials according to GAP (1-4 applications at 0.08-0.1125 kg ai/ha, 15 days PHI) were 0.02 and 0.03 mg/kg; metabolites were not determined. Three US trials reported in 1992 were re-evaluated together with six new trials, all according to new US GAP (<9 applications at 0.07-0.165 kg ai/ha, with a maximum of 1.45 kg ai/ha per season, 0-day PHI). Residues of RH-9090, its conjugate, and the ketone RH-9089 were determined as RH-9090 in all the trials. The total residue in the edible portion in the US trials ranged from 0.35 to 1.53 mg/kg, with residues of myclobutanil from 0.33 to 1.22 mg/kg.

Plums and prunes. Two trials reported to the Meeting complied with US GAP (7 applications at 0.06-0.165 kg ai/ha with a maximum of 1.2 kg ai/ha per season, 0-day PHI). The total residues were 0.1 and 0.73 mg/kg with myclobutanil residues of 0.09 and 0.59 mg/kg. One trial in Italy (4 applications at 0.1-0.15 kg ai/ha, 14-day PHI) was evaluated against Greek GAP (3 applications at 0.055-0.125, 15 days PHI); the total residue was 0.1 mg/kg. There were too few results to estimate a maximum residue level. The existing CXL is 0.2 mg/kg.

In view of the similar use patterns for the individual fruits, the Meeting agreed to evaluate the combined US data as applying to stone fruit, except plums. The myclobutanil residues in stone fruit (edible portion) in rank order (median underlined) were 0.09, 0.11, 0.12, 0.17, 0.2, 0.23, 0.28, 0.33, 0.34, 0.38, 0.59, 0.62, 0.66, 0.68, 0.74, 0.75 (2), 0.85, 0.92, 1.04, 1.12, 1.22 and 1.44 mg/kg. The Meeting estimated a maximum residue level of 2 mg/kg and an STMR of 0.62 mg/kg for stone fruit except plums, and recommended the withdrawal of the individual CXLs for apricot, cherries and peach.

<u>Bananas</u>. Several studies were conducted in the USA and Costa Rica according to the proposed use of myclobutanil in banana packing stations in grower countries. All treatments consisted of one application of myclobutanil, and banana hands were sprayed or dipped at various concentrations.

Those trials in which myclobutanil was used at 200 or 400 mg ai/l and residues were calculated on the whole banana were evaluated to estimate a maximum residue level.

Banana samples taken at intervals of 0 to 28 days after treatment showed that the residues in the pulp increase with time. There was also a loss in weight of the banana hands with storage time, mainly from the peel. Storage periods from 7 to 21 or 28 days represent the shipping periods needed to reach different markets.

Residues in samples with 0-7 days storage are most appropriate for estimating maximum residue levels because they are the highest in the whole fruit, but residues after longer storage times are appropriate for assessing dietary exposure because the residues in the pulp increase with time. Residues of the parent compound were predominant in the total residue; those of the sum of the free and conjugated forms of the hydroxy metabolite (RH-9090) were less than 10% of the total in most of the trials.

The highest residues in whole bananas from each trial at the highest proposed GAP concentration, 400 mg ai/l, from 7 to 28 days ranged from 0.64 to 1.7 mg/kg. Since these results were from trials according only to proposed GAP, the Meeting was unable to estimate a maximum residue level.

Residues of myclobutanil in the edible pulp of the bananas in rank order (median underlined) were 0.1, 0.17, 0.17, 0.19, 0.2, <u>0.21</u>, <u>0.22</u>, 0.27, 0.28, 0.35, 0.39 and 0.41 mg/kg.

<u>Citrus fruit</u>. The results of twelve trials in Spain on the post-harvest treatment of mandarins with myclobutanil were reported to the Meeting. The reports lacked critical analytical data such as LOD, recoveries, and chromatograms. The residues of myclobutanil (applied as a water/wax emulsion or emulsifiable concentrate) in samples of whole fruit from trials complying with GAP (0.05 kg ai/hl or 0.01 kg ai/t fruit, 0-day PHI), ranged from 0.94 to 2.9 mg/kg. The residues from treatments with the EC formulation at 0.05 kg ai/hl, 0-day PHI, in rank order were 0.94, 1.15, 1.33, 1.5, 1.56, 1.7 and 2.0 mg/kg. Fruit samples were also analysed at 7 and 14 days after treatment. No decrease in the residue was observed in most of the trials.

Several trials on the post-harvest treatment of various varieties of orange which complied with Spanish GAP were reported to the Meeting. Myclobutanil was used as a water/wax emulsion and an EC formulation. The residues of myclobutanil ranged from 0.87 to 2.66 mg/kg. The myclobutanil residues in the whole fruit from treatment with the EC formulation according to GAP (0.05 kg ai/hl) were 1.06, 1.3, 1.36, 1.49, 1.53 and 1.8 mg/kg.

Since the citrus trials lacked the critical analytical data mentioned above the Meeting could not recommend an MRL.

Whole-fruit samples of the oranges and mandarins in these trials were separated into peel, pulp and juice. Analysis showed that the myclobutanil residue was almost all in the peel and not found in the pulp. The residue in the juice was approximately 10% of that in the whole fruit (0-day PHI). The residues of the metabolite were not determined.

<u>Berries</u>. Several field trials were conducted on blackcurrants in the UK with various myclobutanil formulations. In all the trials blackcurrant samples were analysed for the parent compound and RH-9090. The residues of myclobutanil from trials according to UK GAP (4-6 applications at 0.09 kg ai/ha, 14 days PHI) ranged from 0.04 to 0.43 mg/kg, with total residues (myclobutanil + RH-9090)

from 0.08 to 0.47 mg/kg. In three of fifteen trials reflecting GAP, residues of the metabolite were equal to or higher than those of the parent compound.

The residues of myclobutanil in blackcurrants in rank order (median underlined) were 0.04, 0.07, 0.08, 0.19, 0.24, 0.24, 0.26, 0.26, 0.29, 0.3, 0.3, 0.31, 0.35, 0.42, 0.43 mg/kg.

The Meeting estimated a maximum residue level of 0.5 mg/kg and an STMR of 0.26 mg/kg for blackcurrants.

Numerous field trials on strawberries have been conducted in the UK, France, Italy and Spain. The residues in seventeen trials in the UK in accordance with GAP (4-6 applications at 0.09 kg ai/ha, 3-day PHI) ranged from 0.1 to 0.5 mg/kg; residues of the metabolite were below the LOD. In one trial in France with more applications than are allowed by GAP, the residue of myclobutanil at 4 days was 0.04 mg/kg. In two trials in Italy which complied with GAP (3 or 4 applications, 0.005 kg ai/hl, 7-day PHI) the residues were 0.05 and 0.09 mg/kg. The residues in strawberries from trials in Spain ranged from 0.02 to 0.15 mg/kg.

The myclobutanil residues in strawberries in rank order (median underlined) were 0.04, 0.05, 0.08, 0.08, 0.09, 0.1, 0.12, 0.15, 0.15, 0.15, 0.17, 0.18, 0.18, 0.19, 0.19, 0.19, 0.19, 0.2, 0.2, 0.22, 0.24, 0.36, 0.48, 0.5 and 0.69 mg/kg.

The Meeting estimated a maximum residue level of 1 mg/kg and an STMR of 0.18 mg/kg for strawberries.

Tomatoes. The results of a large number of indoor and outdoor trials from several countries were reported to the Meeting. The residues found in Belgian trials (indoors) reflecting GAP (3-6 applications at 0.0075 kg ai/hl, 3 days PHI) ranged from 0.05 to 0.16 mg/kg; metabolites were not detected. Four trials in France (6 applications at <0.12 kg ai/ha, 3 days PHI) were evaluated against Spanish GAP (1-6 applications, <0.112 kg ai/ha, 3 days PHI). The residues were 0.02, 0.03, 0.04 and 0.05 mg/kg. The residues from trials in Spain according to GAP ranged from 0.03 to 0.24 mg/kg; the residues of metabolites determined in two trials were below 20% of those of the parent compound. In one trial in Italy according to GAP the residue was 0.02 mg/kg, with metabolites expressed as RH-9090 below the LOD. Four other trials were carried out in Italy in 1996, but as the Meeting doubted whether the data had been recorded properly the residues from them were not included in the evaluation. In trials according to GAP in Morocco (1-3 applications, 0.00625 kg ai/hl, 7-day PHI), the residues were between 0.02 and 0.06 mg/kg. The residues of the metabolite determined in two trials were below the LOD.

The residues in fifteen US field trials according to GAP on several varieties of tomato (0.07 kg ai/ha/application, with a maximum of 0.4 kg ai/season, 0-day PHI), ranged from 0.01 to 0.22 mg/kg.

The myclobutanil residues in tomatoes in rank order (median underlined) were 0.01, 0.02 (7), 0.03 (3), 0.04 (3), 0.05 (7), 0.06 (2), 0.07, 0.08 (4), 0.09 (2), 0.11, 0.15 (3), 0.16, 0.22 and 0.24 mg/kg.

The Meeting estimated a maximum residue level of 0.3 mg/kg and an STMR of 0.05 mg/kg for tomatoes.

Hops. Four of six trials conducted in the UK were according to GAP (6 applications, 0.0045 kg ai/hl, 10 days PHI). The residues of myclobutanil in the dried cones ranged from 0.2 to 1.2 mg/kg. The Meeting considered the database insufficient to estimate a maximum residue level.

The Meeting was informed that a further four trials are in progress in Germany and that they included processing studies.

Processing

Two supervised trials on tomatoes were conducted in the USA, with 4 applications at rates of 0.067 and 0.14 kg ai/ha. Samples harvested 5 days after the last treatment were processed to canned whole tomatoes, juice, purée, pomace and paste. In four processing studies in France, tomatoes treated with 6 applications of myclobutanil (0.107-0.12 kg ai/ha) were harvested 3 days after the last treatment and processed into juice, preserve and purée.

There was no concentration of the residue in tomato juice, canned tomatoes or preserve. The residues in tomato purée were concentrated by factors of 1.0 to 3, with an average of 1.6. The concentration factors for dry pomace were 14 and 17, with an average of 15.5, and for paste 3.7 and 4.2, mean 3.9. In some processed products, residues of the metabolite reached 50% or more of the total residue. On the basis of an STMR of 0.05 mg/kg, the Meeting estimated STMR-Ps of 0.08 mg/kg for tomato purée, 0.78 mg/kg for dry pomace, and 0.2 mg/kg for paste.

Data from three processing trials on blackcurrants in the UK indicated that residues in the juice decreased about 1.5-5 times, with a mean processing factor of 0.35. Canned fruit, in a single trial, showed a decrease in the residue of myclobutanil but a higher concentration of the metabolite, with the same total residue. The Meeting estimated an STMR-P of 0.09 mg/kg for blackcurrant juice from the STMR of 0.26 mg/kg for blackcurrants (whole fruit).

Two processing trials on strawberries in the UK showed that residues do not concentrate in strawberry jam or preserve. The average processing factors were 0.5 for jam and 0.81 for preserve.

The Meeting estimated STMR-Ps of 0.09 mg/kg for jam and 0.15 mg/kg for preserve on the basis of an STMR of 0.18 mg/kg for whole strawberries.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue level listed below are suitable for establishing maximum residue limits and the supervised trials median residue are suitable for use in dietary intake estimations.

Definition of residue for compliance with MRL and for estimation of dietary intake: myclobutanil

Commodity		MRL, mg/kg		PHI, days	STMR or STMR-P, mg/kg
CCN	Name	New	Pevious		
FS 0240	Apricot	w	0.2		
FB 0278	Blackcurrant	0.5	-	14	0.26
	Blackcurrant juice				0.09 (P)
FS 0013	Cherries	w	1		
FS 0247	Peach	w	0.5		
FS 0012	Stone fruits, except plums	2	-	0	0.62
FB 0275	Strawberry	1	-	3	0.19
	Strawberry jam				0.09(P)
	Strawberry preserve				0.15(P)
VO 0448	Tomato	0.3	-	0	0.05
	Tomato, canned				0.05 (P)
	Tomato juice				0.05 (P)

Commodity		MRL, mg/kg		PHI, days	STMR or STMR-P, mg/kg
CCN	Name	New	Pevious		
	Tomato, dry pomace				0.78 (P)
	Tomato paste				0.2 (P)
	Tomato puree				0.08(P)

(P): STMR-P

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PARATHION (058)

EXPLANATION

Parathion was originally evaluated in 1965 and has been reviewed several times since. In 1991 the JMPR recommended an MRL of 0.05 mg/kg for apple.

At the 25th Session of the CCPR (1993, ALINORM 93/24 A, para 81) the manufacturers informed the Committee that they would seek re-registration and indicated that a higher MRL for apple was needed. The proposed MRL was held at step 7B by the 1994 CCPR pending a new JMPR review. In 1995 the CCPR was informed that additional trials on apples were in progress; the results would not be available until 1996.

At the 29th Session of the CCPR (1997, ALINORM 97/24, para 52) the Committee kept the MRL at step 7B pending evaluation by the 1997 JMPR.

The Meeting received new residue data on apples, details of the analytical method used in the trials, and information on current GAP for apples in France.

METHODS OF RESIDUE ANALYSIS

Analytical methods

Samples from the trials reported to the Meeting were analysed by the method validated at Huntingdon Life Sciences (Study No. CHV 55).

The method involves macerating with aqueous acetone and filtering, followed by evaporation of the acetone. The aqueous phase is partitioned with dichloromethane and the organic phase is cleaned up by C-18 solid-phase extraction. Parathion and paraoxon are determined by temperature-programmed GLC with an FPD The limit of determination (LOD) was 0.01 mg/kg for parathion and paraoxon in apples and the limits of detection were 0.0029 and 0.0024 mg/kg for parathion and paraoxon respectively.

Recoveries at fortification levels from 0.01 to 2 mg/kg were 87-105% for parathion and 89-101% for paraoxon.

Stability of pesticide residues in stored analytical samples

In the 1995 evaluation it was reported that parathion and paraoxon added to macerated apples at 1 mg/kg were stable at -20°C for 24 months, with 89-119% of the parathion and 88-106% of the paraoxon remaining.

USE PATTERN

Parathion is an insecticide with registrations in many countries for foliar application to a wide range of horticultural and agricultural crops.

Information on approved uses of parathion was recorded by the 1991 Meeting. The registered uses on apples and pome fruit are listed in Table 1. New information on registered uses on pome fruits was provided only for France.

Table 1. Registered uses of parathion on apples and pome fruits.

Crop	Country	Application			PHI, days
		kg ai/ha	kg ai/hl	No.	
Apples	Finland	0.3-0.6		1-3	21
	France		0.015		
	France		0.0225		
	Mexico	1.5	0.075	1-2	14
	Morocco	0.03-0.09		1-2	
	Netherlands		0.011	1	
	Portugal	0.3-0.45	0.03-0.045	1	21
	Portugal	0.2-0.3	0.02-0.03	1-3	21
	Zimbabwe	0.38-0.75	0.05	2	42
Pome fruit	Greece	0.5	0.05	1	14
	Italy	0.24-0.75	0.016-0.06	1	20
	Spain	0.34-0.9	0.023-0.06	1	21
		1.13	0.075	1	
		0.45-0.75	0.03-0.05	2-3	21

RESIDUES RESULTING FROM SUPERVISED TRIALS

Ten supervised trials carried out in 1968 and 1969 in Germany were evaluated by the 1991 Meeting against French GAP, and supported a maximum residue level of 0.05 mg/kg. Six of the trials were at a lower application rate than French GAP at that time. The residues in apples after one application according to GAP at 0.25-0.35 kg ai/ha and 15 days PHI were 0.03, 0.04, 0.05 and 0.05 mg/kg.

Six field trials on apples were conducted in 1994 on commercially representative sites, four in Central and two in Southern France. The plot sizes were about 75-191 m^2 (14 trees) in a single row. Parathion 500 g/l EC was applied twice at 0.36 kg ai/ha with a motorized knapsack sprayer with a 14-day interval. The second application was made at the stage of enlarging fruit in trials F1 and F3 and at maturity in trials SI and SII.

Samples were taken for a decline study immediately before the second application and then at 0, 3, 7, 14, 21 and 28 days PHI, with a final sample 33-45 days after the last application. Samples were stored at -20°C until analysis and the SAI was 8-11 months. The results are shown in Table 2. Residues of paraoxon were not detected except in trials F3, F4 and SII at a 0-day PHI where the residues were all <0.01 mg/kg. No residues of parathion were detected after 28 days.

Table 2. Parathion residues from supervised trials in France in 1994 (Cheminova Agro, 1997). All EC 500 g/l formulation. Underlined residues are from treatments according to GAP and were used to estimate maximum residue levels

Trial no., Location, Variety	Application			PHI, days	Parathion, mg/kg
	No.	kg ai/hl	kg ai/ha		
F1, Orleans, Melrose	2		0.36	0	0.13
				3	0.09
				7	0.06
				14	0.02
				21	0.01
				28	n.d

Trial no., Location, Variety	Application			PHI, days	Parathion, mg/kg
	No.	kg ai/hl	kg ai/ha		
F2, Orleans, Golden Delicious	2	0.036	0.36	0	0.16
				3	0.07
				7	0.02
				14	0.02
				21	<0.01
F3, Orleans, Golden Delicious	2	0.036	0.36	0	0.26
				3	0.1
				7	0.06
				14	0.04
				21	0.02
F4, Chinon, Golden Delicious	2	0.036	0.36	0	0.51
				3	0.28
				7	0.16
				14	0.13
				21	0.08
S1, Montauban, Golden Delicious	2	0.036	0.36	0	0.4
				3	0.1
				7	0.06
				14	0.02
				21	0.01
				28	n.d
SII, Montauban, Red Chief	2	0.036	0.36	0	0.21
				3	0.14
				7	0.24
				14	0.11
				21	0.08

FATE OF RESIDUES IN STORAGE AND PROCESSING

No new information was submitted. Processing studies were evaluated by the 1991 JMPR.

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

No information was provided.

NATIONAL MAXIMUM RESIDUE LIMITS

The following MRLs were reported to the 1991 JMPR.

Country	Commodity	MRL, mg/kg
Australia	Fruit, except apricots and peaches	0.5
Austria	Fruit	0.5
Belgium	Fruit	0.5
Canada	Apples	1
Denmark	Fruit	0.5
EU	Fruit	0.5[1]
Finland	Fruit	0.5[1]
France	Fruit	0.5[1]
Germany	General (EU)	0.5[1]
Greece	Fruit	0.5[1]
Israel	Fruit	0.5
Italy	Fruit	0.5
Japan	Apples	0.3
Kenya	Fruit except apricots, citrus fruits and peaches	0.5

Country	Commodity	MRL, mg/kg
South Korea	Apples	0.3
Luxembourg	Fruit	0.5
Malaysia	Fruit except apricots, citrus fruits and peaches	0.5
Mexico	Apples	1
Netherlands	Fruit except apricots, citrus fruits and peaches	0.5
New Zealand	Fruit	0.5
Spain	Fruit	0.5
Sweden	Fruit except citrus fruits	0.5[1]
Switzerland	Fruit	0.5
USA	Apples	1
Venezuela	Fruit	1
Yugoslavia	Fruit	0.5

[1]Sum of parathion and paraoxon

APPRAISAL

Parathion was first evaluated by the JMPR in 1965 and extensively re-evaluated in 1991 and 1995. The 1991 JMPR recommended an MRL of 0.05 mg/kg for apple. The proposed MRL was advanced to Step 7B by the 1994 Session of the CCPR and subsequently held there, pending re-evaluation by the present Meeting.

The analytical method used in trials reported to the Meeting was based on temperature-programmed GLC with FP detection. Parathion and paraoxon were both determined with LODs of 0.01 mg/kg and recoveries above 80%.

Information on registered uses and national MRLs was recorded in the 1991 JMPR evaluation. New information on registered uses on pome fruits was provided only for France.

Information was submitted on residues from six supervised trials on apples in central and southern France in 1994. These were studies of residue decline and showed that parathion residues decreased from 0.13-0.51 mg/kg at 0 day to <0.01-0.08 mg/kg after 21 days. Residues of paraoxon were not detected. Since the trials were with a 50% higher application rate than French GAP, the Meeting could not change the previous recommendation.

The Meeting was informed that another eight trials in France and Spain are planned. The Meeting noted that parathion is scheduled for periodic review in 2000.

REFERENCES

Cheminova Agro A/S, 1997. Determination of Residues of Ethyl Parathion and its Metabolite Paraoxon in apples treated with Ethyl Parathion (EC formulation) during Field Trials in France. Study No. CHV 51C, Date 5 June 1997. Unpublished.

PHOSMET (103)

EXPLANATION

Phosmet has been evaluated at several Joint Meetings between 1976 and 1988 (FAO/WHO 1977, 1979, 1980, 1982, 1985, 1988a,b). Maximum residue limits (MRLs) were recommended for a number of commodities of plant and animal origin

Updated information on GAP and details of supervised trials, processing trials and studies of metabolism and the stability of residues during frozen storage have been made available for evaluation within the CCPR periodic review programme.

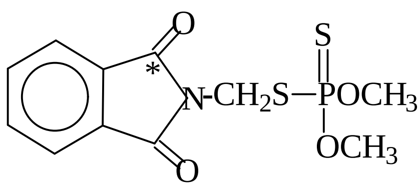
No data to support the direct use of phosmet to control ectoparasites on domestic animals have been submitted for review.

IDENTITY

ISO common name: phosmet
Chemical name:

IUPAC: *O,O*-dimethyl *S*-phthalimidomethyl phosphorodithioate, *N*-(dimethoxyphosphinothioylthiomethyl)phthalimide
CA: *S*-[1,3-dihydro-1,3-dioxo-2*H*-isoindol-2-yl)methyl] *O,O*-dimethyl phosphorodithioate

CAS No.: 732-11-6
Molecular Formula: $C_{11}H_{12}NO_4PS_2$
Structure:



Synonyms: R-1504, PMP
Trade names: 'Imidan'; 'Prolate'; ‘Phthalophos’; 'Kenolate'; 'Appa'

Physical State: Colourless crystalline solid

Melting Point: 72°C
Vapour Pressure: 4.9×10^{7} mm Hg at 25°C
Log P_{ow}: 2.95 at 20°C
Formulations: Phosmet is formulated mainly as wettable powders (WP, up to 70%) and emulsion concentrates (EC).

METABOLISM AND ENVIRONMENTAL FATE

Phosmet labelled at the carbonyl groups of the phthalimide moiety has been used in metabolism and other environmental fate studies. The designation of metabolites and their chemical structural formulae are shown in Table 1.

Animal metabolism

The absorption, distribution, metabolism and excretion of ^{14}C]phosmet has been studied in rats, goats and hens. A study on cows from 1963 was also reported.

Groups of five male and five female rats were given a single oral dose of either 1 or 25 mg [^{14}C]phosmet without prior exposure to the chemical, or 14 daily oral doses of 1 mg unlabelled phosmet/kg followed by a single dose of 1 mg of the labelled compound. Urine and faeces were collected at regular intervals, and selected tissues were collected when the animals were killed at 96 hours after dosing. By 24 hours after dosing, at least 70% of the dose had been excreted with the urine by all dose groups. By 96 hours, the single-dose and the repeated-dose animals had excreted mean urinary levels of 81-89% and 75-77% of the dose respectively. Faecal excretion amounted to 6-13% of the dose. Radioactive residues in tissues accounted for 1.2-2.1% of the dose in all dose groups (Fisher, 1989).

In the early study of Ford *et al.* (1966) in rats, it was observed that <1% of the label in the urine was in the form of phosmet oxon and less than 0.04% of the radioactivity was recovered in the expired air.

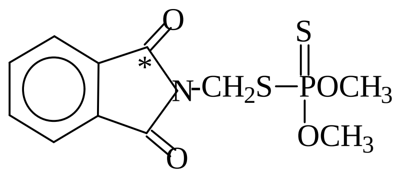
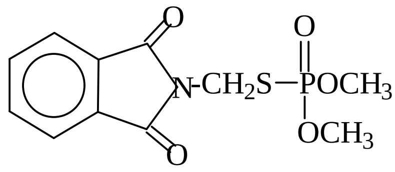
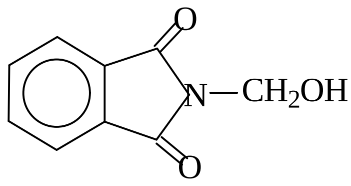
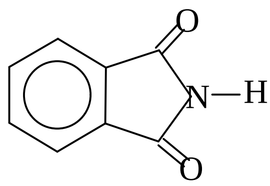
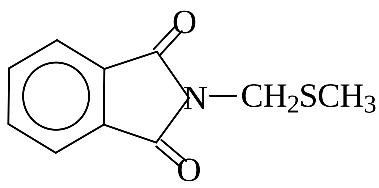
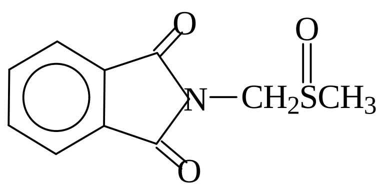
Radiolabelled phosmet was administered orally to two lactating goats for four days at dietary equivalent levels of 8.0-8.8 ppm. Radioactive material was quickly absorbed and excreted. Most of each day's dose was recovered in the urine within the following 24 hours. In total, urinary excretion accounted for 60% of the cumulative dose. Less than 6% remained in the edible tissues at slaughter, 13-14 hours after the final dose.

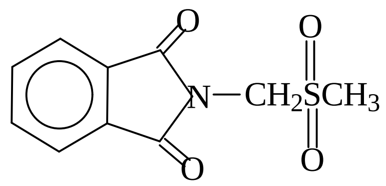
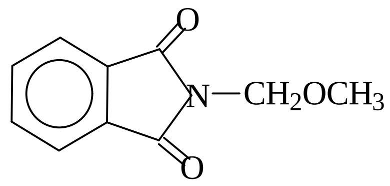
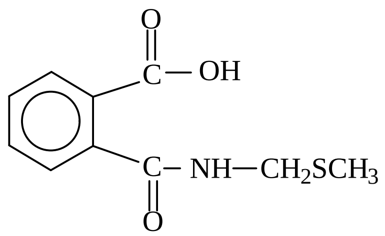
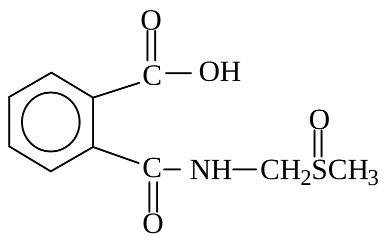
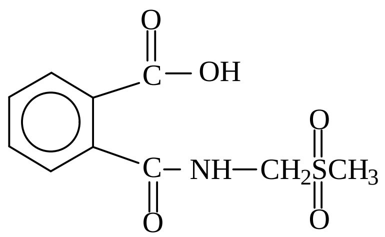
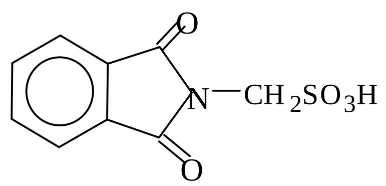
The levels of total radioactive residues (as phosmet equivalents) in the liver, muscle, kidneys, milk and fat ranged from 0.24 mg/kg in kidneys to 0.005-0.007 mg/kg in fat. In milk the highest residue levels were 0.014-0.017 mg/kg, found two to four days after the start of dosing; less than 3% of the ^{14}C was associated with the fat.

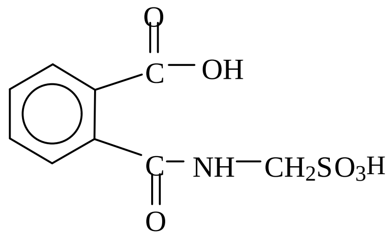
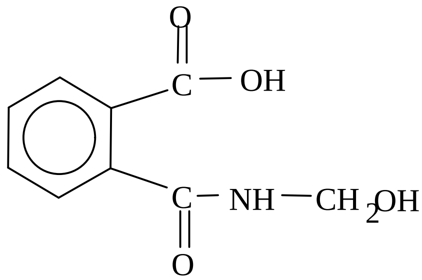
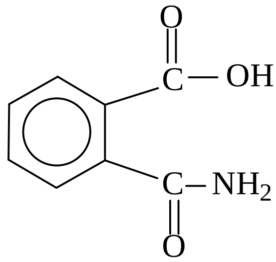
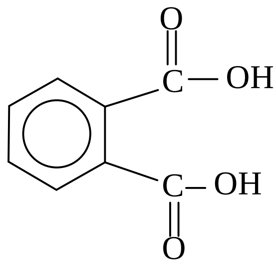
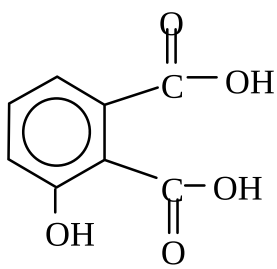
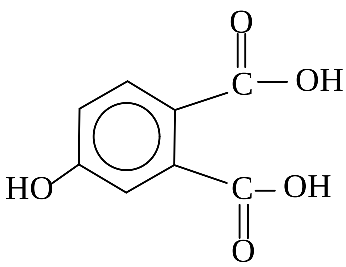
Nine identified metabolites (PiMSM, PiMS(O)M, PiMS(O_2)M, PaAMS(O)M, PaAMS(O_2)Me, Pi, PaAMOH, PaA and Pa, see Table 1) and two unidentified metabolites AQ1 (thought to be a derivative of phthalamic acid with an acidic *N*-substituent) and AQ2 were quantitatively determined in all samples, while one metabolite was tentatively identified as PaAMSM in liver samples. Neither phosmet nor phosmet oxon were detected in edible tissues (<0.002-0.003 mg/kg) or milk (<0.0004 mg/kg). Each of the samples contained the same metabolites but the relative amounts varied. The highest concentrations of individual metabolites found in each of the substrates were 0.035-0.036 mg/kg of AQ1 in kidneys, 0.007-0.009 mg/kg of PiMSM in liver, 0.018-0.022 mg/kg of PaAMS(O_2)M in muscle and 0.004-0.005 mg/kg of PaAMS(O_2)M in milk. These observations indicate that residues of phosmet are not retained in fat and do not accumulate in tissues or milk.

Bound residues accounted for about 70% of the total residues in liver and muscle, 40% in the kidneys and, associated with milk solids, 8-19% of the total residues in milk. A small proportion of the bound residues was released by mild acid hydrolysis and treatment of extracted samples with hydrazine solubilized more than half of the bound residues. The solubilized products of hydrazinolysis consisted mostly of phthalohydrazide. The results indicate that the bound residues in tissues and milk contain the phthalimide moiety, bound via *N*-substituents, with little or no chemical modification. The nature of the solvent-extractable residues, as well as other known chemical properties of the metabolites and related compounds, suggested to the author that the residues that remained bound after hydrazinolysis were *N*-substituted derivatives of phthalamic acid.

Table 1 Common designations, chemical names and structural formulae of phosmet and its metabolites.

Common designation	Chemical name	Structure
1. Phosmet	O,O-dimethyl-S-phthalimidomethyl phosphorodithioate (Asterisk shows position of label)	
2. Phosmet oxon	O,O-dimethyl-S-phthalimidomethyl phosphorothioate	
3. PiMOH	N-hydroxymethylphthalimide	
4. Pi	Phthalimide	
5. PiMSM	N-methylthiomethylphthalimide	
6. PiMS(O)M	N-methylsulfinylmethylphthalimide	

Common designation	Chemical name	Structure
7. $PiMS(O_2)M$	N-methylsulfonylmethylphthalimide	
8. PiMOM	N-methoxymethylphthalimide	
9. PaAMSM	N-methylthiomethylphthalamic acid	
10. PaAMSO(O)M	N-methylsulfinylmethylphthalamic acid	
11. $PaAMS(O_2)M$	N-methylsulfonylmethylphthalamic acid	
12. $PiMSO_3H$	N-sulfomethylphthalimide	

Common designation	Chemical name	Structure
13. $PaAMSO_3H$	N-sulfomethylphthalamic acid	
14. PaAMOH	N-hydroxymethylphthalamic acid	
15. PaA	Phthalamic acid	
16. Pa	Phthalic acid	
17. 3-OHPA	3-hydroxyphthalic acid	
18. 4-OHPA	4-hydroxyphthalic acid	

Several groups of cows were treated with phosmet either in the diet at 100, 45 or 20 ppm or by spray application at 0.5% or 0.25%. Milk was sampled up to 21 days in the feeding study and 28 days in the spray study. Eleven tissues were analysed from cows slaughtered 1 and 6 days after the last phosmet feeding. The residues in all samples were below the limit of determination (0.05 mg/kg) of the colorimetric (phosphomolybdate) or enzyme-inhibition methods used (Batchelder and Patchett, 1966). Five cows were sprayed with 1 g ai/cow in a more recent supervised trial in The Netherlands . Phosmet residues in milk were 0.012, 0.002, 0.0009 and <0.0005 mg/kg at 0, 12, 24 and 36-72 hours after treatment (Ernst, 1983).

Radiolabelled phosmet was administered orally to laying hens for seven days at a dietary equivalent level of 10.5 ppm. Most of each day's dose was recovered in the excreta within the following 24 hours. In total the excreta accounted for 89.6% of the cumulative dose. Edible tissues collected at slaughter (15-18 hours after the final dose) and eggs accounted for only 0.3% of the cumulative dose.

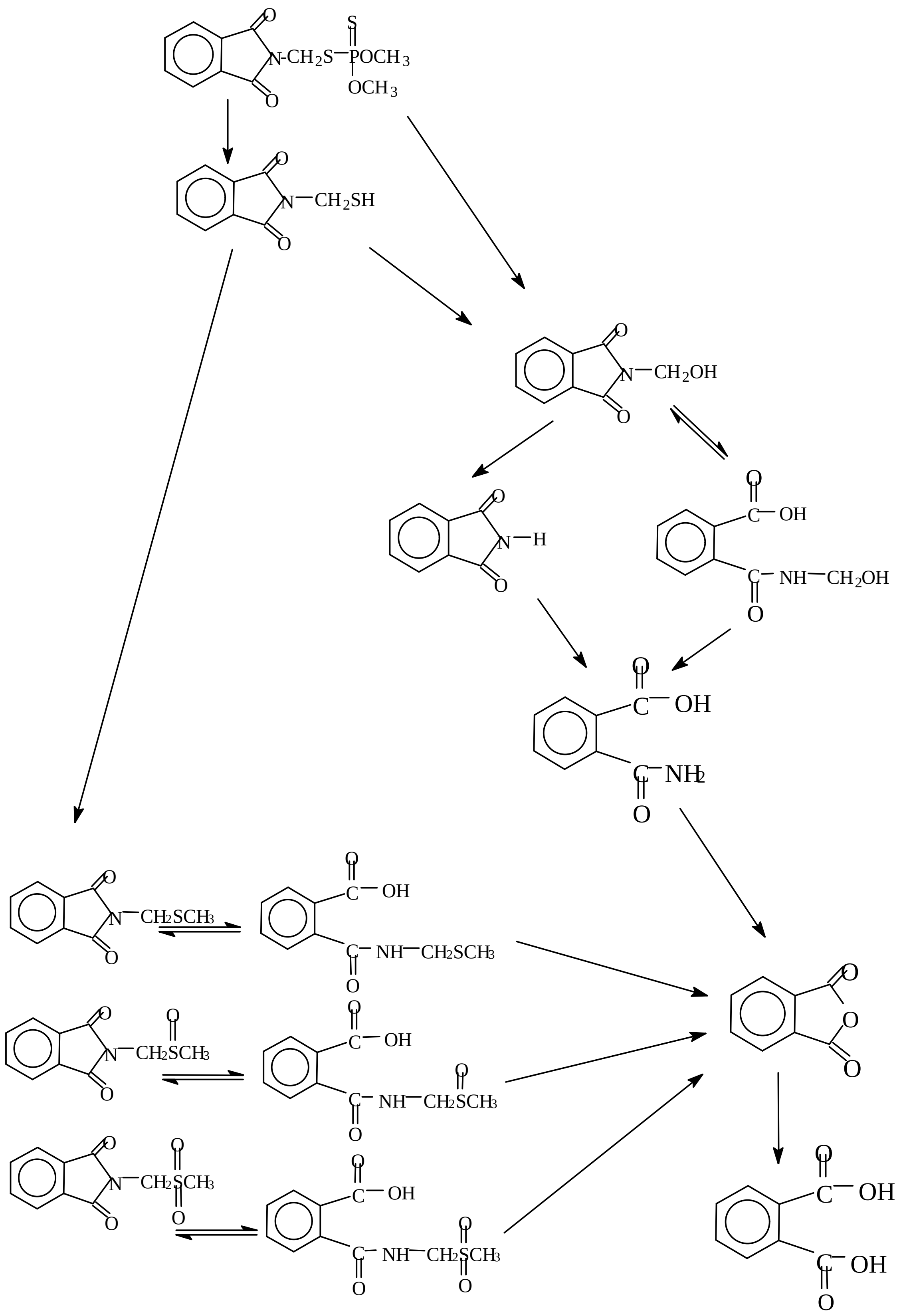
In egg yolks and whites, the highest levels of ^{14}C as phosmet equivalents were 0.040 mg/kg on day 7 and 0.007 mg/kg on day 4 respectively. At slaughter the levels of total radioactivity expressed as phosmet were 0.24 mg/kg in liver, 0.21 mg/kg in kidneys, 0.021 mg/kg in breast muscle, 0.015 mg/kg in thigh muscle, 0.005 mg/kg in fat and 0.068 mg/kg in blood.

Phosmet itself was not detected (<0.005 mg/kg) in any of the edible tissues, but 0.001 mg/kg was found in egg yolks. None of the metabolites exceeded 0.005 mg/kg in the edible tissues or eggs, where the identified metabolites were phthalimide and phthalic acid. These compounds are believed to reflect a sequence of hydrolytic degradation steps that can occur without enzymatic catalysis: loss of the phosphorus-containing moiety, hydrolysis of the phthalimide to yield phthalamic acid, and hydrolysis of the phthalamic acid to phthalic acid.

Hydrolysis of phosmet to *N*-mercaptomethylphthalimide may occur in tissues. An oxidation product of this compound, *N*-sulfomethylphthalamide (phthalimidomethylsulfonic acid), was identified in excreta but not in tissues.

Proposed metabolic pathways of phosmet in goats are shown in Figure 1 (Tarr, 1993a).

Figure 1. Principal metabolic pathways of phosmet in goats deduced from metabolites detected in tissues.



Extraction of tissues and egg yolks recovered only 30-50% of the total ^{14}C in the samples. Unextractable ^{14}C residues, which appear to be covalently bound, were solubilized to a small extent by mild acid hydrolysis. Treatment of extracted samples with hydrazine solubilized approximately a third to half of the bound ^{14}C. The soluble products of hydrazinolysis consisted mainly of phthalohydrazide. Bound ^{14}C residues therefore contain the *N*-substituted phthalimide moiety of phosmet, with little or no chemical modification. In liver, a substantial proportion of the extractable ^{14}C was associated with material that was precipitated upon concentration. This fraction, perhaps bound to soluble proteins, did not react with hydrazine. According to the author, the nature of the characterized residues, as well as the known chemical properties of the metabolites and related compounds, suggested that the ^{14}C residues that remained bound after hydrazinolysis were derivatives of phthalamic acid. The author believed that bound ^{14}C residues were probably cleared from the tissues by hydrolysis to phthalic acid (Tarr, 1993b).

Although the rat liver microsomal $NADPH_2$ enzyme system readily converts phosmet to phosmet oxon (McBain *et al.*, 1968), neither phosmet nor its oxon could be detected in the tissues of goats or hens (Tarr, 1993a,b).

Plant metabolism

Reports of studies on the metabolism of phosmet in sour cherries (Barnes and Goldsby, 1989), corn (Toia *et al.*, 1993b) and potatoes (Toia *et al.*, 1993c) were submitted for evaluation.

[^{14}C]phosmet was applied to sour cherry trees in a greenhouse to determine the metabolic fate of phosmet in a representative orchard crop. A single high rate of application of 4.2 kg/1000 litres (equivalent to 13.7 kg/ha) was used for the study. Fruit samples were taken at 4 hours and 7 and 14 days after treatment.

Phosmet was absorbed rapidly (44% in 4 hours) to the interior of the fruit where most of the metabolism took place. While the parent compound was the major residue on the surface of the fruit (39.1 and 48.4% of the total radioactivity at 7 and 14 days respectively), as many as 16 or 17 different metabolites were found in the pulp.

Phthalic acid was the major metabolite identified and accounted for 17 to 21% of the total radioactivity. Several other metabolites accounting for a small fraction of the radioactivity were identified, including phosmet oxon, phthalimide, *N*-hydroxyphthalimide, phthalic anhydride, *N*-hydroxymethylphthalmide and derivatives of phthalamic acid. However conjugates dominated the metabolic picture. The key conjugate identified was *N*-glucosylphthalimide. Structures of three additional unknowns could be derived from this through either enzymatic hydrolysis or chemical breakdown.

The conjugates and some other metabolites were readily converted to phthalic acid (Pa) by acid hydrolysis. Pa accounted for 85-90% of the extractable radioactivity after hydrolysis. No benzoic acid or ring-hydroxylated products were detected.

Proposed pathways of metabolism in cherries are shown in Figure 2.

Maize plants were grown singly in pots in an outdoor screened location in California and sprayed with carbonyl-labelled phosmet in 50% aqueous acetone containing 0.5% Tween 20. Four plants were treated with 1 kg [^{14}C]phosmet/ha at the onset of silk formation and one plant was harvested at the forage stage. The remaining three plants were treated with a further 1.12 kg [^{14}C]phosmet/ha 14 days before harvest and grown to maturity. During the latter stages, these plants were protected from adverse weather conditions.

At maturity, the fodder, cobs and grain were collected and analysed separately. Two untreated plants served as controls.

The distribution of the total ^{14}C expressed as phosmet in the plant fractions was as follows.

Forage (whole plant): 31 mg/kg
Fodder (husks, leaves and stalks): 267 mg/kg
Cobs:5 mg/kg
Grain: 3 mg/kg

More than 95% of the radiocarbon was extractable from all four fractions. HPLC and TLC analyses of the extracts indicated a large number of metabolites, all more polar than phosmet, and a similar pattern of metabolites in all fractions. Details of the compounds found in the mature fodder and grain are given in Table 2 and a proposed scheme for the metabolism of phosmet in maize is shown in Figure 3.

Table 2. Radioactive compounds found in mature maize, fodder and grain from plants treated with ^{14}C phosmet.

Compound	% of TRR	
	Fodder	Grain
Phosmet	53	0
Phosmet oxon	1.2	0
Phthalic acid (Pa)	5.5	61
PaAMOH	6.8	*
PaAMSM	0.56	*
$PaAMSO_3H$	0.71	*
Phthalimide (Pi)	3.9	*
PiMOM	3.6	*
$PiMSO_2M$	0.54	8
Unknowns	16.3[1]	32.7[1]
Unextracted (mainly hydrolysed to phthalates)	3.2	1.2

TRR = Total radioactive residue
[1]Not observed
[2]At least 15 separate compounds (the highest 2.7% in fodder and 13.4% in grain, mainly converted to phthalic acid on acid hydrolysis

Only traces (approximately 1% of the TRR) of phosmet oxon were found in the forage. Phosmet itself was the major residue (53% of the TRR) in the fodder, but was not found in the grain.

Four application of [^{14}C]phosmet were made to potato plants (*Solanum tuberosum*) growing in a large box filled with a sandy loam soil in a field plot in California. The treatments (1.68 to 2.0 kg ai/ha) were by spray application of [^{14}C]phosmet in 50% acetone/water (containing 0.5% Tween 20) to the plant foliage.

Figure 2. Metabolism of phosmet in cherries.

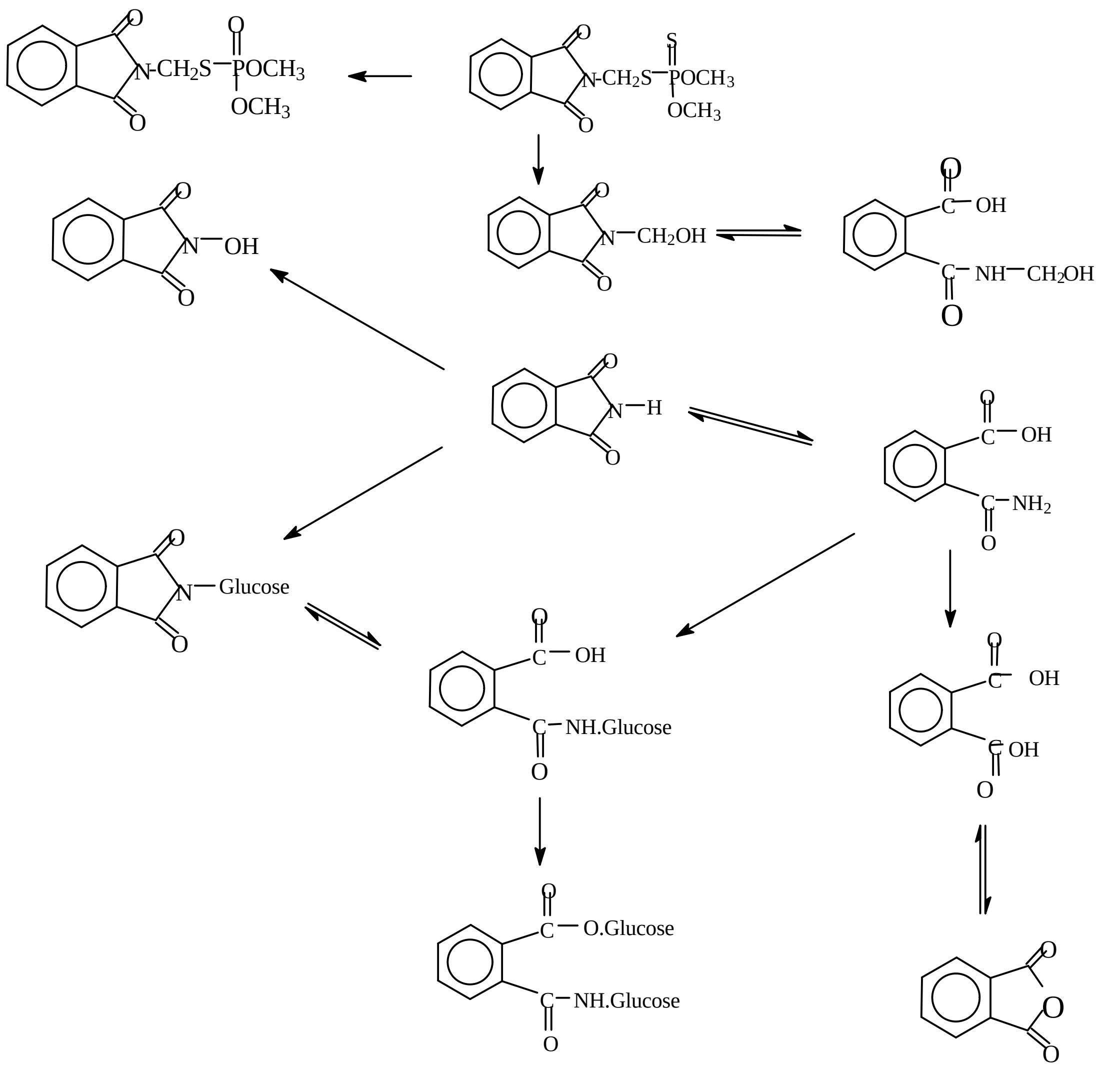
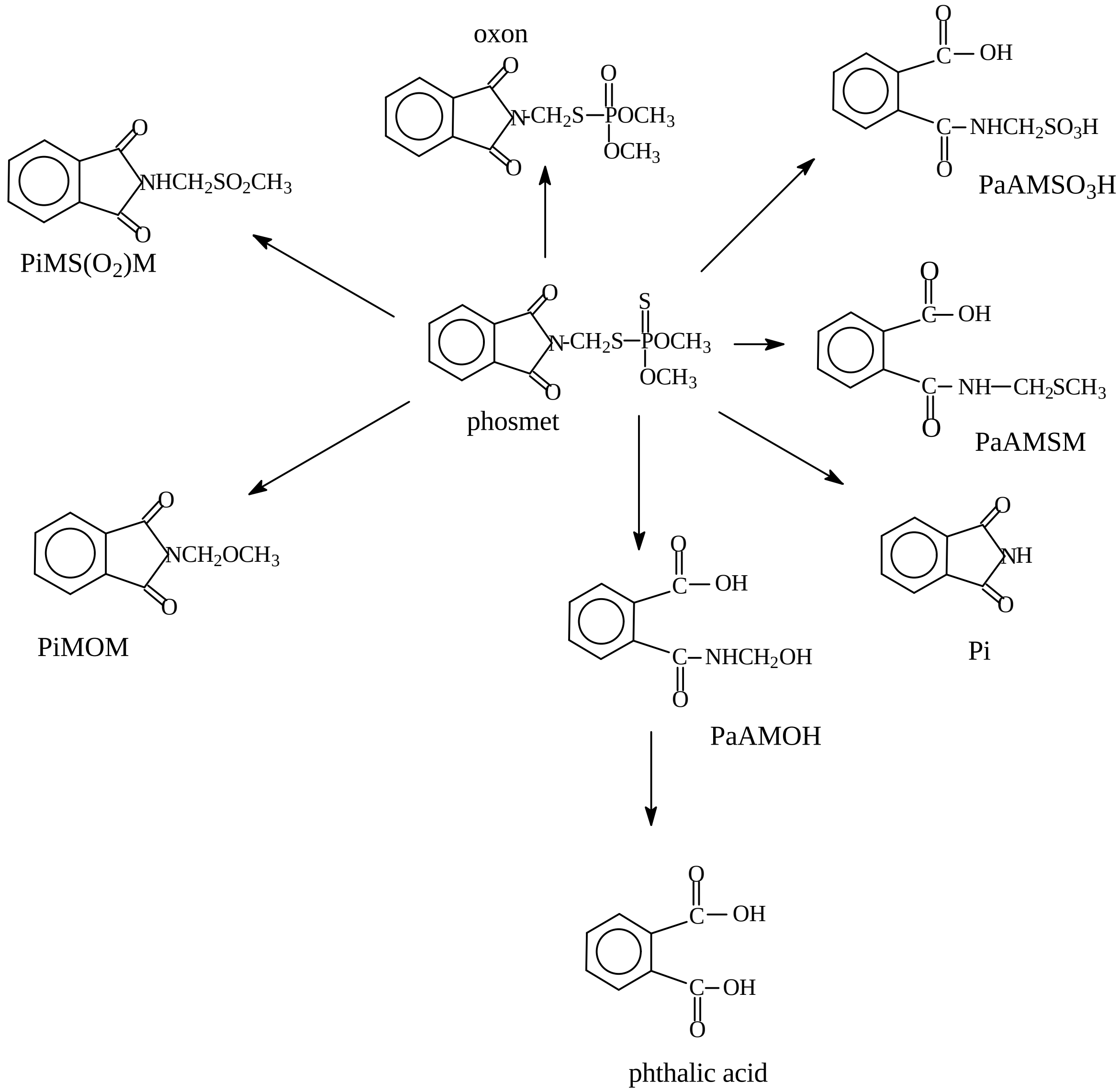


Figure 3. Metabolism of phosmet in maize



Applications were made at day 0 (to small immature plants), day 40, day 62 (7 days before the harvest of thin-skinned potatoes) and day 88, 7 days before the harvest of mature potatoes. The total amounts of phosmet applied were 5.6 kg ai/ha for thin-skinned potatoes and 6.8 kg ai/ha for mature potatoes. A control plot of the same size as the ^{14}C-plot was treated by application of solvent only. Samples were collected before the second and third applications, at thin-skinned potato harvest, and at mature potato harvest.

The total radioactive residue in the potato foliage ranged from 14 to 109 mg/kg, increasing with each treatment. The TRR in the tubers was 1.4-2.1 mg/kg, indicating only a small degree of translocation into them. Solvent extraction removed more than 92% of the residue. Most of the unextracted remainder could be hydrolysed to phthalic acid.

HPLC and TLC analysis of the extracts showed that phthalic acid (Pa) and phthalamic acid (PaA) were the major metabolites, although the latter was readily hydrolysed to phthalic acid during attempts to characterize it. Phosmet, its oxygen analogue and hydroxylated phthalic acids were not found in any of the extracts. Pa and PaA constituted 88%, 35% and 77% of the TRR in the first, third and final samples of the tubers respectively. Most of the unidentified metabolites were hydrolysed to phthalic acid, showing them to be derivatives or conjugates of it.

Environmental fate in soil

In early studies using unlabelled phosmet it was shown that its rate of degradation in soils was influenced more by soil pH than by the activity of micro-organisms. The half-life values for phosmet ranged from 3 days in a pH 7.2 loam to 12.2 days in a pH 5.1 sandy loam soil. Autoclaving increased the half-life only moderately (Menn *et al.*, 1965).

The metabolism of phosmet was studied by McBain (1986) who incorporated the carbonyl-labelled compound into moist loam soil (pH 7.1) at a rate of 4.77 mg/kg. The soil was kept under aerobic conditions for 3 days, then anaerobic conditions for 60 days. The evolved $^{14}CO_2$ amounted to nearly 40% of the applied ^{14}C. The extractable ^{14}C and the ^{14}C in flood water decreased with time. Under anaerobic conditions the metabolism of phosmet continued, but at a slower rate. In addition to unchanged phosmet, phosmet oxon, Pi, PaA, Pa, benzoic acid, PiMOM and PiMOH, were detected. Their concentration remained below 0.04 mg/kg during the study.

In another study with similar soil (pH 7.1) 5 mg/kg of the carbonyl-labelled compound was incorporated (McBain, 1990). After 4 days of aerobic conditions one test system was changed to anaerobic. The recovery of radioactivity was 92% of that applied. The radioactivity evolved as carbon dioxide from the aerobic and anaerobic soils was respectively 14.95 and 8.3%, and that remaining after extraction respectively 16.3% and 11.6% of the applied amount. About 11% of the radioactivity was found in the flood water.

The residue components and their percentages in the total residue, expressed as phosmet found in the aerobic soil were phosmet 36.6%, phosmet oxon 0.5%, Pi 1.53%, PaA <0.01%, Pa 0.88%, PiMOM 5.68%, PiMOH 0.41%, PaAMOH 2.44%, PiMSM 0.37%, PiMS(O)M 2.59%, PiMS(O_2)M 0.34%, PiAMS(O)M 0.97%, PiAMS(O_2)M 0.56%. Some unidentified intermediate products were also detected.

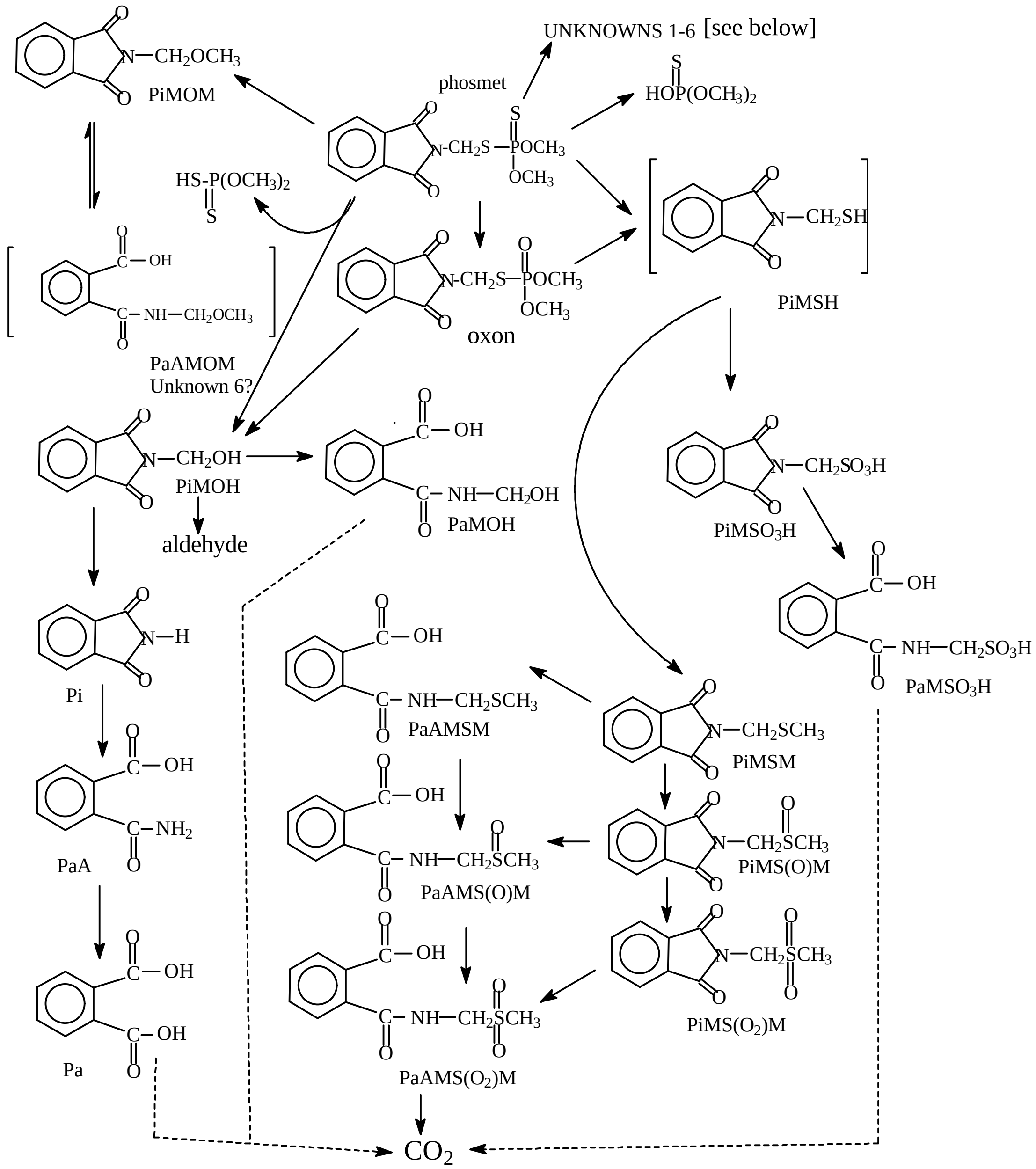
A proposed degradation pathway is shown in Figure 4.

Field dissipation studies indicated that phosmet would have an initial half-life of about 6 days in soils with a pH of about 8. Phosmet remained mainly in the top 10 cm soil layer, and no phosmet residue was detectable below 20-25 cm. Phthalimide, phosmet oxon and PiMOM were all undetectable (<0.02, <0.01 and <0.01 mg/kg respectively (Riggle *et al*., 1990; Roper, 1990, McKay, 1988).

Phosmet did not undergo significant photo degradation when exposed on thin layer plates of soil to natural sunlight for a period of 30 days (Zeigler and Hallenbeck, 1988).

The adsorption and desorption of [^{14}C]phosmet were examined in a sand, a sandy loam, a silty loam and a loam. K_d values were 1.17, 12.4, 15.8 and 13.6 respectively, giving K_{oc} values mainly in the range of 700-975. It was concluded that phosmet would be relatively immobile in all the soils studied except sand (Yeh, 1988).

Figure 4. Proposed degradation pathway for phosmet in soil.



Unknown	Exptl. treatment	Products
1	Oxidation	$PiMSO_3H$, $PaMSO_3H$, PaA
2	Oxidation	unesolved polar products
3	Hydrolysis	$PaAMSO_3H$, PaA, Pi
4	None (levels too low)	
5	Dehydraton (?)	PiMOM
6	Decomp. in MeOH	PaAMOH, Pa, PaA/MeOH product

Uptake of residues from soil

[^{14}C]carbonyl-labelled phosmet was sprayed at about 5.6 kg ai/ha (equivalent to 5 times the single application rate) on a sandy loam soil with an organic matter content of 1.6% and a pH of 6.5, confined in boxes in an outdoor location in California. Radishes, lettuce and wheat were subsequently planted in the soil 30, 120 and 365 days after the treatment. The total radioactive residues in the plants grown to maturity and the corresponding soil residues are shown in Table 3.

Neither phosmet nor its oxygen analogue were detected in the plant extracts. Analysis by HPLC showed a number of polar metabolites, most of which were characterized by chemical and enzymatic hydrolysis as esters or conjugates of phthalic acid (Toia *et al.*, 1993a).

Table 3. Total radioactive residues in plants grown to maturity in soils treated with labelled phosmet.

Sample	^{14}C as phosmet, mg/kg, at (periods in days)		
Soil[1] after application	7.74 (0)		
Soil[1] at planting 0-7.5 cm	4.68 (30)	4.24 (120)	2.00 (365)
Soil[1] at radish harvest	2.91 (71)	3.81 (167)	1.55 (392)
Radish roots	0.46	0.10	0.03
Radish foliage	0.61	0.15	0.05
Soil[1] at lettuce harvest	3.72 (83)	2.82 (171)	0.43 (406)
Lettuce	0.29	0.19	0.042
Soil[1] at wheat forage harvest	2.87 (92)	1.71 (192)	2.12 (406)
Wheat forage	0.59	0.12	0.045
Soil[1,2] at wheat harvest	1.18 (169)	1.60 (290)	2.21 (516)
Wheat chaff	0.76	0.26	0.12
Wheat Straw	0.93	0.46	0.11
Wheat grain	0.54	0.21	0.10

[1]0-7.5 cm layer. The residues in the 7.5-15 cm layer were <0.08 mg/kg
[2]0-7.5 cm layer. The residues in the 7.5-15 cm layer were <0.15 mg/kg at days 290 and 516

Environmental fate in water

Phosmet undergoes fairly rapid hydrolysis at ambient temperatures. The half-life values for phosmet in water at 25^{0}C were 7.5-9.7 days at pH 5, 9.4 hours at pH 7 and 5.5 minutes at pH 9 (Chang, 1987; Robinson, 1992). Degradation is enhanced by light: in a separate study the half-life of phosmet at pH 5 and 25^{0}C was reduced from 9.7 days in the dark to 2.42 days in the light (Robinson, 1992).

The major hydrolysis products formed at pH 5 in the dark were *O,O*-dimethyl hydrogen phosphorodithioate (79.4 mol %), *O*-methyl dihydrogen phosphorodithioate (4.1 mol %), PaA (34.4 mol %), Pi (10 mol %l) and Pa (8.9 mol %). Following irradiation with a xenon lamp at pH 5 dimethyl hydrogen phosphate (72.3 mol %), phosphoric acid (33 mol %), methyl dihydrogen phosphate (7.3 mol %), Pi (62.5 mol %), PA (15.7 mol %) and PaA (12.7 mol %) were also detected. PiMOH was also detected in small amounts. Other minor products were detected but not identified.

The recovery of labelled compounds from the organic and water phases was between 96.2% and 98.9 %. Volatile compounds accounted for <0.05% of the total radioactivity (Robinson, 1992).

METHODS OF RESIDUE ANALYSIS

Methods in current use rely on gas chromatography and determine the oxygen analogue of phosmet as well as phosmet itself, using a nitrogen-phosphorus selective thermionic detector. Variations used for the different types of sample are described below.

Food crops

Methods used for the analysis of samples from supervised field trials are modifications of the official method of analysis as published in the Pesticide Analytical Manual.

Phosmet and its oxygen analogue are extracted from the crop with benzene. Oils, when present, are separated from the extracted residues by hexane-acetonitrile partition. A charcoal shake-out provides adequate clean-up for most crops; peas and pea forage require a charcoal column clean-up and interfering co-extractives may be separated by a silica column clean-up. A thermionic detector is used for quantification of phosmet and its oxygen analogue down to 0.05 mg/kg. Recoveries from a large number of crops with added levels of each compound at 0.05-1.0 mg/kg were 71-101% for phosmet and 70-100% for its oxygen analogue. Carbofenothion interferes with phosmet oxon on OV-1 and OV-17 liquid phases. They can be separated on QF-1 or columns of similar polarity (Adelson *et al.,* 1973).

Improvements to this method have involved the use of a more polar extraction solvent (acetone), evaporation and partitioning into dichloromethane, followed by hexane/acetonitrile partitioning. The hexane is discarded, the acetonitrile evaporated, and the residue re-dissolved in ethyl acetate. An aliquot is analysed on a gas chromatograph equipped with a fused silica capillary column with a cross-linked silicone stationary phase. Phosmet and its oxygen analogue are quantified with a phosphorus-selective detector (FPD). Recoveries were generally >80% and the limit of quantification for a 10 g sample was 0.05 mg/kg (Meyers, 1994a,b).

According to the official method in The Netherlands phosmet is extracted with ethyl acetate in the presence of sodium sulfate. The organic phase can be injected onto a GLC column without further clean-up or for some crops after clean-up on an SX-3 GPC column with cyclohexane/ethyl acetate as eluent. The LODs reported are between 0.01 and 0.05 mg/kg. Phosmet can also be detected on silica gel TLC plates, after oxidation to its oxon, by a cholinesterase-inhibition method (Netherlands, 1996).

Milk, eggs and animal tissues

Phosmet and phosmet oxon are extracted from tissues with toluene and glacial acetic acid or, for milk and eggs, methylene chloride and glacial acetic acid. Fat is removed from the extract by acetonitrile-hexane partition. The acetonitrile, containing the analyte, is diluted with toluene and the solvents concentrated. An aliquot of the residual toluene solution is analysed by programmed-temperature gas chromatography on a fused silica capillary column, with a chemically bonded liquid phase of methyl silicone containing 6% cyanopropylphenyl groups. Phosmet and phosmet oxon are detected and quantified with a mass-selective detector. Mean recoveries from all samples were 84% for phosmet and 95% for phosmet oxon. With a few exceptions, neither phosmet nor phosmet oxon was detected in any control sample above 0.05 mg/kg, the limit of determination (Curry, 1989).

Phosmet can also be extracted from milk with ethyl acetate. The extract is dried over sodium sulfate, an aliquot is evaporated to dryness and the residue is dissolved in hexane. After partition with acetonitrile, the acetonitrile phase is evaporated to dryness and the residue re-dissolved in ethyl acetate. This solution is suitable for GLC or TLC analysis. The LOD is 0.01 mg/kg for whole milk (Netherlands, 1996a).

Soil

Samples of soil (50 g) are extracted by shaking for one hour with water and toluene. The mixture is centrifuged to separate the toluene, water and soil phases and an aliquot of the toluene phase is removed and analysed by gas chromatography. A gas chromatograph equipped with a nitrogen/phosphorus selective thermionic detector and a fused silica capillary column (polyphenylmethylsiloxane) is used with temperature programming for the determination of the residues. Recoveries of phosmet from control soil fortified in the range of 0.05-3.0 mg/kg were 103-120%, and of phosmet oxon at the same levels 68-100%. The limit of determination was given as 0.05 mg/kg (De Guzman and Iwata, 1986).

In an alternative method distilled water (10 ml) is mixed with the sub-sample of soil (50 g) which is then extracted for 30 minutes with ethyl acetate (100 ml). The ethyl acetate extract is dried and an aliquot analysed by gas chromatography as described above. Recoveries of phosmet and phosmet oxon at 0.02-2.0 mg/kg were 96-99% (California Department of Food and Agriculture, 1986a).

Water

A 50 ml sample of water is shaken for two minutes with 5 ml toluene and a small aliquot of the toluene injected directly into a gas chromatograph equipped as described above (North Coast Laboratories, 1986).

Stability of residues in stored analytical samples

Food crops

A storage stability study was carried out with phosmet on a variety of food crops stored at -20 ± 10^{0}C for periods up to 2½ years (wheat grain and wheat straw), or 3 years (green alfalfa hay, almond nut meat, fresh apples, maize ears, fresh orange fruit, peppers, potato tubers and soya bean seed). The crops were chopped, blended or milled to produce homogeneous material and 25 g samples were fortified with phosmet (25 µg), i.e. at 1.0 mg/kg, and analysed at intervals of 0 days, 3 months, 8 months, 1 year, 2 years, 2½ years (wheat only) and 3 years.

The results showed that phosmet is stable at -20 ± 10^{0}C in almonds, apples, soya beans, and wheat grain and straw for a minimum of 2½ years, and in alfalfa, maize, oranges, peppers and potatoes for a minimum of 2 years. The criterion for stability was that the mean residue level found in duplicate sample analyses should be not less than 75% of the value found on day 0 (McKay, 1989).

Apples and processed apple commodities (sauce, juice and dry pomace) were fortified at 0.5 mg/kg with either phosmet or phosmet oxon and stored at -20 ± 10^{0}C for periods up to 39 months. The results indicated that phosmet and phosmet oxon were both stable in fresh apples, apple juice and dry pomace for 39 months. In apple sauce phosmet and its oxon were stable for 28 months, when the last samples were analysed.(Meyers, 1994a).

Fresh, dried and canned peaches were separately fortified at 0.5 mg/kg with phosmet or phosmet oxon and stored at -20 ± 10^{0}C for more than 3 years. Triplicate samples were analysed at intervals. The results showed that phosmet and the oxon were stable in fresh and canned peaches for periods up to 28 months. In dried peaches, phosmet and phosmet oxon both decreased to about 50% of the initial value in 4 months. The drying process involves dipping peaches in bisulfite solution, which may have affected the results (Meyers, 1994b).

Commodities of animal origin

Samples of cow muscle, fat, kidneys, liver and milk, and chicken eggs were each fortified separately with phosmet and phosmet oxon at 0.5 mg/kg and analysed at 0 day, 1 month, 3 months, 6 months and 1 year after storage at -20^{0}C.

The results show that phosmet is stable in milk, liver and fat for a year when stored at -20^{0}C; recoveries after a year of storage were within 10% of the initial values. In eggs phosmet was stable for six months, and its recovery was still above 70% after a year. In kidneys phosmet was stable with recovery above 80% after six months and nearly 70% after a year, indicating reasonable stability. Phosmet was stable in muscle for one month, but even after a year recovery was nearly 70%.

Phosmet oxon, while generally less stable than phosmet, was stable for one year in fat, 6 months in milk and one month in muscle and eggs. The oxon was relatively stable in eggs for up to 3 months, as shown by a recovery of nearly 70%. Phosmet oxon is unstable in liver and kidneys; its recovery decreased to 18% of the initial level after one hour in liver and to 50% after 5 hours in kidneys. Because the compound is so unstable in these organs, it is unlikely that any residues of phosmet oxon would occur in the commercial product (Curry, 1989).

Soil

A clay loam soil (pH 7.3, organic matter 4.1%, sand 28.9%, silt 45.8%, clay 21.2% and cation exchange capacity 1.5 m eq/100 g) was thoroughly mixed and 25 g samples were fortified with phosmet at 1 mg/kg. The samples were stored at -20 ± 10^0C for up to three years and sub-samples taken periodically for analysis in duplicate. Phosmet was stable in the soil at -20^0C for at least two years (McKay, 1989).

USE PATTERN

Phosmet is a broad-spectrum organophosphorus insecticide used to control a variety of insect and mite pests which attack pome, stone and citrus fruit. It is also used on field, pasture and forage crops. Phosmet is non-systemic and acts by contact and ingestion as a cholinesterase inhibitor.

Phosmet suppresses the two-spotted mite *Tetranychus urticae* and the European red mite *Paratetranychus ulmi* when used in spray programmes on deciduous fruit. A list of the major insects controlled is given in Table 4.

The registered or approved uses of phosmet on food crops are shown in Table 5. The direct use of phosmet in treating livestock for the control of warble fly, ticks and lice on cattle, resulting in residues in animal commodities, was not reported to the Meeting and is not included.

Table 4. Insects controlled by phosmet (a partial listing)

Anarsia lineatella	Peach twig borer
Anthonomus piri	Pear blossom weevil
Anthonomus pomorum	Apple blossom weevil
Anuraphis persicae	Black peach aphid
Aphis pomi	Green apple aphid
Argyresthia ephipella	Cherry fruit moth
Aspidiotus perniciosus	San José scale
Bruchus rufimamus	Broad bean weevil
Carpocapsa pomonella	Codling moth
Ceratitis capitata	Mediterranean fruit fly
Colaspidema atrum	Alfalfa beetle
Dacus oleae	Olive fruit fly
Eriosoma lanigerum	Woolly aphid
Hoplocampa testudinae	European apple sawfly
Polychrosis botrana/Clysia ambiguella	Grape-berry moth
Psylla mali	Apple-tree psylla
Psylla piri	Pear-tree psylla
Rhagoletis cerasi	Cherry fruit fly
Saissetia oleae	Olive scale
Sitona lineata	Pear weevil

Table 5. Main registered uses of phosmet on food crops.

Crop	Country	Form.	Application			PHI, days
			Rate, kg ai/ha	Spray Conc., g ai/l	Number	
Alfalfa	Argentina Australia Canada Mexico Uruguay USA	50 WP 150 EC 50 WP 50 50 WP 70 WSB	0.5 0.04-0.05 1.13 0.5 -1 0.5 0.8-1.0	- 0.38-1.1 2.25-5.6 - -	As required 1-3 1 As required 1	7 2 7 7 7 14
Almonds	Chile USA	50 WP 70 WSB	 3.4	0.5-0.6 2.25-5.6	 2	 30
Apples	Argentina	50 W	-	0.6	Every 25-30 days	n[1]
	Australia	500 WP	-	0.75	Every 3 weeks	21
	Brazil	500 WP	-	1.0	Every 10 days	14
	Canada	50 WP	1.9	-	As required	1
	Chile	50 WP	1.0-1.5	0.5-0.75	As required	
	France	50 WP	-	0.5	As required	15
	Greece	50 WP		1.2-1.5	At 30 day intervals	30
	Italy	50 WP	-	0.75-1.0	As required	30
	Mexico	50	0.5-1			7
	Portugal	50 BT	-	0.5	As required	-
	Spain	20 LE	-	0.75-1.25	As required	30
	Tunisia	50 WP	-	0.5-0.6	As required	15
	Uruguay	50 WP	-	0.5-0.6	Every 18-20 days	7
	USA	70 WSB	1.7-4.1	1.2	As required (max 34 kg/ha)	7
Apricots	Greece USA	50 WP 70 WSB	 1.75-3.4	1.2-1.4 1.0-1.2	At 20 days As required	30 14
Blueberries	Canada USA	50 WP 70 WSB	1.13 1.0	1.13 -	1-2 1-2	15 3
Carrots	Canada	50 WP	1.13	-	1-2	40
Celery	Canada	50 WP	1.13	1.13	1-2	40
Cereals (forage crop)	Australia	150 EC	0.04-0.05	0.4-1.0	Every 3-5 weeks	7
Cherries (sour)	Canada Chile Greece USA	50 WP 50 WP 50 WP 70 WSB	1.9 - 1.7-2.0	 0.5-0.9 1.2-1.4 	As required As required As required	7 - 30 7
Citrus (orange, lemon, grapefruit)	Argentina Brazil Chile Greece Italy Spain Tunisia Uruguay	50 WP 500 WP 50 WP 50 WP 50 WP 20 LE 50 WP 50 WP	- - - - - - - 	0.6 1.0 0.5-0.75 1.2-1.4 0.3-0.75 1.5 0.6-0.75 0.5-0.75	As required As required As required As required As required As required As required	n[2] 14 - 30 30 30 15 14
Clover	Greece	50 WP		1.2-1.4		30
Cotton	Argentina Brazil Cotton Mexico USA	50 WP 500 WP 50 WP 50 WP 70 WSB	0.4-0.75 0.5 0.5-1.0 0.4-0.6	- - 1.2-1.4 1.25-5.0 	As required As required As required[3] Every 3-7 days (max 11 kg ai/ha)	15 14 30 - 21
Grapes	Chile Greece Spain USA (west) USA (east)	50 WP 50 WP 20 LE 70 WSB 70 WSB	1.0-1.5 1.5 1.5-2.4 	0.75-0.9 1.2-1.4 0.15 - -	As required As required As required As required	15 30 30 7 14
Kiwi fruit	Chile New Zealand	50 WP 50 WP	1.0-1.5 2.53	0.75-0.9 1.1	As required 5 times	1 21
Maize	Greece	50 WP		1.2-1.4		30
Melons	Portugal	50 BT		0.5		

Crop	Country	Form.	Application			PHI, days
			Rate, kg ai/ha	Spray Conc., g ai/l	Number	
Nectarines	Chile	50 WP	-	0.5-0.6	As required	14
	Uruguay	50		1.0-1.2	At 18-20 days	14
	USA	70 WSB	1.7-3.3	-	As required	14
Oil palm	Malaysia	50 WP	0.5	0.75	As required	14
Olives	Greece	50 WP	-	1.2-1.4		30
	Italy	50 WP	-	0.75-1.5	As required	30
	Portugal	50 BT	-	0.5	As required	-
	Spain	50 WP	-	1.5	As required	30
	Tunisia	50 WP		0.5-1.0	As required	15
Pasture (forage)	Australia	150 EC	0.04-0.05	0.38-1.1	1-3	2
Peaches	Argentina	50 WP	-	0.6	Every 25-30 days	5
	Australia	500 WP	-	0.5	Every 3 weeks	21
	Brazil	500 WP	-	1.0	As required	14
	Canada	50 WP	1.9			1
	Chile	50 WP	-	0.5-0.6	As required	14
	Greece	50 WP		1.2-1.5	At 30 days	30
	Spain	20 LE	-	1.5	As required	30
	Portugal	50 BT	-	0.6		
	Tunisia	50 WP	-	0.5-0.6	As required	15
	Uruguay	50 WP	-	0.5-0.6	Every 18-20 days	14
	USA	70 WSB	1.7-3.3	-	As required (max 13 kg ai/ha)	14
Pears	Argentina	50 WP	-	0.6	Every 25-30 days	5
	Australia	500 WP	-	0.75	Every 3 weeks	21
	Brazil	500 PM	-	1.0	Every 10 days	14
	Canada	50 WP	1.9			1
	Chile	50 WP	-	0.75-0.9	As required	7
	France	50 WP	-	0.5	As required	15
	Italy	50 WP	-	0.75-1.0	As required	30
	Portugal	50 BT	-	0.5	As required	-
	Tunisia	50 WT	-	0.5-0.75	As required	15
	Uruguay	50 WP	-	0.5-0.6	Every 18-20 days	7
	USA	70 WSB	1.7-5.6	-	As required	7
Peas (fresh and dry)	USA	70 WSB	0.75	-	As required	7
Pecans	USA	70 WSB	2.45	-	As required	14
Plums	Argentina	50 WP	-	0.6	Every 25-30 days	20
	Canada	50 WP	1.9			1
	Chile	50 WP	1.7 -3.3	0.75-0.9	As required	7
	USA	70 WSB	1.7-3.3	-	As required	7
Prunes	USA	70 WSB	1.7-3.3	-	As required	7
Potatoes	Canada	50 WP	1.2			7
	France	50 W	0.5	0.5-0.6	As required	15
	Italy	50 WP	-	0.75-1.0	As required	30
	Portugal	50 BT	-	0.6	As required	-
	Spain	50 WP	-	0.75-1.25	As required	30
	Tunisia	50 WP	0.5	0.5-0.6	As required	15
	Uruguay	50 WP	0.88	-	As required	7
	USA	70 WSB		-	As required	7
Quinces	Argentina	50 WP	-	0.5-0.6	As required	5
	Uruguay	50 WP	-	0.5-0.6	Every 18-20 days	7
Sweet potatoes	USA	5% Dust	125-250 g/ tonne		Apply once post-harvest for storage	-
Walnuts	Argentina	50 WP	-	0.6	As required	5
	USA	50 WP	3.3-6.6		Max 5 application	14

[1] Red Delicious and Granny Smith 20 days, others 15 days
[2] Applied as a bait
[3] Not more than 11 kg ai/ha for every agricultural cycle

RESIDUES RESULTING FROM SUPERVISED TRIALS

Citrus fruits (Table 6). Reports of recent supervised trials (1991) on oranges in Argentina and Brazil were submitted. In Argentina, 0.5 kg ai/ha as a 0.6 g ai/l solution was applied six times and the crop harvested 7 days after the last application. Residues of phosmet were found to be confined entirely to the peel (0.27-1.0 mg/kg). Phosmet residues in the whole fruit were in the range 0.07-0.32 mg/kg. Residues of phosmet oxon were below the limit of quantification (<0.05 mg/kg).

In Brazil, two different formulations were used in supervised residue trials at concentrations of 1.0 g ai/l (the recommended rate) and 2.0 g ai/l. Oranges were harvested 14 days after the last of 5 applications. Only the pulp was analysed for parent phosmet residues, which were below the limit of quantification (<0.05 mg/kg).

Pome fruit (Table 7). Supervised residue trials on apples and pears have been carried out in Australia, Brazil, Canada, Germany, The Netherlands, New Zealand, the UK and several States of the USA. Many of the trials were carried out at rates above those now recommended.

Table 6. Residues of phosmet from supervised trials on oranges.

Country Year	Application			PHI, days	Sample	Residue, mg/kg	Reference
	Form. Rate, kg ai/ha	Conc., g ai/l	No.				
Argentina 1991	50 WP 0.5	0.6	6	7	Whole fruit	0.32	Grant and Meyers, 1992a
					Peel	1.0	
					Pulp	<0.05	
			6	7	Whole fruit	0.13	
					Peel	0.60	
					Pulp	<0.05	
			6	7	Whole fruit	0.07	
					Peel	0.27	
					Pulp	<0.05	
Brazil 1991	500 WP-	1.0	5	14	Pulp	<0.05	Suchek, 1992
	450 SC	2.0	5	14	Pulp	<0.05	
	-	1.0	5	14	Pulp	<0.05	
	-450 SC	1.0	5	21	Pulp	<0.05	
		2.0	5	14	Pulp	<0.05	
		2.0	5	21	Pulp	<0.05	

Table 7. Residues of phosmet from supervised trials on pome fruits.

Commodity Country Year	Application			PHI, days	Sample	Residue[1] mg/kg	Reference/Remarks
	Form. Rate, kg ai/ha	Conc., g ai/l	No.				
Apples Brazil 1992	500 PM-	1.0	7	14	Peeled fruit	<0.05	Suchek, 1993a
		2.0	7	14		<0.05	
	450 SC	1.0	7	14		<0.05	
		1.0	7	21		<0.05	
		2.0	7	14		<0.05	
		2.0	7	21		<0.05	
Apples Canada 1965	50 WP 2.35	-	1	0	Whole fruit	2.16	A 1004
				7		1.4	
				14		1.3	
				21		0.87	
Apples Germany 1974	50 WP-	1.5	1	0	Whole fruit	1.4-2.9[2]	Apple report R3/12 1974
				6		1.1-2.1	
				13		0.8-1.5	
				20		0.4-0.7	

Commodity Country Year	Application			PHI, days	Sample	Residue[1] mg/kg	Reference/Remarks
	Form. Rate, kg ai/ha	Conc., g ai/l	No.				
Apples Germany 1977	50 WP 1.125	2.25	6	0 8 15 22 29	Whole fruit	2.1 0.74 0.52 0.65 0.52	Apple report R3/13 1977
Apples Germany 1977	50 WP 1.5	0.75	7	0 7 14 21 28	Whole fruit	1.4 1.4 1.0 0.58 0.47	Apple report R3/14 1977
Apples Germany 1977	50 WP 1.125	0.75	6	0 7 14 21 28	Whole fruit	2.3 1.7 0.83 0.70 0.68	Apple report R3/15 1977
Apples Netherlands 1970	50 W	1.5	1	0 7 14 21	Whole fruit	1.3-2.1[3] 0.7-1.3 0.34-0.95 0.13-0.65	Apple report R3/46 1970 Oxon <0.10 mg/kg
	50 WP	1.5	1	0 7 14 21	Whole fruit	1.5-3.4 1.7-2.3 0.6-2.0 0.24-0.59	Apple report R3/45 1970 Oxon <0.1 mg/kg
Apples USA Washington 1965	50 WP 4.48	-	3	1 7 14 21	Whole fruit	4.9 4.2 2.7 2.6	A 906
Apples USA Washington 1965	50 WP 4.48	-	3	1 7 14 21	Whole fruit	3.8 1.8 1.3 <0.4	A 1660
Apples USA Oregon 1965	50 WP 4.48	-	1	1 7 14 21	Whole fruit	5.7 3.4 2.2 1.0	A 1563
Apples USA California 1965	50 WP 1.96	-	2	1 8 15 23	Whole fruit	1.67 1.68[4] 1.2-1.8 0.9-1.4 1.1-1.8	A 1751
Apples USA 1965	50 WP 1.7	-	9	1 7 14 21	Whole fruit	2.6-3.75[4] 2.65-3.7 0.46-0.66 0.74-0.78	A 1939
Apples USA Virginia 1965	50 WP 3.8	-	1	1 7 14 21	Whole Fruit	8.9-10.0[4] 6.3-7.3 3.2-4.5 2.9-3.2	A 1948
Apples USA Pennsylvania 1965	50 WP 4.2		6	0 7 14 21 28 0 7 14 21 28	Whole fruit	4.0-4.6[4] 2.4-2.8 1.4-1.6 0.9-1.1 0.51-0.75 6.1-6.5 4.1-4.3 2.6-3.3 2.1-2.1 1.0-1.3	A 1953
Apples USA N.Y.	50 WP 2.2	-	6	1 8 15	Whole fruit	5.4 3.4 2.8	A 1977

Commodity Country Year	Application Form. Rate, kg ai/ha	 Conc., g ai/l	 No.	PHI, days	Sample	Residue[1] mg/kg	Reference/Remarks
1965	 3.8 6	 - - 	 8 9	22 7 14 21[5] 1 7 14 21[5]		1.3 0.69 0.68 0.32 1.7 1.4 0.38 0.26	 A 1962 A 1978 A 1983
Apples[6] USA 1990	50 WP 4.48	-	9	7 7	Whole fruit	12.9 3.3	Meyers et al.,1991a
Pears Canada 1965	50 WP 3.36	-	3	1 7 14 28	Whole fruit	1.9 0.65 0.26 0.1	4515
Pears Canada 1965	50 WP 2.24	0.6	2	1 7 14 21	Whole fruit	2.3 0.85 0.70 0.52	Pear report R3/18 1965)
Pears USA 1965 1967	50 WP 5.6 50 WP 6.7-9.0	1.2 -	1-2 3	1 8 15 22 9	Whole fruit	2.4 1.7 1.2 0.8 0.26	Pear report R3/17 1965 oxon 0.18 mg/kg
Pears USA 1973	70 WP 6.3	0.84	3	0 7 14	Whole fruit	3.4 1.3 0.45	Pear report R3/44 1973
Pears UK 1970	50 WP 1.12 2.24	2 2		36 36	Whole fruit	0.22 0.25	Pear report R3/42 oxon 0.03, 0.04 mg/kg

[1]as phosmet
Netherlands (1970): phosmet oxon <0.01-0.06 mg/kg
USA (1990): phosmet oxon <0.05 mg/kg
Other countries except UK: oxon not detected or not analysed
[2]6 varieties grown at one site were sampled separately. The lowest and highest residues are reported
[3]Range in triplicate analyses
[4]Replicate samples taken from the same plot
[5]Sampling was continued until 47-49 days
[6]Used for a processing study

Even at higher than recommended rates, the residues of phosmet found after a PHI of 7 days were all below 5.0 mg/kg, except in the trial in Virginia in 1965 and the 1990 trial carried out as a processing study in the USA where a total of 40 kg ai/ha was applied (as 9 separate applications) and a residue of phosmet of 12.9 mg/kg was found in the whole unwashed apples 7 days after the last application (according to US GAP 33.6 kg ai/ha is the maximum total application).

Residues of phosmet in pears in supervised residue trials in Canada and the USA in 1964 and 1965 reviewed by the 1981 JMPR were all below 5 mg/kg following applications approximating GAP and a PHI up to 7 days.

Stone fruit (Table 8). Supervised residue trials were carried out on apricots (USA), nectarines (USA), peaches (Canada and USA), plums (Chile and USA) and prunes (USA).The results are shown in Table 8.

Two apricot trials in the USA (California) at an application rate of 4.2 kg ai /ha (1.25 times GAP) yielded residues of 1.19 and 2.76 mg/kg at 14 days PHI. The oxon residues were 0.01 and 0.02 mg/kg.

Residues of phosmet in peaches were generally below 5 mg/kg after a 7 or 14-day PHI, except in two trials in the USA. In the 1964-65 trials at rates within or close to those specified by GAP (4-8 applications at rates up to 2.24 kg ai/ha in most of the trials), residues of phosmet up to 11 mg/kg were found 5-10 days after the last application. The highest residue at about 14 days after application was 6.8 mg/kg. In the 1990 US trial phosmet was applied 10 times at 3.36 kg ai/ha (a total of 33.6 kg ai/ha, almost three times the maximum recommended total of 13 kg ai/ha, specifically for a processing study. The residue of phosmet in the peaches was about 13 mg/kg 14 days after the last application. This was the only trial in which finite residues of phosmet oxon were found, at a maximum of 0.08 mg/kg.

Four trials on nectarines were reported. In two of them (Table 8) the samples were taken from 0 to 25 days after application. In the other two trials, with application rates of about 4.2 kg ai/ha, the residues were 0.22 mg/kg and 0.35 mg/kg at days 31 and 29 following one and two applications respectively.

In one set of supervised residue trials on plums in Chile (1991) above the recommended spray concentration the residues were all below 0.55 mg/kg, even at a 0-day PHI.

Two trials in 1965 and 1967 in the USA (California) on plums at GAP rates or higher, produced maximum residues of 0.48 mg/kg at a 9-day PHI, and similar trials also in California on prunes in the same years gave residues of 0.53-2.6 mg/kg in the dried prunes after harvesting at 1-3 day PHIs.

More recent trials were carried out on plums and prunes in California in 1993. Five applications of Imidan 50 WP were made at the label recommended rate of 3.4 kg phosmet/ha, with intervals of about 14 days between applications. Fresh plums and prunes were harvested for analysis 7 days after the final application. Phosmet residues in the pitted whole fruit from the three separate sub-plots were 0.40-0.43 mg/kg in plums and 1.8-2.3 mg/kg in prunes. No residues of phosmet oxon (<0.05 mg/kg) were found in any sample of plums or fresh prunes (Dykeman, 1994a,b).

A separate trial was carried out on prunes for a processing study. The prunes were treated with four applications of Imidan 50 WP at the maximum recommended rate of 3.4 kg ai/ha followed by one application at 6.8 kg ai/ha. The fresh prunes were harvested 7 days after the final application for analysis and processing. The residues of phosmet in the fresh prunes were 2.6 mg/kg in both samples. No residues (<0.05 mg/kg) of phosmet oxon were found (Dykeman, 1994c).

Table 8. Residues of phosmet from supervised trials on stone fruits carried out with 50 WP formulations.

Commodity country Year	Application			PHI, days	Sample	Residue, mg/kg	Reference Remarks
	Rate, kg ai/ha	Conc., g ai/l	No.				
Apricots USA 1967 California	4.2	-	1	0 7 14	Whole fruit[1]	9.4 (0.21) 4.7 (0.016) <u>2.8</u> (0.018)	A 396
Apricots USA 1967 California	4.2	-	1	0 7 14 21 28	Whole fruit[1]	11 (0.36) 4.2 (0.08) <u>1.2</u> (0.01) 1.6 (0.06) 0.09	A 397

Commodity country Year	Application			PHI, days	Sample	Residue, mg/kg	Reference Remarks
	Rate, kg ai/ha	Conc., g ai/l	No.				
Nectarines USA 1967	4.2	-	1	0 7 14 21	Whole fruit[1]	4.0 (0.05) 2.2 (0.01) 0.55 0.05	B 1090
Nectarines USA 1965	2.24	-	1	3 9 16 25	Whole fruit	0.67 0.81 <u>0.45</u> <0.4	A 1734
Peaches Canada 1965	2.24	-	1	1 7 14 21	Whole fruit	10 3.4 <u>1.5</u> 1.7	A 1986
Peaches USA Pennsylvania 1965	2.8	-	4	0 7 14 21 28	Whole fruit[2]	10, 13 11, 11 3.1, <u>6.8</u> 1.9, 2.3 1.7, 2.1	A 1957
Peaches USA California 1965	2.24	-	1	1 7 14 21	Whole fruit[2]	2.6, 3.4 2.0, 2.8 1.2, <u>1.6</u> 0.51, 0.81	A 1416
Peaches USA Virginia 1965	2.24	-	5	1 7 14 21	Whole fruit[2]	3.0, 3.3 0.96, 1.2 0.59, <u>0.87</u> 0.43	A 1940
Peaches USA 1965 Georgia	2.24	-	1	1 7 14	Whole fruit[2]	6.3, 13 4.7, 10 3.0, <u>6.4</u>	A 2403
Peaches USA California 1965	2.24	-	1	3 9 16 25	Whole fruit	3.8 1.8 <u>1.2</u> 0.77	A 1420
Peaches USA California 1965	2.24	-	5	2 9 15 23	Whole fruit[2]	1.6, 2.5 1.1, 1.9 0.68, <u>0.78</u> 0.44, 0.49	A 1726
Peaches USA 1963	5.6	-	1	0-3 5-10 11-16	Whole fruit	6.2 5.2 -	FAO/WHO 1982
Peaches USA, 1965 Connecticut	1.68	-	9	1 7 14	Whole fruit[2]	5.9, 6.3 3.3, 3.7 2.1, 2.9	A 1944
Peaches USA Michigan 1965	1.12	-	8	1 7 12 21	Whole fruit	1.9 0.87 0.28 <0.1	A 2027 A 2031
Peaches USA, 1966 Connecticut	1.12	-	8	1 7 14	Whole fruit[1]	1.8 (0.1) 1.9 (0.14) 0.93 (0.06)	A 2093
Peaches USA 1990	3.36	-	10	14	Whole fruit	13	Meyers *et al.*, 1991b oxon max. 0.08 mg/kg
Plums Chile 1991	1.4	-	1	0 3 7 10	Whole[3] plums	0.16(0.46) 0.08(0.22) 0.12 (<u>0.55</u>) 0.07(0.28)	Grant and Meyers, 1992b
Plums (late Santa Rosa) USA (CA) 1965	2.24	0.6	1	3 9 16 25	Whole plums	0.55 <u>0.48</u> 0.28 <0.2	Plum report R3/32

Commodity country Year	Application			PHI, days	Sample	Residue, mg/kg	Reference Remarks
	Rate, kg ai/ha	Conc., g ai/l	No.				
Prunes (French) USA (CA) 1965	2.8	0.60	3	1 8 14 21	Dried prunes[4]	2.3 2.2 2 1.1	Prune report R3/37 1965
Plums USA (CA) 1967	4.2	0.9	1	27-28	Whole plums	<0.1-0.12	Plum reports R3/33, 34, 41 oxon <0.10 mg/kg
Prunes[5] USA (CA) 1967	4.2	0.90	1	3 38 52	Dried prunes	0.53-1.8 0.07-0.45 <0.1(3)	Prune rep. R3/38, 39, 40, 1967 oxon <0.1 mg/kg
Plums Castleman USA (CA) 1974	3.5	0.63[6]	1	35 65	Whole plums	0.09 <0.05-0.06	Plum reports R3/31, 35 Oxon <0.05 mg/kg
Plums[7] (Angeleno) USA (CA) 1993	3.4	-	5	7	Pitted whole plums	0.41	Dykeman, 1994b oxon <0.05 mg/kg
Fresh prunes USA (CA) 1993	3.4	-	5	7	Pitted whole prunes[8]	1.80 2.3 1.8	Dykeman, 1994a Oxon <0.05 mg/kg
Fresh prunes[7] USA (CA) 1993	4 x 3.4 + 1 x 6.8[10]	-	5	7	Pitted whole prunes	2.6, 2.6	Dykeman, 1994c Oxon <0.05 mg/kg

[1]Oxon residues in parentheses
[2]Residues in duplicate samples from the same plot
[3]Total of 8 trials. Figures are average values of 6-15 determinations, with maximum values in parentheses. Phosmet oxon <0.05 mg/kg. Volume and concentration of spray were not reported
[4]Results are means of triplicate analyses by spectrophotometric method
[5]3 trials
[6]70 WP formulation
[7]Replicate analyses by spectrophotometric method described in FAO/WHO, 1977
[8]Results from three replicate sub-plots
[9]Trial was carried out to obtain treated fruits for processing
[10]Twice the recommended label rate

Grapes (Table 9). The results of supervised residue trials on grapes in Canada and the USA in the period 1963-69 were evaluated by the 1981 JMPR. No new trials have been submitted. The results of the trials are reported in more detail in Table 9.

Table 9. Residues of phosmet from supervised field trials on grapes with 50 WP formulations.

Country Year	Application			PHI, days	Sample	Residue, mg/kg	Reference Remarks
	Rate, kg ai/ha	Conc., g ai/l	No.				
Canada Orlando 1968	1.4	-	4	1 7 14 21	Whole fruit	11 7.2 6.0 2.9	B 0535
USA New York 1963	0.7, 0.7, 0.9	-	3	0 10 14 28	Whole fruit[1]	3.9, 4.3 1.1, 1.4 0.1, 2.5 0.3, 0.8	Cornell 1963
USA New York 1963	1.4, 1.4, 1.8	-	3	0 7 14 28	Whole fruit[1]	8.3 3.6, 4.2 4.0, 4.2 2.2	Cornell 1963

Country Year	Application			PHI, days	Sample	Residue, mg/kg	Reference Remarks
	Rate, kg ai/ha	Conc., g ai/l	No.				
USA New York 1965	0.7, 0.7, 0.9	-	3	7 15 21 28 42	Whole fruit[1]	2.1 1.2, 1.4 0.7, 0.9 0.6, 0.76 0.28, 0.44	Cornell 1965a
USA New York 1965	1.4, 1.4, 1.8	-	3	7 15 21 28 42	Whole fruit[1]	3.8, 4.1 3.2, 3.3 1.7, 2.1 1.1, 1.9 0.9, 1.1	Cornell 1965a
USA New York 1965	0.7, 0.7, 0.84	0.6	3	0 7 14 21 28	Whole fruit[1]	0.32, 1.76 0.36, 0.4 0.84, 0.96 0.32, 0.4 0.16, 0.2	Cornell 1965b
USA New York 1965	0.7, 0.7, 0.84	1.2	3	0 7 14 21 28	Whole fruit[1]	2.3, 2.4 1.36, 2.0 1.3, 1.4 0.96, 1.1 0.4, 0.56	Cornell 1965b
USA New York 1966	0.7, 0.7, 0.9	,	3	0 10 14 21 35	Whole fruit[1]	2.5, 2.6 1.5, 1.6 0.67, 0.75 0.7, 0.75 0.48, 0.55	Cornell 1966
USA New York 1966	1.4, 1.4, 1.8	,	3	0 7 14 21 35	Whole fruit[1]	5.9, 6.2 3.9, 4.8 1.4, 2.8 1.6, 2.0 1.4, 1.5	Cornell 1966
USA New York 1967	1.4, 1.4, 1.8	,	3	0 7 14 21 24 42	Whole fruit[1]	8.2, 8.8 4.4, 5.0 4.0 4.0, 4.8 3.3 2.6, 3.7	Cornell 1967
USA New York 1967	2.1, 2.1, 2.8	-	3	0 7 14 21 28 35	Whole fruit[1]	11 15 8.5, 10 6.8, 9.2 7.0, 7.6 6.2, 6.5 3.8, 6.2	Cornell 1967
USA, CA 1969	1.7 Imidan 5 Dust		2	0 7	Whole fruit	0.42 0.24	B-0665
USA, CA 1969	1.7 Imidan 5 Dust		1	0 7 14 21	Whole fruit[2]	1.6 0.61 (oxon 0.16) 0.2 0.43	B-0651
Grapes[2] USA, CA 1969	1.7 Imidan 5 Dust		1	4 7 31	Whole fruit	0.2-0.48 0.1-0.17 0.22	B-106, 107 B-1073,1075 B-0105

[1]Residues in duplicate samples taken from the same plot [2]5 trials at different locations in California

Olives (Table 10). Supervised residue trials were carried out on olives in France, Italy and Spain in 1975 and 1976, and in Greece in 1965. At the maximum registered concentration of 1.5 g ai/l, the residues in the whole fruit one day after treatment were in the range of 1.1-2.5 mg/kg. Generally, the residues declined, with a half-life of around 10 days or less, to <0.02-0.34 mg/kg after a PHI of 28-30 days.

Kiwifruit (Table 11). Supervised residue trials reported to the 1976 JMPR showed residues at a 21-day PHI mainly below 10 mg/kg, except for two high results (12 and 17 mg/kg) following seven applications at 1.5 times the GAP rate.

Residue data from trials in 1980 and 1986 were reviewed by the 1987 JMPR. The residues after a 21-day PHI were all well below 5 mg/kg (FAO/WHO, 1988a).

Table 10. Residues of phosmet from supervised trials on olives with 50 WP formulation.

Commodity Country Year	Application			PHI, days	Sample	Residue, mg/kg	Reference Remarks
	Rate, kg ai/ha	Conc., g ai/l	No.				
Olives (table)		1	1	1	Whole fruit	1.6 (2.4)	Mestres, 1976
France 1976				7	(pulp)	0.73 (1.1)	
				14		0.56 (0.80)	
				21		0.54 (0.77)	
				28		0.12 (0.18)	
				35		0.008 (0.012)	
		1.5	1	1	Whole fruit	2.3 (3.5)	
				7	(pulp)	1.6 (2.2)	
				14		1.4 (2.1)	
				21		0.43 (0.60)	
				35		0.24 (0.38)	
Olives (table)		-	1	1	Whole fruit	1.2 (1.5)	Mestres, 1975
France				7	(pulp)	0.62 (0.80)	
1975				14		0.15 (0.19)	
				20		0.08 (0.10)	
				29		0.02 (0.02)	
	1.5	-	1	1	"	1.1 (2.7)	
				7		0.39 (0.6)	
				14		0.06 (0.10)	
				20		<0.03	
				29		<0.03	
Olives		-	1	1	Whole fruit	1.4 (1.8)	Mestres, 1975
(for oil)				7	(pulp)	1.3 (1.8)	
France				14		0.44 (0.59)	
1975				21		0.25 (0.33)	
				30		0.11 (0.15)	
			1	1	Whole fruit	1.3 (1.9)	
				7	(pulp)	0.81 (1.1)	
Olives				14		0.11 (0.16)	
				21		0.04 (0.06) 0.07 (0.01)	
Olives (for oil)		1	1	1	Whole fruit	2.2 (3.50	Mestres, 1976
France				7	(pulp)	0.75 (1.1)	
1976				14		0.6 (0.9)	
				20		0.27 (0.42)	
				28		0.13 (0.22)	
				35		0.23 (0.45)	
				42		0.07 (0.13)	
		1.5	1	1	Whole fruit	4.3 (6.6)	
				7	(pulp)	1.5 (2.2)	
				14		0.9 (1.4)	
				20		0.53 (0.9)	
				28		0.34 (0.56)	
				35		0.29 (0.57)	

Commodity Country Year	Application			PHI, days	Sample	Residue, mg/kg	Reference Remarks
	Rate, kg ai/ha	Conc., g ai/l	No.				
				42		0.21 (0.40)	
Olives[1]	-	3.0		10	Whole fruit	0.77 (2.5)	Batchelder, 1966
(for oil)					(oil)	0.44 (1.5)	
Greece						0.6 (2.1)	
1965				15		0.57 (2.4)	
						0.25 (1.4)	
						0.54 (2.1)	
Olives	-	1.0	1	1	Whole fruit	1.7 (3.0)	Mestres, 1977
(for oil)				8	(pulp)	0.4 (0.73)	
Italy				15		0.19 (0.31)	
1976				22		0.14 (0.24)	
				29		0.09 (0.15)	
	-	1.5	1	1		2.5 (4.5)	
				8		1.1 (1.9)	
				15		0.54 (0.93)	
				22		0.19 (0.31)	
				29		0.16 (0.25)	
	-	2.0	1	1		4.5 (7.6)	
				8		1.7 (3.1)	
				15		0.59 (0.96)	
				22		0.34 (0.55)	
				29		0.25 (0.40)	
	-	3.0	1	1		5.8 (9.7)	
				8		1.5 (2.7)	
				15		0.58 (0.95)	
				22		0.25 (0.40)	
				29		0.12 (0.19)	
Olives[2]	-	1.25	4	6	Whole fruit	>20 (0.7)	Agallauel, 1977
(green)				13		0.8 (0.04)	
Spain				20		0.5 (<0.02)	
1976				27		<0.02 (<0.02)	

[1]Phosphomolybdate spectrophotometric method. Mean of three replicates
[2]Phosmet oxon residues were 0.3 and 0.7 mg/kg at 6 days PHI and in the range <0.02-0.07 mg/kg at other intervals

Table 11. Residues of phosmet from supervised field trials on kiwifruit in New Zealand.

Year	Application			PHI, days	Residues, mg/kg	
	Rate, kg ai/ha	Conc., g ai/l	No.		Whole fruit	Flash
1974-75[1]	1.12		2	10	5.8-9.5	0.5-1
	1.12		7	10	7.2-25	0.8-2.5
1976	0.75		7	10	4.8-8.8	0.3-1.3
	1.6-1.7		6	1	5-11	
				7	4-8	
				14	3-8	
				24	5-7	
				35	3-4	
1976	3.7-3.8		6	1	14-27	
				7	14-18	
				14	18-36	
				24	10-12	
				35	10-18	

[1]Abbott and Hayward varieties

Peas (Table 12). Peas were grown in three separate sub-plots in the Washington and Oregon states, and treated three times with 'Imidan' 50 WP at a nominal rate of 1.12 kg phosmet/ha (about 1.5 times the recommended rate) with 7-8 days between applications. Peas, pods and forage were sampled 7 days and dry pea hay 10 days after the last application (Dykeman, 1994e). The samples were analysed for phosmet and phosmet oxon by a GLC with flame photometric detection.

Table 12. Residues of phosmet in peas from supervised trials. in the USA with 50 WP formulations, 1993 (Dykeman, 1994e).

Commodity	Application			PHI, days	Sample	Residue, mg/kg in replicate analytical samples
	Rate, kg ai/ha	Conc., g ai/l	No.			
Peas[1] (succulent)	1.12	-	3	7	Peas Pods Forage	<0.05 (3) 0.2, 0.51, 0.30 3.0, 2.7, 5.7
Peas[2] (succulent)	1.12	-	3	7	Peas Pods Forage	<0.05 (3) 0.26, 0.19, 0.15 3.5, 3.6, 3.1
Peas[2] (dried)	1.12	-	3	7 10	Peas[1] Hay[3]	0.068, 0.084, 0.063 17, 13.6, 10.9
Peas[2] (dried)	1.12	-	3	7 10	Peas[1] Hay[4]	<0.05 (3) 3.3, 2.5, 4.0

[1]Residues of phosmet oxon all <0.05 mg/kg
[2]See also Table 13 (Forage crops)
[3]Residues of phosmet oxon 0.16-0.28 mg/kg
[3]Phosmet oxon 0.06-0.08 mg/kg

Potatoes. Several trials were carried out in The Netherlands (1970), the USA (1970, 1973, 1974 and 1975) and Canada (1970). Some of the data were reported to the 1976 and 1978 Meetings. Only one residue (0.04 mg/kg) was above the limit of determination of either the parent compound or its oxygen analogue.

Supervised trials were carried out in five different States of the USA in 1993 on three varieties of potato. At each site there was an untreated plot and a treated plot divided into three sub-plots, samples from which were analysed separately. 'Imidan' 50 WP was applied one to five times at nominal rates ranging from 0.56 to 1.56 kg phosmet/ha, with 10-14 days interval between applications. Samples of tubers taken 7 to 85 days after the last application were analysed for phosmet and phosmet oxon by GLC. No residues of phosmet or its oxon (<0.05 mg/kg) were found in any of 71 samples from 24 sites (Dykeman, 1994g, 1995b; Potato summary report R3/49).

In a separate trial for a processing study (see below) potato plants were sprayed with 'Imidan' 50 WP four times at 1.12 kg ai/ha and once at 4.5 kg ai/ha and the tubers harvested 7 days after the final application. Residues of phosmet in tubers from the three treated sub-plots were 0.09-0.11 mg/kg and those of phosmet oxon were all <0.05 mg/kg (Dykeman, 1994f).

Two trials in 1994 in the States of Maine and Washington were to compare the residues resulting from the application of the 50 WP and 2.5 EC formulations. The formulations were applied five times at 1.12 kg phosmet/ha, with 10-14 days between applications. Samples of mature potato tubers harvested 7 days after the final application contained no quantifiable residues of phosmet or its oxon (Dykeman, 1995b).

Tree nuts. Data on trials on tree nuts were presented to the 1978 JMPR. It was reported that the residues in almonds, filberts, pecans and walnuts were all below 0.08 mg/kg in the nut meat, most being in the range 0.01-0.05 mg/kg. Residues in almond hulls ranged up to 5.6 mg/kg (FAO/WHO, 1979).

In a supervised residue trial in 1994 in California, walnut trees were treated five times with Imidan 70 WP at a nominal rate of 6.72 kg phosmet/ha with 18-21 day intervals between applications (three replicate plots of two trees with an untreated control plot). Samples of walnuts taken from the replicate plots 14 and 27 days after the final application were analysed for phosmet and phosmet oxon by GLC with flame photometric detection. No residues (<0.05 mg/kg) of phosmet or phosmet oxon were detected in five samples; the sixth, taken at day 14, contained 0.06 mg/kg phosmet (Dykeman, 1995a).

Cotton seed. Six supervised trials were carried out on cotton in Brazil in 1992 with five spray applications of PM and SC formulations at rates between 0.75 and 2.25 kg ai/ha (1.5-4.5 times the Brazilian GAP rate). Residues of phosmet in all the seed samples were <0.05 mg/kg at harvest 15 or 21 days after the last application (Suchek, 1993b).

Fodder crops (Table 13). Supervised residue trials have been reported on a variety of crops for animal consumption: alfalfa (USA), Bermuda grass (USA), lupins (Australia), maize (USA), peas (Australia and USA), rape (Australia) and soya beans (USA). A number of the trials were evaluated by the JMPR in 1976, 1978 and 1984.

Table 13. Residues of phosmet in fodder and forage crops from supervised trials.

Commodity Country or State Year	Application			PHI, days	Residue, mg/kg		Ref/Report no.
	Form. Rate, kg ai/ha	Conc., g ai/l	No.		Phosmet	Oxon[1]	
Alfalfa fresh plant							
	3EV, 0.56		1	1	8.9, 13.8		3157
1962				3	0.88, 1.0		
Alabama				7	0.27		
	1.12		1	1	93, 93		
				3	14, 24		
				7	0.42, 0.65		
				13	0.3		
Alabama	3 EV		1	1	148, 175		3724
1963	1.12			7	9.9, 10.6		
				14	1.8, 2.1		
Alabama	3 EV		1	1	64, 105		3724
1963	1.12			7	4.4, 5.5		
				14	0.76, 0.84		
Alabama	3 E		1	1	38, 45		3778
1963	1.12			7	3.2, 3.7		
				14	<0.2, 0.21		
				21	<0.2, <0.2		
Alabama	3 E		1	1	7.2, 19		3778
1963	1.12			7	1.3, 1.5		
				14	<0.2, 0.24		
				21	<0.2, <0.2		
Md. 1962	3 EV		1	1	64		3826
	1.12			7	16		
				14	1.6		
Md. 1963	3 E		1	1	64		3969
	1.12			7	12.3		
				14	2.1		
				21	2.43		
Md. 1963	3 EV		1	1	126		3827
	1.12			7	13		
				14	2.2		
N.Y. 1964 1965	36% EC 1.12		1	1 8 14 27	46.7, 40.2 2.5, 1.7 0.44, 0.84 <0.4, <0.4		Alfalfa report R3/16 (A1904)

Commodity Country or State Year	Application Form. Rate, kg ai/ha	Conc., g ai/l	No.	PHI, days	Residue, mg/kg Phosmet	Oxon[1]	Ref/Report no.
Nebraska	50 W		1	1	12, 18	0.05, 0.04[2]	B-002
1967	0.56			7	<0.01, 0.33		B-0001
				14	0.12, 0.21		
				21	1.2, 0.22		
Nebraska	50 W		1	1	59, 110[2]	0.27	B-002,
1967	1.12			7	0.5, 0.88		B-001
				14	0.19, 0.21		
				21	0.54, 0.05		
1967	50 W		1	1	50	0.12	A-0440
	1.12			7	1.4		
				14	0.26		
				21	0.15		
1967	50 W		1	0	58, 75	0.43, 0.52	A-02030
	2.24			7	5.7, 5.7	0.04, 0.04	
				14	0.8, 0.87		
				21	0.11, 0.11		
Pennsylvania	3 E		1	1	67		3972
1963	1.12			7	9.1		
				15	0.77		
Virginia	50 W		1	0	101	0.34	A-0447
1967	1.12			7	9.9	0.06	
				14	1.2		
				21	0.42		
New Jersey	3 E		1.	0	42	0.12	A-0405
1967	1.12			7	0.57		
				14	0.4		
				21	<0.01		
Florida	50 W		1	1	4.4, 4.5	0.1, 0.12	B-0102
1967	1.12			7	0.77, 14	0.04	B-0104
				14	3.0, 3.5	0.04, 0.07	B-0106
				21	0.38, 0.50	0.03	B-0108
1968	50 W	2.24	1	0	86	0.67	A-0479
				7	32	0.39	
				14	0.13		
				21	2.6	0.1	
				28	1.3		
Alfalfa hay							
	3 E		1	7	0.27, 6.6		3968
N.Y. 1963	1.12			15	<0.2, 0.59		
				33	<0.2, <0.2		
Pennsylvania	3 E		1	1	31, 80[1]		3971, 3972
1963	1.12			7	11, 3.2		
				15-16	0.6, 1.1		
New Jersey	3 E		1.	3	61	1.53	A-0405
	1.12			10	0.77		
				17	0.15		
				24	0.2		
Iowa 1967	50 W 0.56		1	0	29.5	0.15	B-0003
				7	0.015		B-0012
				14	1.5		B-0013
	1.12		1	0	71	0.26	B-0003
				7	0.07		B-0012
				14	4.5		B-0013

Commodity Country or State Year	Application			PHI, days	Residue, mg/kg		Ref/Report no.
	Form. Rate, kg ai/ha	Conc., g ai/l	No.		Phosmet	Oxon[1]	
Lupins (whole plant analysed)							
Australia 1991	0.053 0.105 0.053 0.105	- - - -	2 2	7 14 7 14	0.14 0.07 0.50 0.09		Marcus 1992a
Peas[3]							
Australia Whole plant	150 EC	-	2	7	1.1, 2.0		Markus, 1992b
1991	0.053			14	3.1		
Seeds and	0.105	-	2	7	4.4		
Pods				14	4.1		
	0.053	-	2	7	0.22, 0.18		
				14	0.10, 0.11		
	0.105	-	2	7 14	0.44 0.27		
Rape (whole plant analysed)							
Whole plant	0.053	-	2	3	0.39		Markus, 1991
Australia				7	0.20		
1988				14	0.14		
				24	0.06		
	0.105	-	2	3	1.1, 1.2		
				7	0.38, 0.50		
				14	0.14, 0.12		
				24	0.07, 0.07		

[1]Oxon residues below the limit of detection are not reported
[2]Oxon max. 0.5 mg/kg at day 0
[3]See also US data in Table 12.

FATE OF RESIDUES IN STORAGE AND PROCESSING

In processing

Studies have been carried out to determine the effect of processing on phosmet residues in apples, apricots, peaches, prunes, grapes, olives and potatoes.

Apricots. Three field trials were reported in which apricot trees were treated once with 2.8 kg ai/ha and the fruits sampled 14 days later. The fruits were dried and analysed about a year later. The phosmet residues were 0.29, 0.63 and 1.63 mg/kg. Phosmet oxon (0.19 mg/kg) was detectable only in the last sample.

Apples. A field trial was conducted in the apple-growing region of Washington, USA. One plot of apple trees was treated with nine applications of 'Imidan' 50 WP at 4.48 kg ai/ha per application, giving a total of 40 kg ai/ha (1.2 times the maximum allowed amount). The last application was made seven days before harvest as required by the current US label. Apples were harvested and shipped fresh and chilled to a pilot-scale food processor which converted the whole unwashed raw apples into the processed products within 14 days.

Whole unwashed raw apples and apple products were stored frozen until analysis. Samples were analysed for phosmet and phosmet oxon by gas chromatography with a limit of determination of

0.05 mg/kg for each compound. The mean recoveries of phosmet and phosmet oxon from fortified samples were 92% and 100% respectively. The fortification range was from 50 to 0.05 mg/kg; all recoveries were above 70%. The results are shown in Table 14 (Meyers *et al*., 1991a).

Table 14. Phosmet and phosmet oxon residues in processed apple products.

Sample	Residue, mg/kg	
	Phosmet	Oxon
Unwashed raw apples (duplicate samples)	14, 12	<0.05, <0.05
Washed apples	15.5	0.090
Peeled sliced apples	1.05	<0.05
Canned sliced apples	0.32	<0.05
Canned apple sauce	0.95	<0.05
Ground apple slurry	11	<0.05
Unclarified apple juice	5.35	<0.05
Canned unclarified apple juice	5.4	<0.05
Filter cake	16	0.055
Canned clarified apple juice	1.4	<0.05
Wet pomace	29	0.072
Dry pomace	89, 88	0.25, 0.25
Dried apples	1.2	<0.05
Peels and cores	43	0.068

<u>Grapes</u>. Four applications of Imidan 50 WP, an average of 7 days apart, were made at a nominal rate of 3.4 kg phosmet/ha (three times the maximum label rate for grapes in western USA) and samples were taken 7 days after the final application (Dykeman, 1994d).

Grapes were processed into raisins and raisin waste by sun-drying in California, and into wet and dry pomace. Samples were analysed for phosmet and phosmet oxon. The results are shown in Table 15.

Table 15. Residues of phosmet and phosmet oxon in processed fractions of grapes.

Sample	Compound	Residue, Range, mg/kg	Mean residue, mg/kg	Mean total toxic residue, mg/kg
Grapes	Phosmet Phosmet oxon	3.8-4-3 0.05-0.07	4.1 0.06	4.2
Raisins	Phosmet Phosmet oxon	3.0-3.8 0.39-0.48	3.4 0.43	3.8
Raisin waste	Phosmet Phosmet oxon	41-45 5.2-6.0	42 5.5	48
Grapes (Engler)[1]	Phosmet Phosmet oxon	2.6-2.8 <0.05(3)	2.7 <0.05	2.7
Wet pomace	Phosmet Phosmet oxon	7.6-8.7 0.10-0.11	8.2 0.10	8.3
Dry pomace	Phosmet Phosmet oxon	15-17 0.11-0.13	16 0.12	15.9

Separate grape samples from the field test facility and stored at the pomace processor.

<u>Peaches</u>. In a field trial in the peach-growing region of Oregon, USA, one plot of peach trees was treated with ten applications of Imidan 50 WP at 3.36 kg ai /ha per application, a total of 33.6 kg ai/ha (about 3 times the recommended maximum). The last application was made 14 days before harvest according to the current US label. Whole unwashed raw peaches were shipped fresh and chilled to a pilot-scale food processor and converted, within two days, into the processed products and wastes: washed peaches, peach pits, first rinse water and peels, peeled raw peaches before canning, canned peaches and dried peaches.

Whole unwashed peaches and their processed products were stored frozen until analysis for phosmet and phosmet oxon with a limit of determination of 0.05 mg/kg for each compound. The results are shown in Table 16.

The residue of phosmet oxon was only 1-2% of that of phosmet. The mean recoveries of phosmet and phosmet oxon from samples fortified in the range 0.05-5.0 mg/kg were 89-101% (Meyers *et al.*, 1991b).

Table 16. Phosmet and phosmet oxon residues in products of peach processing.

Sample	Residue, mg/kg	
	Phosmet	Oxon
Unwashed raw peaches	13, 10	0.082, 0.072
Washed peaches	11	0.20
Peach pits (stones)	<0.05	<0.05
First water rinse and peels	<0.05	<0.05
Peeled raw peaches before canning	0.18	<0.05
Canned peaches	0.08	<0.05
Dried peaches	0.39, 0.30	<0.05, <0.05

In dried peaches the phosmet residue was 11 mg/kg following two field treatments with 2.8 kg ai /ha, and a PHI of 7 days (Report No. B-0180).

<u>Olives</u>. In supervised trials in Greece in 1965 and Spain in 1976, olives received five treatments at 1.25 or 2.5 g ai/l corresponding to about 1.7 and 3.3 times the maximum application allowed by current Spanish GAP.

The harvested olives were washed with hot water and pressed. Water was separated from the pressed oil by decantation. For deodorization and decolorization, steam was bubbled through the crude oil and volatiles were removed under vacuum, the exact process depending on the quality category. Export quality refined oil, obtained by several steam treatments and a vacuum treatment, usually contains <0.1% "acid".

Olives and crude olive oil analysed for phosmet residues from trials in Greece in 1965 gave the results shown in Table 17 (Batchelder, 1966).

Table 17. Phosmet residues in olives and crude olive oil (Greece, 1965).

Treatment, g ai/l	PHI, days	Phosmet residues, mg/kg	
		Olives	Greek olive oil
1.25	10	0.77	2.5
		0.44	1.5
		0.60	2.1
1.25	15	0.57	2.4
		0.25	1.4
		0.54	2.1

The olives from the Spanish trials were harvested 9, 16, 23 and 30 days after the last of five spray applications at 1.25 or 2.5 g ai/l. The crude oil contained the residues of phosmet and phosmet oxon shown in Table 18. The residues in the olives before processing were not reported.

Table 18. Phosmet residues in olive oil after spraying with “midan 5OW, 1976.

Application		PHI, days	Residues, mg/kg, in crude olive oil	
Rate, g ai/l	No.		Phosmet	Oxon
1.25	5	9	5.5	<0.1
		16	<0.4	<0.1
		23	7.6	1.0
		30	5.5	<0.1
2.5	5	9	7.6	<0.1
		16	10.0	0.4
		23	7.6	<0.1
		30	7.6	<0.1

Bulked samples of the oil from the 30-day PHI, together with other oils from olives treated at higher rates, were purified to obtain a "neutral" oil and analysed for phosmet residues with the results shown in Table 19 (Pereiro, 1978).

Table 19. Effect of "neutralizing" crude olive oil on residues of phosmet.

Rate, g ai/l	Phosmet, mg/kg	
	Not neutralized	Neutralized
2.5	6.3	<0.02
5.0	10.7	3.7
10	15.3	4.2

Potatoes. For a processing study on potatoes, Imidan 50 WP was applied to the foliage of growing potato plants four times at the maximum recommended rate of 1.12 kg ai/ha and once at four times that rate (4.5 kg ai/ha), with 11-13 days between applications. Potato tubers were harvested for analysis and processing 7 days after the final application. The trial took place in Washington State, USA, in 1993. Tubers were processed, simulating industrial operations as closely as possible, to yield potato chips, wet peel, dry peel and potato granules. Samples were analysed in triplicate. The residues of phosmet are shown in Table 20 (Dykeman, 1994f). The residues of phosmet oxon were below the limit of quantification (<0.05 mg/kg) in all the samples.

Table 20. Phosmet residues in fractions from processed potatoes.

Sample	Phosmet, mg/kg	Mean, mg/kg
Washed tubers	0.092 0.095 0.11	0.099
Chips	<0.05(3)	<0.05
Wet peel	0.10 0.094 0.087	0.095
Dry peel	0.36 0.30 0.31	0.323
Granules	<0.05(3)	<0.05

Oxon residues were all <0.05 mg/kg

Prunes. In a study in California in 1993 three replicate plots in a prune orchard were treated with “Imidan 50WP four times at the maximum recommended rate of 3.4 kg ai/ha, with an average of 14 days between applications.

Fresh prunes were processed to dried prunes and both commodities were analysed for phosmet and phosmet oxon. The residues of phosmet are shown in Table 21. Phosmet oxon was not detectable in any of the samples.

Table 21. Residues of phosmet in fresh and dried prunes.

Commodity	Phosmet, mg/kg	Mean, mg/kg
Fresh prunes	2.6 2.7 2.6	2.63
Dried prunes	0.72 0.82 0.92	0.82

Residues of phosmet oxon were in all cases <0.05 mg/kg

The lower residue in dried prunes is not surprising despite the loss of moisture since the prunes were dried at a temperature of 54-60°C for two days (typical commercial practice) which would be expected to contribute significantly to the breakdown of phosmet and phosmet oxon (Dykeman, 1994c).

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

Information on residues found in food in commerce was submitted by The Netherlands for the period 1991-94. The results are shown in Table 22. Residues found in kiwifruit during monitoring in 1975-78 were tabulated in the 1979 Evaluations (FAO/WHO, 1980) and are repeated in Table 23.

Table 22. Phosmet residues detected in the Dutch monitoring programme during 1991-94.

Sample	Sample analysed	Number of samples with residues		
		<LOD	<MRL	>MRL
Oranges	1306	1289	17	0
Tangerines	738	724	14	0
Apples	2410	2390	20	0
Pears	663	654	9	0
Peaches	113	111	1	1
Nectarines	103	100	2	1
Plums	148	147	1	0
Grapes	999	997	1	1
Kiwi	309	304	5	0

Table 23. Phosmet residues in kiwifruit in commerce (FAO/WHO, 1980).

Year	No. of samples (different growers)	Residues, mg/kg	
		Mean	Range
1975	20	3.8	0.3-10
1977	28	4.8	0.0-23
1978	9	2.2	0.0-9

NATIONAL MAXIMUM RESIDUES LIMITS

The following national MRLs have been reported to the Meeting.

Crop group	Commodity	Country	MRL	PHI
Citrus fruit	Citrus	Netherlands USA	5 5	- -
Pome fruit	Apples and pears	Australia Canada Netherlands	1 10 1	- - 21

Crop group	Commodity	Country	MRL	PHI
		Switzerland	1	15
		USA	10	7
Stone fruit	Stone fruit	Australia	1	-
	Stone fruit	New Zealand	10	14
	Apricots	USA	5	-
	Cherries	Canada	7	-
		USA	10	-
	Nectarines	USA	5	-
	Peaches	Canada	10	-
		USA	10	-
	Plums	Canada	5	-
		USA	5	-
Small fruits and berries	Blueberries	USA	10	
		Netherlands	10	3
	Grapes	Canada	10	-
		USA	10	-
Tropical and sub-tropical fruit	Kiwifruit	Australia	15	-
		Netherlands	15	-
		New Zealand	15	21
		USA	25	-
Tree nuts	Nuts	Netherlands	0.1	-
		USA	0.1	-
Legume vegetables	Peas, fresh	Netherlands	0.2	
Root and tuber vegetables	Sweet potatoes (post-harvest use)	USA	10	-
		Netherlands	10	-
	Potatoes			
		Netherlands	0.05*	28
		Switzerland	0.05*	-
		USA	0.1	-
Cereal grains	Fresh corn including sweet corn	USA	0.5	-
Tea		Netherlands	0.1*	
Fodder crops	Alfalfa	USA	40	-
	Field corn	USA	10	-
	Pea forage	USA	10	-
	Other commodities	Netherlands	0.05*	
Animal products	Meat and fat (cattle)	Australia	1	
		USA	0.2	-
	Milk	Australia	0.2	-
		Netherlands	0.05*	-

*At or about the limit of determination

APPRAISAL

Phosmet has been evaluated at several Joint Meetings between 1976 and 1988. MRLs were recommended for a number of commodities of plant and animal origin. Updated information on GAP, and reports of supervised trials and studies of processing, metabolism and the stability of residues in stored analytical samples have been made available for evaluation within the CCPR periodic review programme.

Phosmet is a broad-spectrum organophosphorus insecticide used to control a variety of insect and mite pests which attack pome, stone and citrus fruit. It is also used on field, pasture and forage crops. Phosmet is non-systemic and acts by contact and ingestion as a cholinesterase inhibitor. It is registered in a number of countries, mainly for protecting fruits and vegetables. The direct use of phosmet on livestock for the control of warble fly, ticks and lice of cattle, resulting in residues in animal commodities, was not reported to the Meeting.

Carbonyl-labelled [^{14}C]phosmet was used in studies of metabolism and environmental fate.

The absorption, distribution, metabolism and excretion of [^{14}C]phosmet has been studied in rats, goats and hens. The chemical is rapidly absorbed, distributed and excreted, predominantly in the urine, in all three species. Biotransformation also appeared to be similar in the species studied. Hydrolysis of the phosphorus-containing moiety to yield *N*-mercaptomethylphthalimide is followed by methylation and oxidation at the sulfur atom to give sulfoxides and sulfones. These metabolites, together with *N*-mercaptomethylphthalimide, are hydrolysed to generate a series of phthalamic acids and finally phthalic acid.

The principal metabolites in tissues and milk reflect a single metabolic sequence: hydrolytic displacement of the phosphorus-containing moiety to yield *N*-mercaptomethylphthalimide, followed by methylation and oxidation of the thiol group. Hydrolytic degradation via *N*-hydroxymethylphthalimide also occurred. These reactions generated a series of phthalimide derivatives, which were hydrolysed to the analogous phthalamic acids. Treatment of extracted samples with hydrazine solubilized more than half of the bound residues. Solubilized products of hydrazinolysis consisted mostly of phthalohydrazide. The results indicate that bound residues in tissues and milk contain the *N*-substituted phthalimide moiety, with little or no chemical modification. Residues of phosmet do not accumulate significantly in edible tissues or eggs. Although the rat liver microsomal NADPH enzyme system readily converts phosmet to phosmet oxon, neither phosmet nor its oxygen analogue could be detected in the tissues of the goats or hens.

Lactating goats were dosed with [^{14}C]phosmet at the equivalent of 8-8.8 ppm in the diet for four days. Most of each day's dose was recovered in the urine within the following 24 hours. In total, urinary excretion accounted for 60% of the cumulative dose. Less than 6% remained in the edible tissues at slaughter, 13-14 hours after the final dose. The total radioactivity ranged from 0.006 mg/kg phosmet equivalent in the fat to 0.24 mg/kg in the kidneys. Nine metabolites containing the phthalimide moiety were identified. Neither phosmet nor phosmet oxon was detected in the edible tissues (<0.002-0.003 mg/kg) or milk (<0.0004 mg/kg).

Laying hens dosed for seven days at a level equivalent to 10.5 ppm in the diet excreted 89.6% of the cumulative dose. Edible tissues collected at slaughter and eggs accounted for only 0.3% of the cumulative dose. In egg yolks the highest level of ^{14}C (as phosmet equivalents) was 0.040 mg/kg on day 7, and in whites 0.007 mg/kg on day 4. At slaughter the levels of total radioactivity expressed as phosmet were 0.24 mg/kg in liver, 0.21 mg/kg in kidneys, 0.021 mg/kg in breast muscle, 0.015 mg/kg in thigh muscle, 0.005 mg/kg in fat and 0.068 mg/kg in blood. Phosmet itself was not detected (<0.005 mg/kg) in any of the edible tissues, but 0.001 mg/kg was found in egg yolks. None of the metabolites exceeded 0.005 mg/kg in the edible tissues or eggs. The metabolites identified in the edible tissues and egg yolks were phthalimide and phthalic acid.

Plant metabolism studies on sour cherries, cotton, maize and potatoes were reported. Forty four per cent of the applied radioactivity was absorbed by sour cherries within 4 hours. The main surface residue was the parent compound, while 16 or 17 metabolites occurred in the fruit. Phthalic acid was the major metabolite and accounted for 17-21% of the total radioactivity. Several other metabolites accounting for a small fraction of the radioactivity were identified. These included phosmet oxon, phthalimide, and phthalamic acid derivatives. No benzoic acid or ring-hydroxylated products were detected. Related conjugates of *N*-glycosylphthalimide accounted for 27-32% of the total radioactivity, but phthalic acid accounted for 85-90% of the extractable radioactivity after acid hydrolysis.

In maize the major part of the total residue was present in the maize fodder (267 mg/kg expressed as phosmet equivalent) and forage (31 mg/kg). Cobs (5 mg/kg) and grain (3 mg/kg) contained much lower residues. The metabolism of phosmet in maize involves various hydroxylation (oxidation), hydrolysis and conjugation reactions, giving products that are distinctly more polar than phosmet. The pattern of metabolites was similar in all parts of the plant, but their ratios varied. The parent phosmet amounted to 53% of the total residue in fodder, with the oxon (1.2%) and derivatives of phthalimide and phthalic acid present in small amounts, whereas in the grain phthalic acid was the single identified residue

(61%) and the parent compound was not detectable. Most of the radiocarbon in the unidentified metabolites (32.7%) was accounted for as phthalic acid after acid hydrolysis.

In potatoes the foliage contained most of the residue (14-109 mg/kg), and translocation to tubers (1.4-2.1 mg/kg) was limited. Phthalic acid and phthalamic acid were the major metabolites. Phosmet, its oxygen analogue and hydroxylated phthalic acids were not observed in any of the extracts.

The environmental fate of phosmet was studied in soil and water. Degradation in soil was studied under aerobic followed by anaerobic conditions. Under anaerobic conditions the degradation continued, but at a slower rate. The main components of the residue, expressed as phosmet equivalent, found in aerobic soil were phosmet (36.6%), phosmet oxon (0.5%), *N*-methoxymethylphthalimide (5.68%), *N*-methylsulfinylmethylphthalimide (2.59%), *N*-hydroxymethylphthalimic acid (2.44%) and phthalimide (1.53%). In addition, 7 identified metabolites containing the phthalimide moiety (each <1%) and some unidentified intermediate products were also detected. Hydrolysis was shown to be an important factor in limiting the persistence of phosmet in soils, and the initial degradation products were metabolized by soil micro-organisms. After hydrolysis the aryl moiety, with or without a mercapto group depending on the point of cleavage, was further degraded through a variety of reactions including oxidation of the mercapto group to sulfonic acid, its methylation followed by oxidation to the sulfoxide, and imide bond cleavage. Ultimately, mineralization to carbon dioxide occurred. The products under aerobic and anaerobic conditions were largely the same.

Phosmet did not undergo significant photodegradation when exposed on thin layer plates of soil to natural sunlight for a period of 30 days.

Phosmet undergoes fairly rapid hydrolysis at ambient temperatures, with half-lives in water at 25°C of 7.5-9.7 days at pH 5, 9.4 hours at pH 7 and 5.5 minutes at pH 9. Degradation is enhanced by light.

The major hydrolysis products formed at pH 5 in the dark were *O,O*-dimethyl *O*-hydrogen phosphorodithioate (79.4 mol %), *O*-methyl *O,O*-dihydrogen phosphorodithioate, phthalamic acid, phthalimide, and phthalic acid. Following irradiation with a xenon lamp at pH 5, dimethyl hydrogen phosphate, (72.3 mol %), phosphoric acid, methyl dihydrogen phosphate, phthalimide, phthalamic acid and phthalic acid were detected. Other minor products were also detected but not identified.

Residues in rotational crops were studied in radishes, lettuce and wheat which were planted in the soil 30, 120 and 365 days after treatment with [*carbonyl*-^{14}C]phosmet at a rate equivalent to 5.6 kg ai/ha. The total radioactive residue taken up by the plants varied from about 2% to 64% depending on the plant and the time between soil treatment and harvest. Neither phosmet nor its oxygen analogue were detected in the plant extracts. The radioactive residue consisted of a number of polar metabolites, most of which were characterized by chemical and enzymatic hydrolysis as esters or conjugates of phthalic acid.

The current analytical methods for residues are based on extraction with acetone or ethyl acetate, clean-up on charcoal, silica gel or SX-3 gel columns, and gas-chromatographic determination. Phosmet and its oxon are determined simultaneously. Recoveries are above 70%. The typical limits of determination in plant materials, milk and animal tissues are 0.01-0.05 mg/kg. In most of the supervised trials the LOD reported was 0.05 mg/kg.

Storage stability studies showed that phosmet is stable at -20 ± 10°C in almonds, apples, soya beans, and wheat grain and straw for a minimum of 2½ years and in alfalfa, maize, oranges, peppers and potatoes for a minimum of 2 years.

Definition of the residue

Phosmet is the major residue component; the oxon is either not detected or is less than 10% of the parent compound in most cases. In addition, the other metabolites are water-soluble compounds without the phosphorodithioate group and are less toxic than the parent compound. The significant residue for both regulatory control and dietary intake purposes is therefore the parent compound.

The Meeting noted that phosmet was previously classified as fat-soluble. On the basis of its octanol/water partition coefficient and the distribution of residues between fat and meat, the Meeting concluded that the compound is not fat-soluble.

Definition of the residue for compliance with MRLs and for the estimation of dietary intake: phosmet

Supervised trials

Supervised trials were conducted on oranges in Argentina and Brazil. In the Argentine trials residues were determined in whole fruit, peel and pulp, but in Brazil only the pulp was analysed and the results cannot be used to estimate maximum residue levels. The application rate in the three Argentine orange trials corresponded with the current use pattern and resulted in residues in the whole fruits of 0.07, 0.13 and 0.32 mg/kg. The pulp did not contain detectable residues (<0.05 mg/kg) in any of the trials.

The data were too limited to estimate a maximum residue level for oranges, and since no residue data were provided for other citrus commodities, the Meeting recommended the withdrawal of the existing CXL for citrus fruits (5 mg/kg).

A number of trials were carried out on apples and pears in Brazil, Canada, Germany, The Netherlands, the UK and the USA. No GAP was reported for Germany, The Netherlands or the UK. Trials were according to current GAP in Canada (1.9 kg ai/ha) and the USA (1.7–4.1 kg ai/ha for apples; 1.7-5.6 kg ai/ha for pears) or at somewhat higher rates. The residues in the fruit were generally below 5 mg/kg at 7 days PHI. The residues in pears (1.7, 1.3 and 0.85 mg/kg) were lower than in apples. The Brazilian trials resulted in residues below 0.05 mg/kg in apples 14 days after application at single or double GAP rates. The residues in apples from the Canadian and US trials at approximately maximum GAP rates in rank order were 1.8, 1.8, 2.8, 3.3, 3.4, 3.4, 3.7, 4.2, 4.3 and 7.3 mg/kg.

The Meeting estimated a maximum residue level of 10 mg/kg, and an STMR level of 3.4 mg/kg for apples. Owing to the lack of sufficient data, the Meeting concluded that no maximum residue level could be estimated for pears and recommended the withdrawal of the existing CXL (10 mg/kg).

Field trials on apricots, nectarines and peaches treated at rates up to 1.3 times the US GAP rate resulted in residues up to 6.8 mg/kg at 14 days PHI. The residues in apricots (♦) and peaches treated at 0.7-1.3 times the maximum rates according to Canadian and US GAP in rank order were 0.87 1.2, 1.5, 1.6, 2.9, 4.2♦, 4.7♦, 6.4 and 6.8 mg/kg. The residues in nectarines were lower, 0.45 and 0.55 mg/kg, and could not be combined with those of apricots and peaches.

The Meeting estimated maximum residue levels of 10 mg/kg and STMR levels of 2.9 mg/kg for apricots and peaches, and recommended the withdrawal of the existing CXL for nectarines (5 mg/kg).

Following treatments at about 1-1.3 times current GAP rates, residues in plums of 0.41, 0.55 and 0.48 mg/kg, and in fresh and dried prunes of 2.3 and 2.2 mg/kg were reported. The information was insufficient to estimate a maximum residue level for plums (including prunes).

Grapes were treated at rates of 1.4-2.2 kg ai/ha which accord with GAP for the eastern states of the USA (1.5-2.5 kg ai/ha). Residues up to about 10.2 mg/kg were found 7 days after the last application and up to 9.2 mg/kg after 14 days. The residues from treatments according to GAP in rank order were 0.17, 0.24, 0.61, 2.8, 3.3, 4.0, 4.2 and 9.2 mg/kg.

The Meeting estimated a maximum residue level of 10 mg/kg and an STMR of 3.1 mg/kg for grapes.

In supervised trials on olives in France, Italy and Spain the residues declined to <0.02-0.34 mg/kg after PHIs of 28-30 days. The trials in France were evaluated against Spanish and Italian GAP. The residues from GAP applications in rank order were <0.02, 0.09, 0.12, 0.16, 0.24 and 0.34 mg/kg.

The available information indicates that a maximum residue level of 0.5 mg/kg and an STMR of 0.14 mg/kg for olives would be appropriate, but because there was no suitable supporting processing study the Meeting could not make any recommendation.

Of the supervised residue trials on kiwifruit carried out in New Zealand during 1974-76 only one complied with current GAP. The Meeting recommended the withdrawal of the CXL for kiwifruit (15 mg/kg).

In two supervised trials on peas carried out in two states of the USA, phosmet residues were below the limit of determination (<0.05 mg/kg) in succulent peas, <0.05-0.08 mg/kg in dried peas, 0.15-0.51 mg/kg in succulent pods, 2.7-5.6 mg/kg in succulent pea forage and 2.5-17 mg/kg in dry pea hay. Phosmet oxon residues were <0.05 mg/kg in peas and green forage, and 0.06-0.28 mg/kg in hay. The oxon residue was less than 10% of that of the parent compound.

The Meeting concluded that the data were not sufficient to estimate maximum residue levels, and recommended the withdrawal of the existing CXLs for peas (pods and immature seeds), peas (dry), pea hay or fodder (dry) and pea vines (green).

Numerous trials on potatoes in Canada, The Netherlands and the USA indicated that the translocation of the compound to the tuber was limited, and residues in the tubers following applications at recommended and double rates were <0.05 mg/kg. Residues up to 0.11 mg/kg were detected in trials at fivefold rates however, which indicates that this is not a nil residue situation.

The Meeting estimated a maximum residue level of 0.05* mg/kg and an STMR of 0.05 mg/kg for potatoes. This is the level of the current CXL.

Residues from six supervised trials on cotton in Brazil at 1.5-4.5 times the GAP rate were all below the limit of determination (0.05 mg/kg).

The Meeting concluded that no detectable residue is likely to occur in cotton seed if GAP is followed, and estimated a maximum residue level of 0.05 mg/kg and an STMR level of 0 mg/kg.

Supervised trials were reported on alfalfa, Bermuda grass, lupins, maize forage, peas, rape and soya bean plants used for animal feed. Most of the trials were on alfalfa.

The residue data on forage and fodder crops showed that residues were generally high (commonly 40-80 mg/kg) immediately after application to alfalfa, but declined fairly rapidly. After 14 days they were mainly in the range 0.2-2 mg/kg. The residues of phosmet on lupins, maize, peas and rape were lower and generally below 2.0 mg/kg 7 days after the last application. The residues in fresh alfalfa from applications according to GAP in rank order were 0.13, 0.21, 0.24, 0.26, 0.3, 0.4, 0.77, 0.84, 0.84, 1.2, 1.6, 2.1, 2.1, 2.24 and 3.5 mg/kg. The Meeting did not estimate any maximum residue levels for animal feed items (see "Animal products" below).

The data, if any, were insufficient to estimate maximum residue levels in blueberries, feijoa, maize, maize fodder and forage, pea hay or fodder, sweet corn, sweet potatoes and tree nuts. The Meeting therefore recommended the withdrawal of the existing CXLs for these commodities.

Animal products. Although no detectable residues of phosmet or its oxon occurred in edible animal products in metabolism studies, the Meeting was not able to estimate any maximum residue levels for animal feeds or animal products because of the high residues in animal feed items and the lack of animal transfer studies. Consequently, the Meeting recommended the withdrawal of the existing CXLs for alfalfa fodder and forage, cattle meat and milks.

Processing

Studies have been carried out to determine the effect of processing on residues of phosmet in apples, grapes, peaches, olives, potatoes and prunes.

Field-treated apples containing 12-14 mg/kg phosmet residues were processed to unclarified and clarified juice and wet and dry pomace, which contained 5.3, 1.4, 29 and 89 mg/kg respectively. The oxon residue was less than 1% of the phosmet residue in all samples. Most of the phosmet residue is evidently in or on the peel, since processing decreased residues about 2.5-10 times in the products which were separated from the peel. Fractions which are normally processed with the peel, such as wet and dry pomace and the combined peels and cores, showed about a 2-6-fold concentration of the residues. The Meeting therefore concluded that maximum residues up to 60 mg/kg might occur in dry apple pomace.

Field-treated grapes were processed to raisins and raisin waste by sun-drying, and into wet and dry pomace. There was no concentration of the residue in the raisins but concentration occurred by factors of 12 in raisin waste, 3 in wet pomace, and about 6 in dry pomace.

Potatoes, treated with excessive amounts of phosmet to obtain detectable residues (0.1 mg/kg), were processed to yield potato chips, potato granules, wet peel and dry peel. There was no detectable residue in potato chips or granules (LOD ≤0.05 mg/kg). Residues in the wet peel were at the same level as in the washed potatoes, but were concentrated about threefold in the dry peel. This was accounted for by an 85% loss of moisture partly offset by the loss of some phosmet during drying (the theoretical residue would be 0.72 mg/kg).

Olives were processed to crude oil and neutralized oil. The residue in the crude oil was about four times that in the original olives, and purification ("neutralization") of the crude oil reduced the residues about threefold. The process used for neutralization was not reported, so the residues in the oil could not be used to estimate those likely to result from industrial processing. The Meeting concluded that the database was not sufficient to estimate maximum residue levels in crude or refined olive oil.

Fresh prunes were processed into dried prunes and both commodities were analysed for phosmet and phosmet oxon. The average phosmet residue in fresh prunes was 2.63 mg/kg, and in dried prunes 0.82 mg/kg. Phosmet oxon was not detectable in any of the samples. The decrease in dried prunes was attributed to the loss of residues during the drying process at 54-60°C, which more than offset the loss of moisture. Since it had not been possible to estimate a maximum residue level for fresh prunes the Meeting could not estimate one for dried prunes.

RECOMMENDATIONS

The unchanged parent compound is the major residue component, phosmet oxon is either not detected or it is present in less than 10% of the parent compound in most cases. In addition, the metabolites are less toxic than the parent compound and they are water soluble compounds without the phosphorodithioate group. Therefore the significant residue for both regulatory control and dietary intake purposes is defined as the parent compound alone. The Meeting noted that the compound was previously classified as fat soluble. Based on its octanol/water partition coefficient and residue distribution in fat and meat, the Meeting concluded that the compound is not fat soluble.

Definition of residue for compliance with MRLs and for estimation of dietary intake: phosmet

Commodity		Recommendation			PHI,[1] days
		MRL mg/kg		STMR mg/kg	
CCN	Name	New	Previous		
AL 1021	Alfalfa forage green	w	40		
FP 0226	Apple	10	10	3.4	7
FS 0240	Apricot	10	5	2.9	7
FB 0020	Blueberries	w	10		
MM 0812	Cattle meat	w	1 (fat) V		
FC 0001	Citrus fruits	w	5		
SO 0691	Cotton seed	0.05		0	14
FI 0335	Feijoa	w	2		
FB 0269	Grapes	10	10	3.1	7-14
FI 0341	Kiwi	w	15		
AF 0645	Maize forage	w	10		
AS 0645	Maize fodder	w	10		
GC 0645	Maize	w	0.05		
ML 0106	Milks	w	0.02 V		
FS 0245	Nectarine	w	5		
AL 0072	Pea hay and fodder	w	10		
AL 0528	Pea vines green	w	10		
FS 0247	Peach	10	10	2.9	14
FP 0230	Pear	w	10	w	
VP 0063	Peas (pods and succulent seeds)	w	0.2		
VD 0063	Peas (dry)	w	0.02*		
VR 0589	Potato	0.05*	0.05	0.05	7
VO 0447	Sweet corn (corn-on-the-cobs)	w	0.05		
VR 0508	Sweet potato	w	10 (Po)		
TN 0085	Tree nuts	w	0.1		

[1]PHI on which the recommendations are based

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TEBUCONAZOLE (189)

EXPLANATION

Tebuconazole is a triazole fungicide used as a seed dressing and spray. It was reviewed for the first time in 1994. Maximum residue levels were estimated for a number of commodities of plant and animal origin. New information on formulations, analytical methods and registered uses and data from additional supervised trials and processing studies are reviewed below.

Formulations

Table 1 shows the main types of formulation registered for use internationally. EW = emulsion, oil in water; EC = emulsifiable concentrate; FS = flowable concentrate for seed treatment; DS = powder for dry seed treatment; SC = suspension or flowable concentrate; WG = water-dispersible granule; WP = wettable powder.

Table 1. Formulations of tebuconazole.

Form.	Active ingredient(s)	Concentration	Form.	Active ingredient(s)	Concentration
1.5 DS	tebuconazole	1.5 %	29 FS	tebuconazole triflumuron	25 g/l 4 g/l
2 DS	tebuconazole	2 %	29 FS	tebuconazole cypermethrin	25 g/l 4 g/l
2.5 DS	tebuconazole	2.5 %	29 DS	tebuconazole triflumuron	% 0.4 %
015 ES	tebuconazole	15 g/l	29 DS	tebuconazole cypermethrin	% 0.4 %
2 WS	tebuconazole	2 %	29 DS	tebuconazole triflumuron	% 0.4 %
0.26 FS	tebuconazole	26 g/l	3.6 F	tebuconazole	38.7 %
2.6 FS	tebuconazole	260 g/l	250 EC/ 25 EC	tebuconazole	250 g/l
060 FS	tebuconazole	60 g/l	25 WP	tebuconazole	25 %
025 FS/ 2.5 FS	tebuconazole	25 g/l	250 EW/25 EW	tebuconazole	250 g/l
040 FS	tebuconazole triazodine	20 g/l 20 g/l	430 SC	tebuconazole	432 g/l
61.9 WS	tebuconazole captane anthraquinone	% % 22.5 %	375 FS	tebuconazole imidacloprid triazoxide	15 g/l 350 g/l 10 g/l
5 WS	tebuconazole imazalil	2 % 3 %	50 WG	tebuconazole dichlofluanid	10 % 40 %
515 FS	tebuconazole thiram	15 g/l 500 g/l	50 WG/ 50 WP	tebuconazole tolylfluanid	10 % 40 %
035 ES	tebuconazole imazalil	15 g/l 20 g/l	225 EC	tebuconazole triadimefon	125 g/l 100 g/l
315 FS	tebuconazole guazatine	15 g/l 300 g/l	375 EC	tebuconazole triadimenol	250 g/l 125 g/l
500 EC	tebuconazole fenpropidin	200 g/l 300 g/l	400 EC	tebuconazole prochloraz	133 g/l 267 g/l
500 EC	tebuconazole propiconazole	250 g/l 250 g/l	030 FS	tebuconazole fludioxonil	5 g/l 25 g/l

Form.	Active ingredient(s)	Concentration	Form.	Active ingredient(s)	Concentration
060 FS	tebuconazole fludioxonil cyprodinil	10 g/l 25 g/l 25 g/l	050 FS	tebuconazole fludioxonil difenoconazole	5 g/l 25 g/l 20 g/l
45 DF	tebuconazole	45 %	45 WG	tebuconazole	45 %
250 EW	tebuconazole	250 g/l	25 WG	tebuconazole	25 %
25 EW	tebuconazole	250 g/l	65 WP	tebuconazole tolylfluanid	15 % 50 %
290 EC	tebuconazole tridemorph	125 g/l 165 g/l	300 SC	tebuconazole carbendazim	167 g/l 133 g/l

METHODS OF RESIDUE ANALYSIS

Tebuconazole is determined in plant material and soil by gas chromatography (GLC) after extraction with organic solvents and clean-up on columns of various materials.

Brennecke (1991) developed a method (No. 00249) for the determination of tebuconazole, dichlofluanid and tolylfluanid in plant material. The sample is cleaned up by a laboratory robot system using liquid-solid extraction on diatomaceous earth and column chromatography on silica gel, after the active ingredients are extracted with acetone or dichloromethane. Aqueous samples such as beverages are transferred directly to the robot. Quantification is by GLC with a thermionic nitrogen/phosphorus detector (NPD). The recoveries from untreated control samples of paprika, peaches, peach juice, tomatoes, grapes, must, wine and zuccini fortified with 0.02 to 5.0 mg/kg of tebuconazole were 80 to 109%. The LOD (limit of determination) was 0.02 mg/kg in all commodities.

Maasfeld and Minor (1992) revised Method No. 0007 (Maasfeld, 1987) reported in 1994 and developed a special extraction procedure for the determination of tebuconazole in peanuts and their processed products. Tebuconazole is extracted from crude and refined peanut oil with hexane and partitioned into acetonitrile. Soapstock samples are extracted with ethyl acetate and partitioned sequentially against N HCl and hexane/acetonitrile. All extracts are cleaned up on gel permeation and silica gel columns and the residues are determined by GLC with an NPD. The recoveries from untreated control samples fortified at 0.05 mg/kg were 82-94% with LODs for peanuts, oil and soapstock of 0.01-0.05 mg/kg.

A method (No. F60) originally developed for the determination of fuberidazole, fluotrimazole and triadimefon in plant material and soil samples (Specht, 1977) can also be used for the determination of tebuconazole. The sample is extracted with acetone/water and the compound partitioned into dichloromethane. After clean-up on a Florisil column the residue is determined by GLC with an NPD. The LOD was 0.05 mg/kg with a mean recovery of 87.4%.

Analytical methods for the determination of tebuconazole in plums and garlic were described by Mestres *et al*. (1995) and Mestres and Reulet (1996a,b) respectively. Samples were extracted with dichloromethane/ethyl acetate, the extract was concentrated and the residues dissolved in ethyl acetate. There was no further clean-up. Tebuconazole was again determined by gas chromatography with an NP thermionic detector. Recoveries after fortification of control plum samples with 0.04 and 0.45 mg/kg were 99% and 97%, and the limit of determination was 0.02 mg/kg. Recoveries from untreated garlic samples fortified with 0.013-0.4 mg/kg were 91-110% and the LOD was 0.015 mg/kg.

An analytical method for the determination of tebuconazole in onions was described by Delgado (1991). Samples were extracted with acetone and purified by partitioning with cyclohexane/ethyl acetate. After evaporation, the residue was dissolved in toluene and determined by gas chromatography with a nitrogen phosphorus detector. The recoveries from control samples fortified with 0.02 and 0.38 mg/kg were 108% and 106%, and the LOD was 0.02 mg/kg.

USE PATTERN

Table 2 shows the registered uses of tebuconazole on the crops for which trials are reviewed in this evaluation as of February 1997. The list has been largely extended since 1994 for bananas, peaches and pears.

Table 2. Registered uses of tebuconazole. Ai = active ingredient; F = field; G = greenhouse; - = not stated; N.A. = not applicable. Application is by foliar spray unless otherwise indicated.

Crop	Country	Product	Application				PHI, days
			No.	Max. rate, kg ai/ha	kg ai/hl	F/G	
Apples	Brazil	25 WP	1-4	0.09-0.15	0.0075-0.013	F	20
	France	25 WG	1-4	0.03-0.11	0.0075	F	21
	Indonesia	25WP	8	0.125-0.25	-	F	10
	Israel	25WP 50 WP	1-3 1-2	0.05 0.05	- -	F	21 21
	Italy	25 WG	1-4	0.28	0.0186	F	30
	Spain	25 WG	4-6	0.1-0.15	0.01-0.015	F	21
	Turkey	25 WP	1-2	0.09-0.13	0.0062	F	14
Bananas[1]	Australia	430 SC	5-6	0.1	0.1-0.5	F	1
	Cameroon	250 EW	1-3	0.1	-	F	0
	Colombia	250 EW	6-8	0.1	0.71-0.83	F	0
	Costa Rica	250 EW	4-8	0.1	0.53-0.83	F	-
	Ecuador	250 EW	6-8	0.1	0.71-0.83	F	0
	Guatemala	250 EW/EC	2-8	0.1	0.33-1	F	0
	Honduras	250 EW	2-8	0.1	0.33-1	F	0
	Indonesia	250 EC	1-4	0.05-0.1	0.01-0.02	F	10
	Ivory Coast	250 EW	1-6	0.1	-	F	-
	Nicaragua	250 EC/EW	2-8	0.1	0.33-1	F	0
	Philippines	250 EC	6-8	0.075-0.125	0.25-0.42	F	-
	USA	45 WG	6.7 [2]	0.1	0.13	F	0
Barley [3] (seed treatment)	Australia	2.9 DS/ 29 FS	1	0.0025	-	F	N.A.
	Belgium	040 FS	1	0.003	-	F	N.A.
	Chile	2WS/515FS	1	0.003/0.0022-0.003	-	F	N.A.
	France	375 FS	1	0.003	-	F	N.A.
	Germany	040 FS	1	0.002	-	F	N.A.
	Great Britain (UK)	040 FS	1	0.003	-	F	N.A.
	Ireland	025 FS	1	0.003	-	F	N.A.
	Italy	035 ES/515FS	1	0.003	-	F	N.A.
	South Africa	025 FS/015ES	1	0.0025/0.0026	-	F	N.A.
	Spain	025 FS	1	0.003-0.004	-	F	N.A.
	USA	0.26FS/2.6 FS	1	0.002	-	F	N.A.
Cherries	USA	45 WG	1-6	0.25	0.0067	F	0
Cucumber	Chile	250 EW	1-2	0.125 -0.375	0.0125-0.025	F	14
		375 EC	1-2	0.083 -0.125	-	F	35
	Israel	50 WP	1-3	0.15	0.015	F or G	14
	Spain	50 WP	1-3	0.2-0.3	0.02-0.03	F or G	7
Garlic	Brazil	25 WP	1-4	0.25	0.025-0.05	F	14
	Israel	250 EC	1-3	0.19	-	F	21

Crop	Country	Product	Application				PHI, days
			No.	Max. rate, kg ai/ha	kg ai/hl	F/G	
	Spain	250 EW	1	0.5 (soil drench)	-	F	-
Grapes	Brazil	25 WP	4-7	0.25-0.38	0.025	F	14
	Chile	250 EW	1-2	0.31-0.44	-	F	14
	France	250 EW	1 -3	0.075-0.1	0.011-0.075	F	14
	Germany	50 WP	3-4	0.15-0.5	0.025	F	35
	Israel	250 EC 50 WP	1-3	0.05 0.15	0.05 0.015	F	21/14
	Italy	25 WG	1-4	0.1-0.38	0.01-0.038	F	50
	South Africa	375 EC	1-8	0.013-0.09	0.05-0.036	F	14/35[4]
		300 SC	1-2	0.063-0.19	-	F	28
	Spain	250 EW 50 WP	1-3 1-3	0.063-0.13 0.25-0.38	- -	F F	21 21
Oats [3] (seed treatment)	Australia	2.9 DS/29 FS	1	0.0025	-	F	N.A.
	Chile	515 FS	1	0.0022-0.003	-	F	N.A.
	Germany	040 FS	1	0.003	-	F	N.A.
	Ireland	025 FS	1	0.003	-	F	N.A.
	Italy	035ES/515 FS	1	0.003	-	F	N.A.
	Spain	025 FS	1	0.003	-	F	N.A.
	USA	0.26FS/2.6 FS	1	0.002	-	F	N.A.
Onion	Brazil	25 WP	1-4	0.25	0.025-0.05	F	14
	Israel	250 EC	1-2	0.19	-	F	21
	New Zealand	250EW/430SC	2-3	0.38	0.038	F	35
	South Africa	250 EW	1-6	0.19	0.038	F	-
	Spain	250 EW	1	0.5 (soil drench)	-	F	-
Peaches and nectarines	Chile	250 EC/EW	1	0.38-0.53	0.031-0.044	F	35
	France	25 WG	1-3	0.063-0.15	0.013	F	7
	Italy	25 WG	1-2	0.15-0.3	0.013-0.025	F	15
	Peru	250 EW	1-3	0.125	0.0125	F	21
	USA	45 WG	1-6	0.25	0.0067	F	0
Peanuts	Australia	430 SC	1-5	0.075 -0.189	0.075-0.189	F	21
	Argentine	250 EC	3-5	0.13	0.052-0.087	F	35
	Brazil	25 WP	2-3	0.13	0.042-0.063	F	30
	Guatemala	250 EW	2-3	0.13- 0.19	-	F	21
	Indonesia	250 EC	1-6	0.13- 0.25	0.025-0.05	F	10
	Israel	250 EC	1-3	0.25	-	F	21
	Nicaragua	250 EW	2-3	0.13- 0.19	-	F	21
	South Africa	250 EW	1-5	0.1-0.15	0.02-0.42	F	14
	USA	3.6 F	1-4	0.23	0.25	F	14
Pears	Israel	25 WP	1-3	0.05	-	F	21
	Italy	25 WG	1-4	0.15-0.28	0.01-0.19	F	15
	Spain	25 WG	4-6	0.1-0.15	0.01-0.015	F	21
	Turkey	25 WP	1-2	0.09-0.13	0.0062	F	14
Plums	Israel	250 EC	1-3	0.05	-	F	21
Sweet peppers	Spain	50 WP	1-3	0.2 -0.3	-	F or G	7
Wheat [3] (seed treatment)	Argentina	2 WS	1	0.0025	0.167-0.125	F	N.A.
	Australia	2.9 DS/29DS	1	0.0025	-	F	N.A.
	Chile	2 WS/515FS	1	0.003/0.0022-0.003	-	F	N.A.
	Ireland	025 FS	1	0.003	-	F	N.A.
	Italy	035 ES/515 FS	1	0.003	-	F	N.A.
	South Africa	025 FS/015 ES	1	0.0012/0.0013	-	F	N.A.
	Spain	025 FS	1	0.003-0.004	-	F	N.A.
	USA	0.26FS/2.6FS	1	0.002	-	F	N.A.

[1]Numbers of treatments are numbers/year. Numbers per application cycle are restricted to a maximum of four
[2]Average no./year

[3]Application rate, kg ai/100 kg
[4]14 days for table grapes, 35 days for wine grapes

RESIDUES RESULTING FROM SUPERVISED TRIALS

In 1994 residue data were submitted on pome fruit (apples and pears), stone fruit (apricots and peaches), grapes, bananas, onions, beans, peas, cucumbers, summer squash, egg plants, sweet peppers, tomatoes, potatoes, cereal grains (barley, maize, oats, rice, rye and wheat), peanuts, and rape. Because trials data were insufficient and/or information on GAP was lacking, MRLs were recommended only for grapes, summer squash (zucchini), tomatoes, barley, rye, wheat, peanuts and rape seed.

The data from new residue trials are discussed by crop group and summarized in Tables 3 to 17. Trials with the same entry in the Tables were carried at the same site. Unless otherwise indicated, all trials were with foliar sprays. Underlined residues are from trials according to GAP (±30%) and were used to estimate maximum residue levels. Double-underlined residues are from maximum treatments allowed by GAP and were used to estimate STMR levels.

Pome fruits

Apples (Table 3). Four trials were conducted in Brazil within or above the recommended rate (1-4 applications of 0.09-0.15 kg ai/ha) giving residues from below the LOD (0.1 mg/kg) to 0.5 mg/kg at a PHI of 20 or 21 days.

In two trials in Canada with six applications of 0.23 kg ai/ha the residues after 86 days were below the LOD (0.01 mg/kg) and 0.02 mg/kg.

In two trials in France at 1.5 and twice the recommended GAP the residues were 0.09 and 0.06 mg/kg after 21 (GAP) and 28 days respectively. In Germany, seven trials according to the proposed use (1-4 applications of 0.1-0.15 kg ai/ha) and four trials at lower rates gave residues at a PHI of 56 days from below the LOD (0.02 mg/kg) to 0.04 mg/kg. In ten trials in Korea above the recommended rate (1-3 applications of 1.0 kg ai/ha) the residues at a PHI of 21 days varied from 0.04-0.14 mg/kg.

In two trials in Italy and one in Spain according to GAP the residues were 0.12-0.18 mg/kg at a PHI of 28 or 21 days.

In sixteen trials in the USA at a nominal rate according to the proposed use (0.13-0.25 kg ai/ha) residues after intervals from the GAP PHI of 75 days to 129 days were below the limit of determination (0.01 mg/kg) except in one trial at an actual rate of 0.44 kg ai/ha where 0.02 mg/kg was found. In two other trials with half and twofold application rates the residues were <0.01 mg/kg.

Table 3. Residues of tebuconazole in apples. Whole fruit analysed.

Country Report No. (year) States	Application				PHI, days	Residues, mg/kg	Trial Reference
	Product	No	kg ai/ha	kg ai/hl			
Brazil (1990)	25 WP	8	0.075	0.0075	0 3 7 14 21	0.1/ 0.1/ <0.1/ <0.1 <0.1	BRA-118154-A/

Country Report No. (year) States	Application				PHI, days	Residues, mg/kg	Trial Reference
	Product	No	kg ai/ha	kg ai/hl			
Brazil (1990)	25 WP	8	0.15	0.015	14 21	<0.1 <0.1	BRA-118154-B
(1992)		4	0.15	0.015	20	0.2	BRA-138194-B
(1992)		4	0.3	0.03	20	0.5	BRA-138194-B
Canada (1989)	45 WG	6	0.23	0.0068	86	0.02	510-FR046-89D
Canada (1989)	45 WG	6	0.23	0.0068	86	0.01	510-FR047-89D
		3	0.15	0.015	0 28	0.10 0.06	0282-93
Germany RA-2972/94	50 WG	4	0.15	0.01	0 56	0.23 <0.02	0458-94
(1994)		4	0.15	0.01	0 56	0.39 <0.04	0459-94
		4	0.15-0.17	0.05	0 56	0.05 <0.02	0457-94
RA-2069/93 (1993)	50 WP	4	0.01	0.0008	0 14 28 56 65/67	0.27/0.22 0.07/0.03 0.02/0.02 <0.02/<0.02 <0.02/<0.02	0047-93/ 0130-93
		4	0.01	0.0008	0 14 28 56 65/67	0.25/0.15 0.06/0.04 0.04/0.03 0.02/<0.02 0.02/	0131-93/ 0132-93
RA-2001/94 (1994)		4	0.15-0.16	0.01-0.05	0 14 28 56	0.06 0.02 <0.02 <0.02	0001-94
		4	0.15-0.16	0.01-0.05	0 14 28 56 63	0.19 0.04 0.02 <0.02 <0.02	0002-94
		4	0.15-0.16	0.01-0.05	0 14 28 56 63	0.07 <0.02 <0.02 <0.02 <0.02	0003-94
		4	0.15-0.16	0.01-0.05	0 14 28 56 63	0.21 0.08 0.07 0.03 0.02	0004-94
Italy RA-2062/93 (1993)	25 WG	4	0.25	0.017	0 7 10 14 21 28	0.23 0.14 0.10 0.14 0.17 0.12	0031-93
		4	0.25	0.017	0 7 10 14 21 28	0.53 0.51 0.40 0.39 0.22 0.13	0284-93
Korea[1] (1992)	25 WP	3	1.25	0.025	14 21 28	0.15 0.04 0.04	KOR-R2104-93 KOR-R2103-93 KOR-R2102-93

Country Report No. (year) States	Application				PHI, days	Residues, mg/kg	Trial Reference
	Product	No	kg ai/ha	kg ai/hl			
					35	0.04	KOR-R2101-93
		4	1.25	0.025	21 28	0.08 0.05	KOR-R2106-93 KOR-R2105-93
		5	1.25	0.025	21 28	0.14 0.07	KOR-R2108-93 KOR-R2107-93
		6	1.25	0.025	21 28	0.13 0.12	KOR-R2110-93 KOR-R2109-93
Spain (1994)	25 WG	6	0.11	0.01	0 7 14 21 28	0.41 0.35 0.24 <u>0.18</u> 0.16	0367-94
USA[2] BR106219	45 DF	6	0.16	0.0067	129	<0.01	FCA-FR100-91H
(1992) CA,		6	0.16	0.034	75	<0.01	HIN-FR071-91H
IN, MI , NY		6	0.25	0.0067	106	<0.01	454-FR067-91H
PA, VA ,		6	0.25	0.0067	102	<0.01	454-FR099-91H
and WA		6	0.25	0.055	127	<0.01	455-FR068-91H
		6	0.25	0.11	77	<0.01	757-FR069-91D
		6	0.21	0.0055	119	<0.01	757-FR102-91D
		6	0.25	0.013	104	<0.01	758-FR070-91D[3]
		6	0.19	0.040	103	<0.01	855-FR101-91D
MR100067	45 DF	6	0.12	0.019	84	<0.01	HIN-FR014-89D
(1990)		6	0.055	0.0067	125	<0.01	STF-FR015-89D
IN, KS, MI,		6	0.13	0.016	115	<0.01	751-FR010-89D
NC, NY, PA,		6	0.15	0.0067	92	<0.01	757-FR011-89D
and WA		6	0.19	0.020	92	<0.01	758-FR012-89D
		6	0.19	0.0067	109	<0.01	855-FR013-89D
		6	0.027	0.0067	104	<0.01	454-FR007-89D[4]
		6	0.44	0.0067	104	0.02	454-FR008-89D
MR100066 (1990) KS	45 DF	6	0.50	0.034	125	<0.01	STF-FR017-89D

[1]All trials were at the same site, with one PHI/trial
[2]0.25 kg ai/kg nominal rate. Actual rates differed owing to different tree sizes
[3] 0.50 kg ai/kg nominal rate
[4] <0.12 kg ai/kg nominal rate

<u>Pears (Table 4)</u>. In a trial in Spain according to GAP (4-6 x 0.1-0.15 kg ai/ha, 21-day PHI) the residue at 21 days was 0.09 mg/kg. Four trials in the USA according to the proposed use (0.13-0.25 kg ai/ha nominal rate) yielded residues after 74 to 106 days from below the LOD (0.01 mg/kg) to 0.03 mg/kg. Two other trials at a nominal rate <0.09 kg ai/ha (actual rates 0.024 and 0.075 kg ai/ha) showed similar results.

Table 4. Residues of tebuconazole in pears. Whole fruit analysed.

Country Report No. (year) States	Application				PHI, days	Residues, mg/kg	Trial Reference
	Product	No	kg ai/ha	kg ai/hl			
Spain (1994)	25 WG	6	0.1-0.14	0.01	0 7 14 21 28	0.33 0.27 0.15 <u>0.09</u> 0.04	0368-94
USA[1]	45 DF	6	0.20	0.0068	106	<0.01	451-FR018-89D
MR100069		6	0.024	0.0068	84	<0.01	454-FR019-89D[2]

Country Report No. (year) States	Application				PHI, days	Residues, mg/kg	Trial Reference
	Product	No	kg ai/ha	kg ai/hl			
(1989) CA, MI, NY, OR and WA		6	0.075	0.0033	63	0.02	457-FR021-89D[2]
		6	0.20	0.022	104	<0.01	758-FR022-89D
		6	0.19	0.0067	74	0.01	855-FR023-89D
		6	0.086	0.0068	83	0.03	455-FR020-89D

[1]0.19 kg ai/kg nominal rate. Actual rates differed owing to different tree sizes
[2] <0.09 kg ai/kg nominal rate

Stone Fruits

Cherries (Table 5). Five trials were conducted in Italy at or below the proposed use rate (1- 2 applications of 0.28 kg ai/ha). At the proposed PHI of 7 days the residues were 0.18 and 0.20 mg/kg in the fruit without stone and 0.29 and 0.33 mg/kg in the whole fruit. In another trial the residues after 5 days were 0.50 and 0.40 mg/kg in the fruit without stone and whole fruit respectively.

Twelve trials in the USA were slightly below the GAP nominal rate (1-6 applications of 0.25 kg ai/ha) with one trial at a lower rate. Residues in the whole fruit at a PHI of 0 days were between 0.09 and 3.1 mg/kg.

Table 5. Residues of tebuconazole in cherries.

Country Report No. (year) States	Application				PHI, days	Sample[1]	Residues, mg/kg	Trial Reference
	Product	No	kg ai/ha	kg ai/hl				
Italy RA-2019/92 (1992)	25 WG	2	0.19	0.019	0 5 10 14	fruit/ whole fruit	0.59/0.48 0.50/0.40 0.29/0.25 0.26/0.22	0289-92
RA2067/93 (1993)		2	0.19	0.019	0 3 7 10	fruit	0.30 0.23 0.18 0.16	0029-93
		2	0.19	0.019	0 7	fruit	0.26 0.20	0290-93
RA-2075/96 (1996)		2	0.28	0.019	0 3 7 10	whole fruit	0.62 0.35 0.29 0.30	0592-96
		2	0.28	0.019	0 3 7 10	whole fruit	0.67 0.30 0.33 0.22	0594-96
USA[2] MR99826 (1989)	45 DF	6	0.19	0.020	0 3 7 10/14	whole fruit	0.41 0.60 0.25 0.17	451-FR024-89D
CA, MI, NY, OR and WA		6	0.19	0.020	0 3 7 10/14	whole fruit	0.61 0.40 0.39 0.07	855-FR029-89D
		7	0.19	0.0067	0 3 7 14	whole fruit	3.1 2.0 1.2 1.2	454-FR025-89D
		6	0.056	0.003	0	whole fruit	1.0	456-FR026-89D[3]

Country Report No. (year) States	Application				PHI, days	Sample[1]	Residues, mg/kg	Trial Reference
	Product	No	kg ai/ha	kg ai/hl				
					3 7 14		0.85 0.78 0.55	
		6	0.16	0.017	0 3 7 14	whole fruit	0.40 0.50 0.14 0.09	758-FR027-89D
		6	0.19	0.067	0 3 7 14	whole fruit	0.53 0.34 0.19 0.03	855-FR028-89D
MR99826-1 (1991) CA, ID, MI, NY, OR,		6	0.16	0.022	0 1 3 7	whole fruit	0.09 0.12 0.08 0.08	FCA-FR013-91D
WA and WI		6	0.25	0.054	0 1 3 7	whole fruit	1.4 1.4 1.3 0.97	451-FR011-91D
		6	0.063	0.0067	0	whole fruit	0.19	451-FR018-91D
		6	0.15	0.0067	0 1 3 7	whole fruit	0.31 0.19 0.08 0.06	454-FR012-91D
		6	0.19	0.010/0.04 0	0 1 3 7	whole fruit	0.92 0.67 0.70 0.33	758-FR014-91D
		6	0.19	0.010/0.04 0	0 1 3 7	whole fruit	0.76 0.86 0.71 0.52	855-FR017-91D
		6	0.16	0.034	0 1 3 7	whole fruit	1.2 0.47 0.32 0.13	851-FR015-91D

[1]Fruit: fruit without stone; whole fruit: fruit with stone
[2] 0.19 kg ai/ha nominal rate. Actual rates differed owing to different tree sizes
[3] Nominal rate <0.095 kg ai/ha

Peaches and Nectarines (Table 6). In two trials on peaches in France at nearly twice the GAP rate the residues in fruit with and without the stone at a PHI of 7 days were 0.09-0.11 mg/kg.

In Italy, one trial was conducted on peaches and two on nectarines with four applications instead of the one or two allowed by GAP. The residues after 7 or 10 days were <0.02-0.17 mg/kg.

In seven trials on peaches in the USA the applications rates were slightly below the GAP nominal rate (0.25 kg ai/kg). Residues in the whole fruit at a PHI of 0 days were 0.20-0.81 mg/kg. One trial with a lower application rate gave a residue of 0.04 mg/kg.

Table 6. Residues of tebuconazole in peaches and nectarines.

Country, Report No.,	Application	PHI,	Sample[1]	Residues,	Trial

(year), Fruit, States	Product	No	kg ai/ha	kg ai/hl	days		mg/kg	Reference
France (1988) peaches	25 WP	3	0.28/ 0.25	0.025	7 7	fruit/ whole fruit	0.10/0.11 0.09/0.11	0448-88/ 0449-88
Italy (1996) peaches	25 WG	4	0.28	0.019	0 3 7 10	whole fruit	0.18 0.34 0.06 0.14	0591-96
(1993) nectarines		4	0.28	0.019	0 7	whole fruit	0.34 0.17	0590-96
		4	0.28	0.019	0 3 7 10	fruit	0.09 0.05 0.06 <0.02	0289-93
USA [2] MR103208 (1990) peaches OR, WA, CA, SC, PA, MI, GA .	45 DF	6	0.13	0.0069	0 3 7 14	whole fruit	0.81 0.54 0.38 0.27	FCA-FR013-90D
		6	0.13	0.0069	0 3 7 14	whole fruit	0.34 0.26 0.17 0.12	451-FR007-90D
		6	0.18	0.0067	0	whole fruit	0.44	752-FR010-90D
		6	0.03	0.0063	0 3 7 14	whole fruit	0.04 0.03 0.02 <0.01	454-FR008-90D
		6	0.19	0.035	0 3 7 14	whole fruit	0.46 0.41 0.18 0.07	455-FR009-90D
		6	0.05	0.0067	0 3 7 18	whole fruit	0.26 0.09 0.03 0.03	752-FR014-90D
		6	0.19	0.026/ 0.022	0 3 7 14	whole fruit	0.20 0.12 0.13 0.04	757-FR011-90D
		6	0.19	0.026/ 0.022	0 3 7 14	whole fruit	0.21 0.17 0.16 0.04	855-FR012-90D

[1]Fruit: fruit without stone; whole fruit: fruit with stone
[2]0.19 kg ai/kg nominal rate. Actual rates differed owing to different tree sizes

Plums (Table 7). In nine trials in France at a higher rate (in 1988, 1991 and 1992) or a higher spray concentration (in 1994) than the proposed use (1-3 applications of 0.13-0.15 kg ai/ha) the residues were 0.03-0.40 mg/kg in fruit with or without the stone at a PHI of 7 days. In another ten trials according to the proposed use the corresponding residues were below the LOD (0.01 or 0.02 mg/kg) to 0.1 mg/kg after 7 to 79 days.

Three trials in Italy according to the proposed use (1-2 applications of 0.28 kg ai/ha) gave residues from 0.03-0.11 mg/kg at a PHI of 7 days.

Table 7. Residues of tebuconazole in plums.

Country, Report No., (year)	Application				PHI	Sample[1]	Residues,	Trial
	Product	No	kg ai/ha	kg ai/hl	days		mg/kg	Reference
France (1988)	25 WP	3	0.25	0.025	7	Fruit/ whole fruit	0.40/0.35	0450-88
(1991)		3	0.25	0.025	0 3 7	fruit	0.11 0.14 0.24	0391-91
(1991)		3	0.25	0.025	0 3 7	fruit	0.39 0.17 0.38	0392-91
(1991)		3	0.25	0.025	3 7	fruit	0.17 0.28	0393-91
		3	0.21-0.33	0.019	0 7 14 0 7 14	fruit whole fruit	0.09 0.03 0.03 0.09 0.03 0.03	0412-92
RA-2109/93 (1993)	25 WG	3	0.13-0.15	0.012-0.013	0 5 7 14 7 14	fruit whole fruit	0.03 0.02 <0.02 <0.02 <0.02 <0.02	0479-93
		3	0.13-0.15	0.012-0.013	0 5 7 14 7 14	fruit whole fruit	0.03 <0.02 0.03 <0.02 0.03 <0.02	0480-93
		1	0.13	0.013	49/77	fruit	<0.01/ <0.01	RPRUN932-03-C/ RPRUN933-02-C
(1994)		3	0.13	0.031	7 14	fruit	0.23 0.20	RPRUN294-06-A RPRUN294-07-A
(1993)	25 WG	2	0.13	0.025	29	fruit	<0.01/ <0.01	RPRUN932-03-A/ RPRUN932-03-B
		1	0.13	0.025	12	fruit	<0.01/0.03	RPRUN933-02-A/ RPRUN933-02-B
(1994)		3	0.13	0.025	7 14	fruit	0.1 0.1	RPRUN294-06-B RPRUN294-07-B
Italy (1992)	25 WG	2	0.3	0.019	0 7 10 14	fruit/ whole fruit	0.15/0.14 0.10/0.09 0.09/0.08 0.05/0.05	0284-92
RA-2067/93 (1993)		2	0.3	0.019	0 3 7 10	fruit	0.13 0.04 0.03 0.03	0035-93
		2	0.3	0.019	0 7	fruit	0.23 0.11	0291-93

[1]Fruit: fruit without stone; whole fruit: fruit with stone.

Grapes (Table 8). Fifteen trials were conducted in the USA with eight applications of 0.13 kg ai/ha, the proposed use pattern. The residues at a PHI of 13 or 14 days were between 0.10 and 3.95 mg/kg.

Table 8. Residues in bunches of grapes from trials with eight applications of 45 WG formulation of tebuconazole in the USA (CA, MI, NC, NY, OR and WA) at 0.13 kg ai/ha.

Report no. (year)	Application kg ai/hl	PHI, days	Residues, mg/kg	Trial reference
MR107132 (1995)	0.026	7 14 21	0.20 0.20 0.15	FCA-FR006-91D
	0.027	7 14 21	0.84 0.67 0.55	454-FR001-91D
	0.027	6 13 19	2.85 3.95 4.63	457-FR002-91D
	0.0090	7 14 25	1.72 1.77 1.67	458-FR003-91D
	0.014	7 14 21	0.85 0.94 0.74	751-FR098-91D
	0.014	7 14 21	0.37 0.27 0.18	758-FR004-91D
	0.027	7 14 21	0.51 0.56 0.21	855-FR005-91D
MR95677 (1988)	0.014	14 21	0.43 0.28	151-FR087-87D
	0.045	14 21	0.29 0.46	151-FR008-87D
	0.0067	14 21	0.37 0.27	451-FR089-87D
	0.045	14 21	0.56 1.0	454-FR090-87D
	0.027	14 21	1.5 0.56	456-FR092-87D
	0.0090	14 21	1.2 1.4	457-FR093-87D
	0.019-0.029	14 20	0.39 0.41	458-FR094-87D
	0.014	14 21	0.10 0.07	855-FR095-87D

Bananas (Table 9). The list of registered use of tebuconazole in bananas has been largely extended since 1994 (Table 2). Tebuconazole is registered in the USA for use on bagged bananas. In Australia, bagging before spraying is recommended to minimise fruit marking.

A total of six trials were conducted in Australia. In one trial according to GAP (5-6 applications of 0.1 kg ai/ha) the residues in the peel, pulp and whole fruit were at the limit of determination (0.01 mg/kg) at the GAP PHI of 1 day. In four trials at a higher rate, the residues were <0.05 and 0.03 mg/kg at a PHI of 1 day in bagged bananas (two trials). In two trials with unbagged bananas the residues were 0.16 mg/kg in the whole fruit and 0.14 and 0.17 mg/kg in the pulp.

In two trials in Brazil with five applications of 0.13 or 0.25 kg ai/ha the residues in pulp were below the limit of determination (<0.1 mg/kg) after 14 days.

In six of seven trials in the USA according to GAP with five applications of 0.1 kg ai/ha, the residues in the whole fruit, peel and pulp were [0.01 mg/kg at a PHI of 0 days. In the seventh trial the residue in the whole fruit was 0.03 mg/kg.

Table 9. Residues of tebuconazole in bananas. Bananas were bagged unless otherwise indicated.

Country, Report No. (year)	Application				PHI, days	Sample[1]	Residues, mg/kg	Trial Reference
	Product	No	kg ai/ha	kg ai/hl				
Australia (1988)	250 EC	4	0.075	0.0015	0	peel/pulp	<0.05/<0.05	
					1		<0.05/<0.05	AUS-43-88C
					3		<0.05/<0.05	
					5		<0.05/<0.05	
					7		<0.05/<0.05	
					10		<0.05/<0.05	
					14		<0.05/<0.05	
		4	0.15	0.003	0	peel/pulp	<0.05/<0.05	
					1		<0.05/<0.05	AUS-43-88D
					3		<0.05/<0.05	
					5		<0.05/<0.05	
					7		<0.05/<0.05	
					10		<0.05/<0.05	
					14		<0.05/<0.05	
(1992)	250 EW	6	0.1	0.046	0	whole fruit	0.03	
					1		0.01	AUS-40-90-E
					3		<0.01	
					5		<0.01	
					7		<0.01	
					0	peel	0.03	
					1		0.01	
					3		<0.01	
					5		<0.01	
					7		<0.01	
					0	pulp	0.03	
					1		0.01	
					3		<0.01	
					5		<0.01	
					7		0.01	
		6	0.2	0.093	0	whole fruit	0.03	
					1		0.03	AUS-40-90-G
					3		0.01	
					5		0.01	
					7		<0.01	
					0	peel	0.02	
					1		0.02	
					3		0.01	
					5		0.02	
					7		<0.01	
					0	pulp	0.03	
					1		0.03	
					3		0.01	
					5		0.01	
					7		<0.01	
		6	0.2	0.093	0	whole fruit	0.08	AUS-40-90-F (unbagged)
					1		0.16	
					3		0.10	
					5		0.08	
					7		0.03	
					0	peel	0.05	
					1		0.21	
					3		0.20	
					5		0.06	

Country, Report No. (year)	Application				PHI, days	Sample[1]	Residues, mg/kg	Trial Reference
	Product	No	kg ai/ha	kg ai/hl				
					7		0.02	
					0	pulp	0.10	
					1		0.14	
					3		0.06	
					5		0.09	
					7		0.02	
		6	0.2	0.093	0	whole fruit	0.15	AUS-40-90-H (unbagged)
					1		0.16	
					3		0.25	
					5		0.14	
					7		0.07	
					0	peel	0.18	
					1		0.13	
					3		0.32	
					5		0.16	
					7		0.06	
					0	pulp	0.14	
					1		0.17	
					3		0.22	
					5		0.13	
					7		0.06	
Brazil	250 EC	5	0.13	0.83	14	pulp	<0.1	BRA-140382-A
(1993)		5	0.25	1.67	14	pulp	<0.1	BRA-140382-B
USA MR99827 (1989) Hawaii, Puerto Rico	45 WG	5	0.1	0.045-0.053	0	whole fruit	<0.01	458-FR057-88D1
					7	unwashed	<0.01	
					14		<0.01	
					0	whole fruit	<0.01	
					7	washed	<0.01	
					14		<0.01	
					0	peel	0.01	
					7	unwashed	0.01	
					14		<0.01	
					0	pulp	<0.01	
					7	unwashed	<0.01	
					14		<0.01	
USA MR99827 (1989) Hawaii, Puerto Rico	45 WG	5	0.1	0.045-0.053	0	whole fruit	<0.01	
					7	unwashed	0.01	458-FR058-88D1
					14		0.02	
					0	whole fruit	<0.01	
					7	washed	0.01	
					14		<0.01	
					0	peel	<0.01	
					7	unwashed	0.02	
					14		0.02	
					0	pulp	0.01	
					7	unwashed	0.01	
					14		0.02	
USA MR99827 (1989) Hawaii, Puerto Rico	45 WG	5	0.1	0.045-0.053	0	whole fruit	<0.01	458-FR057-88D1
					7	unwashed	<0.01	
					14		<0.01	
					0	whole fruit	<0.01	
					7	washed	<0.01	
					14		<0.01	
					0	peel	0.01	
					7	unwashed	0.01	
					14		<0.01	
					0	pulp	<0.01	
					7	unwashed	<0.01	

Country, Report No. (year)	Application				PHI, days	Sample[1]	Residues, mg/kg	Trial Reference
	Product	No	kg ai/ha	kg ai/hl				
					14		<0.01	
USA MR99827 (1989) Hawaii, Puerto Rico	45 WG	5	0.1	0.045-0.053	0	whole fruit	<0.01	
					7	unwashed	0.01	458-FR058-88D1
					14		0.02	
					0	whole fruit	<0.01	
					7	washed	0.01	
					14		<0.01	
					0	peel	<0.01	
					7	unwashed	0.02	
					14		0.02	
					0	pulp	0.01	
					7	unwashed	0.01	
					14		0.02	
USA MR99827 (1989) Hawaii, Puerto Rico	45 WG	5	0.1	0.045-0.053	0	whole fruit	<0.01	750-FRO59-88D1
					7	unwashed	<0.01	
					14		<0.01	
					0	whole fruit	<0.01	
					7	washed	<0.01	
					14		<0.01	
					0	peel	0.01	
					7	unwashed	0.01	
					14		<0.01	
					0	pulp	<0.01	
					7	unwashed	<0.01	
					14		<0.01	
USA MR99827 (1989) Hawaii, Puerto Rico	45 WG	5	0.1	0.045-0.053	0	whole fruit	<0.01	750-FRO60-88D1
					7	unwashed	<0.01	
					14		<0.01	
					0	whole fruit	<0.01	
					7	washed	<0.01	
					14		<0.01	
					0	peel	0.01	
					7	unwashed	0.01	
					14		<0.01	
					0	pulp	<0.01	
					7	unwashed	<0.01	
					14		<0.01	
MR99827-1 (1991) Hawaii, Puerto Rico	45 WG	5	0.1	0.031-0.043	0	whole fruit	0.03	458-FR007-91D
					7	unwashed	0.03	
					14		0.04	
					0	whole fruit	0.03	
					7	washed	0.03	
					14		0.03	
MR99827-1 (1991) Hawaii, Puerto Rico	45 WG	5	0.1	0.031-0.043	0	whole fruit	<0.01	
					7	unwashed	<0.01	458-FR008-91D
					14		<0.01	
					0	whole fruit	<0.01	
					7	washed	<0.01	
					14		<0.01	
		5	0.1	0.031-0.043	0	whole fruit	<0.01	750-FR009-91D
					7	unwashed/	<0.01	
					14	washed	<0.01	

Bulb vegetables

Garlic (Table 10). In four trials in Brazil with five and six applications at 0.25 and 0.50 kg ai/ha the residues were below the LOD (0.05 mg/kg) after a PHI of 14 days. GAP calls for 1-4 foliar applications of 0.25 kg ai/ha. Five trials in France according to the proposed use (1-2 foliar applications of 0.25 kg ai/ha) gave residues from below the LOD (0.02 mg/kg) to 0.06 mg/kg after a PHI of 21 days.

In five trials in Korea with 4 x 0.38 mg ai/ha, foliar spray, the residues varied from <0.01 mg/kg at 245 days to 1.4 mg/kg at 51 days. The residues in two trials with soil drenches at 3.3 kg ai/ha were <0.01 mg/kg at 275 days and 0.65 mg/kg at 51 days.

Table 10. Residues of tebuconazole in garlic. Bulbs analysed.

Country, (year)	Application				PHI, Days	Residues, mg/kg	Trial Reference
	Product	No	kg ai/ha	kg ai/hl			
Brazil (1992)	250 EC	5	0.25	0.05	14	≤0.05	BRA-138740-A
		5	0.50	0.1	14	<0.05	BRA-138740-B
	25 WP	6	0.25	0.05	14	<0.05	BRA-140128-A
		6	0.50	0.1	14	<0.05	BRA-140128-B
France 1994	250 EC	2	0.25	0.041	21	<0.02	RAIL 0194-01
1995	250 EW	2	0.25	0.044	0 5 10 14 21	<0.02/<0.02 0.02 0.03 <0.02 <0.02/ 0.06	RAIL0195/ 84-1/ RAIL0195/ 09-1
1996		2	0.25	0.063	0 6 10 14 21	0.03/0.03 0.03 0.02 0.04 0.02/0.03	RAIL0195/ 84-2/ RAIL0195/ 09-2
Korea (1990) (1991)	250 EC	1	3.3 soil drench	0.017	275	<0.01	KOR-950-90
		1	0.38	0.025	245	<0.01	KOR-951-90
		2	0.38	0.025	90	0.01	KOR-952-90
		3	0.38	0.025	70	0.02	KOR-953-90
		3	0.38	0.025	51	0.15	KOR-954-90
		3	3.3 soil drench	0.017	51	0.65	KOR-955-90
		4	0.38	0.025	51	1.4	KOR-956-90

Onions (Table 11). In one trial in France, one in Germany, one in Italy and four in The Netherlands with 2 or 4 foliar applications of 0.19-0.25 kg ai/ha, close to the proposed German use pattern of 1 or 2 foliar applications at 0.25 kg ai/ha, the residues after 20-28 days were below the LOD (0.02 or 0.05 mg/kg).

In four trials in Brazil above the GAP rate (1-4 x 0.25 kg ai/ha), the residues were <0.1-0.3 mg/kg at a PHI of 14 days.

In two trials in New Zealand according to GAP (2-3 foliar applications of 0.38 kg ai/ha) the residues were 0.14 mg/kg at day 28 and below the LOD (0.05 mg/kg) after 76 days. In two trials in Australia at 0.50 kg ai/ha the residues were below the LOD (0.01 mg/kg) after 79 days and 0.3 mg/kg after 154 days.

In Spanish GAP application is by soil drench. In two trials with foliar applications the residues at 14 days were at or below the LOD (0.02 mg/kg).

Table 11. Residues of tebuconazole in onions. Bulbs analysed.

Country, Report No. (year)	Application				PHI	Residues,	Trial
	Product	No	kg ai/ha	kg ai/hl	days	mg/kg	Reference
Australia (1990)	250 EC	1	0.50	0.25	154	0.3	AUS-4-90
(1994)		2	0.50	0.20	79	<0.01	AUS-44-94
Brazil (1992)	250 EC	6	0.25 0.50	0.063 0.13	14 14	0.16 0.3	BRA-137021-A BRA-137021-B
		6	0.25 0.50	0.063 0.13	14 14	<0.1 0.3	BRA-136972-A BRA-136972-B
France RA-2085/96 (1996)	250 EW	2	0.25	0.089	0 5 10 14 21	1.3 0.13 0.05 <0.05 <0.05	0284-96
Germany (1996)	250 EW	2	0.25	0.042	0 21	<0.05 <0.05	0345-96
Italy (1989)	50 WG	2	0.25	0.025	0 10 14 20	0.05 <0.02 <0.02 <0.02	0374-89
New Zealand (1990)	250 EC	3	0.38	0.038	0 6 13 20 28	4.1 0.69 0.36 0.28 <u>0.14</u>	NSL-DECF
		3	0.38	0.038	76	<.05	NSL-ENDF3
Netherlands RA-2070/93 (1993)	50 WP	4	0.19-0.20	0.067	0 7 14 21 28	<0.02 <0.02 <0.02 <0.02 <0.02	0070-93
		4	0.19-0.20	0.067	0 7 14 21 28	<0.02 <0.02 <0.02 <0.02 <0.02	0296-93
		4	0.19-0.20	0.067	0 28	0.02/0.02 <0.02/<0.02	0298-93/ 0299-93
Spain (1991)	250 EC	2 2	0.50 0.50	0.05 0.05	14 14	<0.02 0.02	SPA-505-91 SPA-707-91

Cucumbers (Table 12). In two trials in Italy according to the proposed rate (1-4 x 0.125 kg ai/ha) the residues were below the LOD (0.02 mg/kg) after 7 days. In five indoor trials in Spain according to GAP (1-3 x 0.2-0.3 kg ai/ha) the residues at a PHI of 7 days were 0.03-0.19 mg/kg.

Table 12. Residues of tebuconazole in cucumbers. Whole cucumbers analysed.

Country, Report No. (year)	Application				PHI,	Residues,	Trial
	Product	No	kg ai/ha	kg ai/hl	days	mg/kg	Reference
Italy RA-2066/93	25 WG	5	0.1	0.01	0 3	0.05 <0.02	0028-93

Country, Report No. (year)	Application				PHI, days	Residues, mg/kg	Trial Reference
	Product	No	kg ai/ha	kg ai/hl			
(1993)					7 10	<0.02 <0.02	
		5	0.1	0.01	0 7	0.08 <0.02	0294-93
Spain[1] RA-2022/92 (1992)	50 WP	3	0.3	0.02	0 3 7 10	0.47/0.24 0.23/ 0.19/0.08 0.10/0.04	0154-92/ 0156-92
		3	0.3	0.02	0 3 7 10	0.50 0.20 0.10 0.06	0155-92
RA-2071/93 (1993)		3	0.2-0.29	0.02	0 3 5 7 10	0.12 0.07 0.06 0.03 0.03	0355-93
		3	0.2-0.29	0.02	0 3 7	0.30 0.09 0.03	0356-93

[1]Indoor

Sweet peppers (Table 13). In three trials in Spain according to GAP (1-3 applications of 0.2-0.3 kg ai/ha) the residues were 0.07-0.14 mg/kg at a PHI of 7 days.

Table 13. Residues of tebuconazole in sweet peppers in Spain from 2 applications of 0.2 kg ai/ha of 50 WP (0.02 kg ai/hl). Whole peppers analysed.

Report No. (year)	PHI days	Sample analysed	Residues mg/kg	Trial Reference
RA-2022/92 (1992)	0 3 7 10	fruit	0.19 0.13 0.13 0.10	0151-92
	0 3 7 10	fruit	0.20 0.19 0.14 0.14	0152-92
	0 7 11	fruit	0.42 0.07 0.05	0153-92

Barley (Table 14). In eleven trials in the USA, seed was treated once at the GAP application rate of 0.0020 (10 trials) or 0.024 kg ai/100 kg (one trial). At harvest (81 to 129 days) the residues in the grain were below the LOD (0.01 or 0.02 mg/kg).

Table 14. Residues of tebuconazole in barley in the USA (CA, ID, MN, NE and WA) from 1 seed treatment.

Report No. (year)	Application Product	kg ai/100kg	PHI, days	Sample	Residues, mg/kg	Trial Reference
MR103841	31 FS	0.0020	44	forage	<0.03	FCA-FR044-91H
(1992)		0.0020	42	forage	<0.03	HIN-FR064-91H
			110	grain	≤0.02	
			110	straw	<0.05	
		0.0020	59	forage	<0.03	251-FR041-91H
			95	grain	≤0.02	
			95	straw	<0.05	
		0.0020	34	forage	<0.03	252-FR065-91H
			105	grain	≤0.02	
			105	straw	<0.05	
		0.0020	48	forage	<0.03	452-FR042-91H
			126	grain	≤0.02	
			126	straw	<0.05	
		0.0020	85	forage	<0.03	454-FR043-91
			129	grain	≤0.02	
			129	straw	<0.05	
MR99125 (1991)	312 SC	0.024	58	forage	<0.01	457-FR007-88H
			58	hay	0.27	
			93	grain	<0.01	
			93	straw	0.02	
		0.0020	38	forage	<0.01	251-FR003-88H
			40	hay	<0.01	
			81	grain	≤0.01	
			81	straw	<0.01	
		0.0020	31	forage	<0.01	452-FR004-88H
			36	hay	<0.01	
			98	grain	≤0.01	
			98	straw	<0.01	
		0.0020	60	forage	<0.01	453-FR005-88H
			60	hay	<0.01	
			104	grain	≤0.01	
			104	straw	0.04	
		0.0020	39	forage	<0.01	454-FR006-88H
			39	hay	<0.01	
			111	grain	≤0.01	
			111	straw	<0.01	

Oats (Table 15). In eleven trials in the USA according to GAP the residues in the grain at harvest (78 to 122 days) were below the LOD (0.01 mg/kg).

Table 15. Residues of tebuconazole in oats in the USA (IA, IL, IN, KS, MN, NE, NY, TX and WI). All trials with 1 seed treatment of 0.0020 kg ai/100kg.

Report No. (year)	Product applied	PHI, days	Sample	Residues, mg/kg	Trial Reference
MR103939 (1992)	31 FS	42	forage	<0.02	HIN-FR066-91H
		110	grain	≤0.01	
		110	straw	<0.06	
		59	forage	<0.02	251-FR045-91H
		95	grain	<0.01	
		95	straw	≤0.06	
		38	forage	<0.02	252-FR049-91H
		105	grain	≤0.01	
		105	straw	<0.06	

Report No. (year)	Product applied	PHI, days	Sample	Residues, mg/kg	Trial Reference
		39 105 105	forage grain straw	<0.02 <0.01 <0.06	255-FR046-91H
		53 109 109	forage grain straw	<0.02 <0.01 <0.06	353-FR048-91H
		30 83 83	forage grain straw	<0.02 <0.01 <0.06	853-FR047-91H
MR99124 (1989)	312 SC	55 55 105 105	forage hay grain straw	<0.01 <0.01 <0.01 <0.01	HIN-FR012-88H
		57 60 122 122	forage hay grain straw	<0.01 <0.01 <0.01 <0.01	STF-FR013-88H
		51 51 120 120	forage hay grain straw	<0.01 <0.01 <0.01 <0.01	151-FR009-88H
		38 40 88 88	forage hay grain straw	<0.01 0.02 <0.01 <0.01	251-FR010-88H
		36 36 78 78	forage hay grain straw	<0.01 <0.01 <0.01 <0.01	851-FR011-88H

Wheat (Table 16). Six trials in the USA were according to GAP. At harvest (81 to 275 days) the residues in the grain were below the LOD (0.01/0.04 mg/kg).

Table 16. Residues of tebuconazole in wheat in the USA (IN, ID, MN and WA) from 1 seed treatment at 0.002 kg ai/100kg.

Report No. (year)	Application Product	PHI, days	Sample	Residues, mg/kg	Trial Reference
MR103917 (1992)	31 FS	192 275	forage grain/straw	<0.02 <0.04/<0.05	HIN-FR028-91H
		61 160	forage grain/straw	<0.02 <0.04/<0.05	454-FR026-91H
		59 95	forage grain/straw	<0.02 <0.04/<0.05	851-FR032-91H
MR98555 (1991)	312 SC	38 40 81	forage hay grain/straw	0.04 0.08 <0.01/<0.01	251-FR001-88H
		31 36 98 89	forage hay grain straw	<0.01 <0.01 <0.01 <0.01	452-FR002-88H
		39 111	forage/hay grain/straw	<0.01/ <0.01 <0.01/ <0.01	454-FR042-88H

Peanuts (Table 17). In thirteen trials in the USA the number and rate of applications were above GAP (1-4 applications of 0.23 kg ai/ha). Residues in the kernels were from below the LOD (0.01 and 0.05 mg/kg) to 0.08 mg/kg after 7 to 14 days (the GAP PHI).

Table 17. Residues of tebuconazole in peanuts in the USA (AL, FL, GA, MS, OK and TX) from 7 applications of 3.6 F formulation.

Report No. (year)	Application		PHI days	Sample	Residues, mg/kg[1]	Trial Reference
	kg ai/ha	kg ai/hl				
MR 99129 (1991)	0.25	0.11 -0.13	5 14	kernels/hulls/hay	<0.02/0.38/10.6 <0.01/0.17/12.5	352-FR046-88D
	0.25	0.17-0.27	7 14	kernels/hulls/hay	0.04/0.08/28.8 0.08/1.8/17.0	353-FR047-88D
	0.25	0.17-0.27	7 14	kernels/hulls/hay	<0.01/0.18/2.5 <0.01/0.27/1.8	754-FR049-88D
	0.25	0.17-0.27	7 14	kernels/hulls/hay	0.05/2.0/8.4 0.03/2.2/5.0	BMS-FR050-88D
	0.26- 0.29	0.12 -0.14	3 7	kernels/hulls/hay	0.02/0.54/14.4 0.01/0.71/20.6	TGA-FR051-88D
MR100073 (1991)	0.25	0.13 -0.68	7 14	kernels/hulls/hay	0.05/0.56/7.9 0.03/0.37/5.1	VBL-FR042-89D
	0.25	0.53	6 13	kernels/hulls/hay	0.04/0.54/2.4 0.03/0.49/18.3	352-FR043-89D
	0.25	0.13 -0.68	7 14	kernels/hulls/hay	<0.01/0.02/10.9 <0.01/0.14/3.7	353-FR044-89D
	0.25	0.13 -0.68	7 14	kernels/hulls/hay	0.01/0.46/13.6 <0.01/0.45/11.3	TGA-FR045-89D
MR101344 (1991)	0.25 -0.30	0.12 -0.27	7 14	kernels/hulls/hay	<0.05/1.8/22.3 <0.05/1.2/9.1	353-FR016-90D
	0.25 -0.30	0.12 -0.27	7 14	kernels/hulls/hay	0.05/0.5/18.0 <0.05/0.55/15.5	751-FR017-90D
	0.25-0.30[2]	0.12 -0.27	7 14	kernels/hulls/hay	<0.05/0.28/13.4 <0.05/0.46/8.6	TGA-FR019-90D
	0.25 -0.30	0.12 -0.27	7 14	kernels/hulls/hay	0.05/0.79/13.9 <0.05/0.85/9.4	VBL-FR020-90D

[1] Although the trials exceeded GAP conditions, they were considered for estimating maximum residue levels
[2] The first two applications were at 0.057 kg ai/ha and 0.027 kg ai/hl

FATE OF RESIDUES IN STORAGE AND PROCESSING

In storage

No data were available

In processing

The trials reported in 1994 have been supplemented by trials on plums, grapes and peanuts.

Plums. Plum trees were treated three times with 0.25 kg ai/ha of a 25 WG formulation of tebuconazole. The initial residue (day 0) was 0.09 mg/kg. In the plums taken for processing (day 7) it was 0.03 mg/kg.

The plums were washed and stoned with a plum stoner. Plum jam was prepared on a household scale by cooking crushed plums with sugar. The industrial production of plum preserve was simulated on a laboratory scale. Washed, stoned and cut plums were autoclaved together with a sugar solution in preserving pans (4 minutes at 90°C). The preparation of dried prunes also simulated industrial processing. Washed plums were blanched, dipped in a potassium carbonate solution and oven-dried for 14-20 hours at 70-75°C. The results are shown in Table 18.

Table 18. Tebuconazole residues and processing factors in plums and processed products.

Product	Residue (mg/kg)	PF
Raw plums	0.03	--
Washed plums	0.02	0.7
Jam	0.03	1
Preserve	<0.02	<0.7
Dried prunes	0.14	4.7

Grapes. Grapes were treated four times with 0.25 kg ai/ha of a 45 WG formulation of tebuconazole. Samples for processing were taken immediately after the last application. The residues in the unprocessed grapes was 0.16 mg/kg. The grapes were processed to raisins (sun- and oven-dried), raisin waste (sun- and oven-dried), wet and dried pomace, and juice. Oven-drying was at 60ºC.

The preparation of grape juice involved separation of the stems, crushing the berries, enzymatic depectinization of the crushed berries, finishing or pressing, clarification after heating to about 80°C and settling for 4 to 6 weeks, separation by decantation, filtration, and canning after heating to about 90°C. The pomace resulting from finishing or pressing was dried in an air drier at about 60°C. The results are shown in Table 19.

Table 19. Tebuconazole residues and processing factors in grapes and processed products

Product	Residue (mg/kg)	PF
Grapes	0.16	--
Raisins, Sun- oven-dried	0.14 0.21	0.9 1.3
Raisin waste Sun- oven-dried	0.64 1.7	4 10.6
Pomace, wet dried	1.2 3.5	7.5 21.9
Juice	<0.08	<0.5

Three supervised trials in Germany followed by processing to must, wine and juice were reported in the 1994 monograph. The manufacturer resubmitted the data (Table 20) as the original figures were incorrect. The processing factors for must, juice and wine varied from 0.04 or <0.05 to 0.22.

Table 20. Effect on residues of processing grapes treated with 1 to 3 applications of 0.3-0.625 kg ai/ha 50 WG, PHI 49 days. Germany, 1989. Revised results.

Trial No.	Residues, mg/kg				
	Fruit	Must	Juice	Wine	Processing factor Must/Juice/Wine
0260-89	1.0	0.13	0.06	0.15	0.13/0.06/0.15
0261-89	0.46	0.07	0.02	0.07	0.15/0.04/0.15
0262-89	0.36	0.08	<0.02	0.05	0.22/<0.05/0.14

Peanuts. Plants were treated four times with Folicur 432 SC at an application rate of 1.26 kg ai/ha, five times the maximum allowed seasonal rate, to produce measurable residues. The plants were dug up 14 days after the last application and allowed to dry in the field for six days before final harvesting. The residues in unprocessed peanuts were 0.07 mg/kg.

Peanut meal, crude oil, soapstock and refined oil were produced by procedures simulating commercial processing. The peanuts were mechanically hulled and pressed, yielding crude oil and presscake. The presscake was extracted to leave peanut meal and the crude oil was treated with sodium hydroxide to yield soapstock and alkali-treated oil. Refined oil was produced by bleaching and deodorising the alkali-treated oil. The results are shown in Table 20.

Table 21. Tebuconazole residues and processing factors in peanuts and processed products.

Product	Residue, mg/kg	PF
Nut meat	0.07	--
Meal	0.06	0.86
Soapstock	0.24	3.4
Crude oil	0.14	2.0
Refined oil	<0.01	<0.14

NATIONAL MAXIMUM RESIDUE LIMITS

In addition to those reported in 1994, the following national MRLs were reported to the Meeting.

Country	Commodity	MRL, mg/kg
Argentina	Barley, Oats	0.2
	Potato	0.01
Brazil	Bean, Garlic, Guava, Melon, Onion, Peanut, Pumpkin, Strawberry	0.1
	Beetroot	0.2
	Carrot	0.6
	Citrus fruit	5.0
	Grape	2.0
Cuba	Banana	0.2
Czech, Republic	Barley	0.05
	Barley straw	5.0
France	Apple	0.2
	Apricot	0.3
	Barley, Buckwheat (common), Oats, Rape, Rye, Triticale, Wheat	0.05
	Barley straw, Wheat straw	4.0

Country	Commodity	MRL, mg/kg
	Grape	0.5
	Peach	0.3 T
	Pea (field)	0.1 T
Italy	Apple, Pear	0.1
	Apricot	0.2 T
	Barley, Wheat	0.05 T
	Grape	1.0 T
	Peach	0.1 T
Japan	Apple, Banana, Barley, Onion, Pea (garden), Potato, Rye	0.2 T
	Apricot, Cherry	0.3 T
	Asparagus, Citrus fruit, Sugar beet, Grape fruit, Hop, Lemon, Lime, Maize, Mandarin, Mushroom, Oil plants(seed), Orange, Orange (Japanese summer), Strawberry, Sugar cane, Tea	0.05 T
	Aubergine, Sweet pepper, Wheat, Wheat flour	0.5 T
	Cucumber	0.02 T
	Ginger, Tomato, Spinach	1.0 T
	Grape	2.0 T
	Peanut, Japanese radish (root)	0.1 T
Netherlands	All food	0.05*
New Zealand	Rye grass (seed crops)	0.1
South Africa	Tomato	0.1
	Grape	0.5
	Mango	0.05
	Potato	0.2
USA	Banana, Barley, Oats, Wheat	0.05
	Barley (forage, hay and straw), Oats (forage, hay and straw), Wheat (forage, hay and straw), Peanut	0.1
	Cattle (kidney, liver and meat by-products)	0.2 T[1]
	Cherry, Peanut hull	4.0
	Grass (forage)	8.0[1]
	Grass (hay)	25.0[1]
	Milk	0.1[1]
	Nectarine, Peach	1.0

[1]Sum of tebuconazole (*RS*)-1-*p*-chlorophenyl-4,4-dimethyl-3-(1*H*-1,2,3-triazol-1-ylmethyl)pentane-3,5-diol (HWG 2061)
T: Temporary MRL

APPRAISAL

Tebuconazole is a triazole fungicide used as a seed dressing and spray. It was first evaluated in 1994 when use patterns, methods of residue analysis, results from supervised trials, studies of metabolism and environmental fate, and storage and processing data were reported by the manufacturer. MRLs were recommended for barley, barley straw and fodder, grapes, peanut, peanut fodder, rape seed, rye, rye straw and fodder, summer squash, tomatoes, wheat, wheat straw and fodder, cattle edible offal, meat and milk, and chicken edible offal, eggs and meat. In studies of metabolism in wheat, grapes and peanuts, tebuconazole was the significant residue. Information received since the 1994 evaluation was reviewed by the present Meeting.

New methods of analysis of plant materials and soil were reported. After extraction with organic solvents and clean-up on Florisil, C-18 or silica columns, and/or gel permeation chromatography, tebuconazole is determined by gas chromatography with a nitrogen-phosphorus detector. In some cases, no clean-up step was required. The limits of determination were 0.01-0.05 mg/kg.

Two hundred and eighteen trials were reported to the Meeting, with information on registered uses on the relevant crops. Processing studies were on plums, grapes and peanuts.

The Meeting concluded that the definition of the residue for compliance with MRLs and for estimations of dietary intake should be tebuconazole.

Supervised trials

Pome fruits

GAP is established for the use of tebuconazole on apples in Brazil, France and Indonesia and on apples and pears in Italy, Israel, Turkey and Spain. PHIs vary from 10 to 30 days. There are proposed uses on apples and pears in the USA and apples in Germany in which the recommended PHIs are 75 and 56 days respectively. Results from trials on pome fruits show that residues decrease continuously with time after sprayed applications of tebuconazole.

Apples. In one trial in Brazil, two in Italy and one in Spain according to local GAP (1 to 6 applications of 0.09-0.23 mg/kg ai/ha) the residues at a PHI of 20-21 or 28 days in rank order were 0.12, 0.13, 0.18 and 0.20 mg/kg. In one further trial in France according to current GAP which was reported to the 1994 Meeting, the residue at a PHI of 21 days was 0.06 mg/kg. In three trials in Brazil, two in France and ten in Korea with more applications and/or higher rates (up to 1.25 kg ai/ha) than recommended GAP the residues varied from 0.04-0.5 mg/kg with PHIs of 14 to 35 days.

In two trials in Canada, 18 in the USA and 11 in Germany with applications below, at, or above proposed GAP rates in Germany and the USA (1-6 x 0.1-0.25 kg ai/ha) most residues were below the LOD of 0.01-0.02 mg/kg, with 7 values of 0.02-0.04 mg/kg at PHIs of 56 days or longer.

Pears. In one trial in Spain according to GAP (4-6 applications of 0.1-0.15 kg ai/ha) the residue was 0.09 mg/kg at a PHI of 21 days. In six trials in the USA at or below the proposed rates the residues varied from below the LOD (0.01 mg/kg) to 0.03 mg/kg after PHIs of 63 to 106 days.

Three trials in Italy according to GAP (1-4 applications of 0.15-0.28 kg ai/ha, PHI 15 days) and one trial in France according to Spanish GAP were reported in 1994. The residues in Italy were 0.43, 0.12 and 0.20 mg/kg after 14, 10 and 10 days respectively, and in France <0.05 mg/kg after 14 and 30 days. As the residues in the pears appeared to decrease slowly the residues after 10 and 15 days would probably be similar.

As GAP for apples and pears is similar in countries with registrations for both the residues from trials according to GAP in the two crops can be considered to form a single population. The residues from trials according to established GAP in rank order (median underlined) were <0.05, 0.06, 0.09, 0.12 (2) 0.13, 0.18, 0.20 (2) and 0.43 mg/kg.

The Meeting estimated a maximum residue level of 0.5 mg/kg and an STMR of 0.12 mg/kg for pome fruits.

Stone fruits

Tebuconazole is registered for use on peaches in Chile, France, Italy and Peru, on plums in Israel and on peaches and cherries in the USA. PHIs vary from 0 in the USA to 35 days in Chile. The results from trials on stone fruit show that residues after spray applications decrease steadily and fairly slowly.

Cherries. Tebuconazole is registered for use on cherries only in the USA. In five trials in Italy at or below proposed Italian GAP (1 or 2 applications of 0.28 kg ai/ha) the residues in the fruit with and without stone were 0.18-0.50 mg/kg after 5 to 7 days.

GAP in the USA allows 1-6 applications at a nominal rate of 0.25 kg ai/ha with a 0-day PHI. Twelve trials were carried out at a nominal rate of 0.19 kg ai/ha, the actual rate depending on the size of the trees. The residues at a PHI of 0 days in rank order were 0.09, 0.19, 0.31, 0.40, 0.41, 0.53, 0.61, 0.76 (median), 0.92, 1.2, 1.4 and 3.1 mg/kg (the last from 7 applications). The residues shown bold were from the highest actual application rates and have been used to estimate an STMR. The residue in another trial at half the application rate was 1.0 mg/kg at a 0-day PHI.

The Meeting estimated a maximum residue level of 5 mg/kg and an STMR of 0.76 mg/kg.

Peaches and nectarines. Two trials on peaches in France and one on peaches and two on nectarines in Italy were according to Italian GAP (1 or 2 x 0.15-0.3 kg ai/ha). The residues were below the LOD (0.02 mg/kg) to 0.17 mg/kg in stoned or whole fruit at a PHI of 7 to 10 days. In four trials on peaches in France according to current GAP, reported in 1994, the residues in stoned and whole fruit at a PHI of 7 days varied from 0.03-0.22 mg/kg.

In eight trials on peaches in the USA according to GAP (0.25 kg ai/ha) the residues in whole fruit at a PHI of 0 days were 0.20-0.81 mg/kg, and in one trial with an application below the GAP rate the residue was 0.04 mg/kg.

Residues from trials according to GAP in whole and stoned peaches in rank order were 0.03, 0.05, 0.11 (2), 0.13, 0.20, 0.21, 0.22, 0.26, 0.34, 0.44, 0.46 and 0.81 mg/kg.

The Meeting estimated a maximum residue level of 1 mg/kg and an STMR of 0.21 mg/kg for peaches.

Plums. Only Israel has a registered use for tebuconazole on plums. There is a proposed use in France.

In France the residues in the stoned or whole fruit from nine trials at a higher rate or spray concentration than the proposed use (1-3 applications of 0.13-0.15 kg ai/ha) were 0.03-0.38 mg/kg at a PHI of 7 days. In ten further trials according to the proposed use the residues were below the LOD (0.01 or 0.02 mg/kg) to 0.1 mg/kg after PHIs of 7 to 79 days.

As no trials according to approved GAP were reported, the Meeting could not estimate a maximum residue level.

Grapes. Tebuconazole is registered for use on grapes in Brazil, Chile, France, Germany, Israel, Italy, Spain and South Africa. The 1994 JMPR recommended an MRL of 2 mg/kg.

In 14 trials in the USA at the use pattern for which registration has been applied and a PHI of 14 days the residues were between 0.10 and 1.7 mg/kg, and in one further trial 4.0 mg/kg at 13 days.

As no additional results from trials according to GAP were reported, the Meeting made no change to the previous recommendation.

Bananas. Tebuconazole is registered for use on bananas in Australia, Cameroon, Colombia, Costa Rica, Ecuador, Guatemala, Honduras, Indonesia, the Ivory Coast, Nicaragua, the Philippines and the USA. A PHI of 0 or 1 day is recommended in all these countries.

In one trial in Australia and seven in the USA according to national GAP (5-7 applications of 0.1 kg ai/ha, bagged bananas) the residues in the whole fruit were <0.01 (6), 0.01 and 0.03 mg/kg and in the pulp <0.01 (5) and <0.05 mg/kg. Three other trials in Australia, one at a lower and two at a higher rate, gave similar results and could be used to support the results in the trials according to GAP. Two trials on unbagged bananas gave residues of 0.16 mg/kg in the whole fruit at a PHI of 1 day. Two trials in Brazil giving residues in the pulp below the LOD (0.1 mg/kg) after 14 days could not be evaluated owing to the lack of information on GAP.

The Meeting estimated a maximum residue level of 0.05 mg/kg and an STMR (based on residues in the pulp) of 0.01 mg/kg for tebuconazole in bananas.

Bulb vegetables

Tebuconazole is registered for use on garlic and onions in Brazil, Israel, and Spain (soil drench) and on onions in New Zealand and South Africa.

Garlic. In one trial in Brazil approximating GAP (1-4 applications of 0.25 kg ai/ha) and three others at a higher rate or with 6 applications the residues were below the LOD (0.05 mg/kg) after the GAP PHI of 14 days. Five trials in France according to proposed GAP gave residues from below the LOD (0.02 mg/kg) to 0.06 mg/kg after a PHI of 21 days.

In seven trials in Korea at various application rates and with spray or soil drench applications the residues were below the LOD after 275 days to 1.4 mg/kg after 51 days. No GAP was available with which to evaluate the trials.

The data from trials according to GAP were insufficient to estimate a maximum residue level.

Onions. In one trial in France, one in Germany, one in Italy and four in The Netherlands, at or close to the proposed German use pattern (1-2 foliar applications of 0.25 kg ai/ha), and in four trials in Brazil which exceeded GAP conditions (1-4 x 0.25 kg ai/ha) the residues after 14-28 days were below the LOD (0.02, 0.05, or 0.1 mg/kg) to 0.3 mg/kg. In Spain, where soil drench application is recommended, two trials with foliar applications gave residues at or below the LOD (0.02 mg/kg) after 14 days.

In two trials in New Zealand according to GAP (2-3 foliar applications of 0.38 kg ai/ha), the residues were 0.14 mg/kg at day 28 and below the LOD (0.05 mg/kg) after 76 days. The GAP PHI is 35 days. In two trials in Australia with 1 or 2 applications of 0.5 kg ai/ha, the residues were below the LOD (0.01 mg/kg) and 0.3 mg/kg after 79 and 154 days respectively.

There were insufficient data from trials according to GAP to estimate a maximum residue level.

Cucumbers. Tebuconazole is registered for use on cucumbers in Chile, Israel and Spain. PHIs vary from 7 to 35 days. There is a proposed use in Italy.

In two trials in Italy according to the proposed rate (1-4 applications of 0.125 kg ai/ha), the residues at a PHI of 7 days were below the LOD (0.02 mg/kg). Eight trials were in Spain according to current GAP (1-3 applications of 0.2-0.3 kg ai/ha), five indoor trials reported to the present Meeting and three field trials reported to the 1994 Meeting. The residues at a PHI of 7 days in rank order were <0.02, 0.02, 0.03 (2), 0.04, 0.08, 0.10 and 0.19 mg/kg.

The Meeting estimated a maximum residue level of 0.2 mg/kg and an STMR of 0.035 mg/kg.

Sweet peppers. Tebuconazole is registered for use on sweet peppers only in Spain, with 1-3 applications of 0.2-0.3 kg ai/ha.

In three trials in Spain reported to the present Meeting and four reported in 1994, all according to current GAP, the residues at a PHI of 7 days in rank order were 0.07, 0.13, 0.14 (2), 0.18, 0.23 and 0.36 mg/kg.

The Meeting estimated a maximum residue level of 0.5 mg/kg and an STMR of 0.14 mg/kg.

Cereal grains

Tebuconazole is registered for use on barley, oats and/or wheat as a seed or foliar treatment in many countries, including Australia, Spain, South Africa, Germany, the UK and the USA.

Barley. The 1994 JMPR recommended an MRL of 0.2 mg/kg based on residues from foliar applications.

In nine trials in the USA with seed treatment according to GAP the residues in grain samples were below the LOD (<0.01 (4) and 0.02 (5) mg/kg) at harvest (81 to 129 days). In one trial a 12-fold rate gave a residue of <0.01 mg/kg.

The Meeting did not change the 1994 estimate of 0.2 mg/kg as a maximum residue level.

Oats. In eleven trials with seed treatment in the USA according to GAP, all residues in grain samples were below the LOD (0.01 mg/kg) at harvest (78 to 122 days). The residues in straw and forage were also all below the LOD (0.01, 0.02 or 0.06 mg/kg). Residues in hay, determined in 5 trials, were <0.01 mg/kg in 4 trials and 0.02 mg/kg in the fifth.

Residues in the grain from trials with foliar treatments according to GAP (two in Australia and one in Sweden) reported to the 1994 Meeting were 0.06, 0.09 and 0.12 mg/kg.

On the basis of the US trials and the practical LOD for rye of 0.05 mg/kg indicated by the 1994 JMPR, the Meeting estimated a maximum residue level of 0.05* mg/kg for tebuconazole in oats. As the residues in straw, forage and hay from seed treatments were also below the LOD, except in one sample of hay, the Meeting estimated an STMR of 0 mg/kg for tebuconazole in oats. The Meeting recognized that these estimates would not accommodate foliar applications.

Wheat. The 1994 JMPR recommended an MRL of 0.05 mg/kg on the basis of residues from foliar applications.

In six trials with seed treatment according to GAP in the USA, the residues in grain samples at harvest (81 to 275 days) were below the LOD (0.01 or 0.04 mg/kg). The residues in 13 trials with foliar treatment in Germany and the UK reported to the 1994 JMPR, according to GAP at that time, were <0.05 mg/kg.

The Meeting confirmed the previous recommendation of 0.05 mg/kg as an MRL.

Peanuts. Tebuconazole is registered for use on peanuts in Australia, Argentina, Brazil, Guatemala, Indonesia, Israel, Nicaragua, South Africa and the USA. The 1994 JMPR recommended an MRL of 0.05 mg/kg.

In thirteen US trials with 7 applications, instead of the 4 allowed by GAP, at rates slightly above the authorized 0.23 kg ai/ha, the residues in the kernels at or about the GAP PHI of 14 days in rank order were <0.01 (4), 0.01, 0.03 (3), <0.05 (4) and 0.08 mg/kg.

The Meeting confirmed the 1994 JMPR recommendation, as it is unlikely that residues would exceed 0.05 mg/kg.

Processing

Plums. Plum trees were treated three times with 0.25 kg ai/ha. In a processing study of samples taken after 7 days residues were reduced by a factor of 0.7 in washed and preserved plums, remained unchanged in jam and were increased by a factor of 4.7 in dried prunes. The Meeting agreed that one study was not sufficient to estimate processing factors.

Grapes. Grapes taken after the last of four applications of 0.25 kg ai/ha were processed. Processing factors were 0.9 and 1.3 for sun- and oven-dried raisins respectively, <0.5 for juice, 4 and 10.6 for sun- and oven-dried raisin waste, 7.5 for wet pomace and 21.9 for dry pomace. Processing studies reviewed by the 1994 JMPR showed processing factors of 1.4 and 1.2 for sun- and oven-dried raisins, 0.04, <0.05, 0.06 and 0.4 for juice, 2.7 and 1.5 for sun- and oven-dried raisin waste, and 1.8 and 5.8 for wet and dry pomace.

Residues in grapes, must and wine were determined in 37 trials reported to the 1994 JMPR (2 to 5 applications of 0.3-0.625 kg ai/ha). In three of these trials juice was also analysed but the results were reported incorrectly by the company in 1994; the correct values were supplied for the present Meeting. The mean and individual processing factors from all the trials were juice <0.21 (0.04, <0.05, 0.06, 0.4, <0.5), raisins 1.2 (0.9, 1.2, 1.3, 1.4), raisin waste 4.7 (4, 10.6, 2.7, 1.5), wet pomace 4.7 (1.8, 7.5), dry pomace 13.9 (5.8, 21.9), must 0.36 (range 0.12-0.78), wine 0.25 (range 0.05-0.78).

On the basis of the draft MRL of 2 mg/kg for grapes and the processing factor of 1.2 for raisins, the Meeting estimated a maximum residue level of 3 mg/kg for tebuconazole in dried grapes.

Peanuts. Plants treated at 5 times the maximum rate gave processing factors of 0.9 for peanut meal, 3.4 for soapstock, 2.0 for crude oil and 0.1 for refined oil. The Meeting agreed that one study was not sufficient to estimate processing factors for peanut products.

RECOMMENDATIONS

The Meeting recommends the estimated residue levels shown below to be used as maximum residue limits (MRL) and for dietary intake calculation of tebuconazole in oats:

Definition of residue for estimation of maximum residue and dietary intake levels: tebuconazole

CCN	Commodity	MRL, mg/kg	PHI, days	STMR, mg/kg
Fl 0327	Banana	0.05	0/1	0.01[a]
FS 0013	Cherries	5	0	0.76
VC 0424	Cucumber	0.2	7	0.035
DF 0269	Dried Grapes	3	7	
GC 0647	Oats	0.05*	78-122	0
FS 0247	Peaches	1	0/7	0.1
FP 009	Pome fruits	0.5	15/21	0.12
VO 0445	Sweet Peppers	0.5	7	0.14

a. based on residues on edible portions.

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TEBUFENOZIDE

EXPLANATION

Tebufenozide is a fat-soluble insecticide used to control Lepidoptera pests in fruits, vegetables and other crops. It was first reviewed by the 1996 JMPR when an ADI was allocated and MRLs were recommended. Kiwifruit was one of the crops for which residue data were provided, but a maximum residue level was not estimated because the data from trials in accordance with GAP were considered insufficient.

At the 29th (1997) Session of the CCPR the delegation of New Zealand requested a re-evaluation of tebufenozide resideus in kiwifruit on the basis of revised GAP in New Zealand.

USE PATTERN

Current New Zealand GAP indicates that tebufenozide is applied to kiwifruit to control leafrollers by high-volume spraying to run off. The first application is pre-bloom, the second at petal fall, and these may be followed by two further applications at intervals of 14 days. The details are shown in Table 1.

Table 1. Registered uses of tebufenozide on kiwifruit in New Zealand.

Formulation	Application			PHI, days
	No.	kg ai/ha	kg ai/hl	
70 WP	2-4	<0.12	0.006	90

RESIDUES RESULTING FROM SUPERVISED TRIALS

Kiwifruit. In a number of residue trials in New Zealand in 1990-95 (Tillman, 1995) single vine plots, with 5 replicates per treatment in a randomized block design, were sprayed using a motorized hand-lance plot-sprayer to the point of run-off. Each vine received approximately 6 litres of spray per application, which was reported as equivalent to 2000 l/ha.

Trials were conducted in 1990-93 to determine the efficacious use rates and effects of timing of applications, and to assess the residues in the crop at various pre-harvest intervals. Whole fruit samples were analysed by HPLC with UV detection, with HPLC-MS for confirmation. All the results were corrected for recoveries which ranged from 88.8 to 96.6%.

The trials in 1994-95 were with three alternative use patterns: (a) four applications, at pre-bloom, 75-95% petal fall, first cover (21-day interval) and second cover (21-day interval); (b) four applications as in (a) followed by three further applications at 21-day intervals; (c) two applications, at pre-bloom and 75-95% petal fall. Whole kiwifruit were analysed by HPLC with a limit of detection of 0.01 mg/kg, with HPLC-MS confirmation (Deakyne *et al.*, 1995).

Only a few of the trials were in accordance with current New Zealand GAP. The residues in these ranged from 0.05 to 0.22 mg/kg.

Two trials were conducted in the USA in 1995 (Deakyne, 1996). Four applications of the 70WP formulation were made at either 0.15 or 0.30 kg ai/ha, giving a total treatment of 0.60 or 1.20 kg ai/ha. The air-blast applications were at intervals of 6 to 14 days to plots of 0.50-0.54 ha. Single samples were taken 90 days after the last application. Whole fruit was analysed by the method of Deakyne *et al.* (1995). The residues were all below 0.5 mg/kg, even from the double rate.

The results of the New Zealand and US trials are shown in Table 2.

Table 2. Residues of tebufenozide in kiwifruit. Underlined rsidues are from treatments according to GAP.

Country, Location, Year	Form.	Application			PHI, days	Residue, mg/kg	Reference
		No.	kg ai/ha	kg ai/hl			
New Zealand	SC	4	a	0.006	10	0.77	Tillman, 1995
1990-1991					21	0.86	
					52	0.21	
					115	<u>0.22</u>	
New Zealand	SC	4	a	0.012	10	1.69	Tillman, 1995
1990-1991					21	1.55	
					52	0.55	
					115	0.34	
New Zealand	SC	8	0.092-0.10	0.004	4	0.69	Tillman, 1995
1992					7	0.57	
					14	0.65	
					21	0.44	
					28	0.22	
					42	0.41	
New Zealand	SC	8	0.138-0.15	0.006	4	0.85	Tillman, 1995
1992					7	0.82	
					14	0.94	
					21	0.92	
					28	0.63	
					42	0.65	
New Zealand	SC	8	0.277-0.30	0.012	4	2.5	Tillman, 1995
1992					7	2.6	
					14	2.28	
					21	1.77	
					28	1.5	
					42	1.3	
New Zealand	WP	3	b	0.004	21	0.1	Tillman, 1995
1992					42	0.06	
					72	0.01	
					98	0.02	
					127	0.01	
					147	0.03	
New Zealand	WP	3	b	0.006	21	0.18	Tillman, 1995
1992					42	0.16	
					72	0.06	
					98	0.05	
					127	<u>0.08</u>	
					147	0.08	
New Zealand	WP	3	b	0.012	21	0.37	Tillman, 1995
1992					42	0.25	
					72	0.1	
					98	0.02	
					127	0.11	
					147	0.07	
New Zealand	WDG	10	0.084-0.108	0.006	7	0.75	Tillman, 1995
1993					14	0.6	

Country, Location, Year	Form.	Application			PHI, days	Residue, mg/kg	Reference
		No.	kg ai/ha	kg ai/hl			
					21	0.62	
					28	0.58	
					35	0.44	
					42	0.48	
New Zealand	WDG	10	0.168-0.216	0.012	7	1.5	Tillman, 1995
1993					14	1.4	
					21	2.1	
					28	1.2	
					35	1.3	
					42	1.5	
New Zealand	WP	4	c	0.003	1	0.28	Tillman, 1995
1994-1995					7	0.39	
					14	0.19	
					28	0.16	
					107	0.03	
New Zealand	WP	4	c	0.006	1	0.65	Tillman, 1995
1994-1995					7	0.82	
					14	0.4	
					28	0.32	
					107	0.08	
New Zealand	WP	4	c	0.012	1	1.1	Tillman, 1995
1994-1995					7	1.5	
					14	1.1	
					28	1.1	
					107	0.18	
New Zealand	WP	7	c	0.006	1	0.39	Tillman, 1995
1995					7	0.42	
					14	0.33	
					28	0.29	
New Zealand	WP	7	c	0.012	31	0.62	Tillman, 1995
1995							
New Zealand	WP	2	c	0.006	154	0.05	Tillman, 1995
1994							
New Zealand	WP	4	c	0.003	0	0.46	Tillman, 1995
1994/95					7	0.3	
					14	0.26	
					28	0.19	
					122	0.06	
New Zealand	WP	4	c	0.006	0	0.94	Tillman, 1995
1994-1995					7	0.63	
					14	0.93	
					28	0.72	
					122	0.19	
New Zealand	WP	4	c	0.012	0	1.7	Tillman, 1995
1994-1995					7	1.6	
					14	1.1	
					28	0.47	
					122	0.4	
New Zealand	WP	7	c	0.006	0	0.92	Tillman, 1995
1995					8	0.82	
					14	0.84	
					28	0.58	
New Zealand	WP	7	c	0.012	28	1.3	Tillman, 1995
1995							
New Zealand	WP	2	c	0.006	163	0.04	Tillman, 1995
1994							

Country, Location, Year	Form.	Application			PHI, days	Residue, mg/kg	Reference
		No.	kg ai/ha	kg ai/hl			
USA, CA. 1995	WP	4	0.150	0.010-0.011	90	0.15	Deakyne, 1996
	WP	4	0.300	0.020-0.022	90	0.49	
USA, CA. 1995	WP	4	0.150	0.010-0.011	90	0.09	Deakyne, 1996
	WP	4	0.300	0.019-0.021	90	0.19	

a Each vine received approximately 5-7 litres of spray solution at each application, applied to the point of runoff.
b Each vine received approximately 5 litres of spray solution at each application.
c Each vine received approximately 6 litres of spray solution at each application.

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

No information was provided.

NATIONAL MAXIMUM RESIDUE LIMITS

The national MRL for kiwifruit in New Zealand was reported to be 0.5 mg/kg.

APPRAISAL

Tebufenozide was first evaluated by the 1996 JMPR, which recommended MRLs for grapes, pome fruits, husked rice and walnuts. Trials on kiwifruit could not be related to GAP in New Zealand and no maximum residue level could be estimated.

The New Zealand Government and the manufacturer requested the JMPR to re-evaluate the residue data on kiwifruit in the light of revised New Zealand GAP, in which the PHI has been increased from 21 to 90 days.

The residues in the trials reported in 1996 which reflect the revised New Zealand GAP (median underlined) were 0.05, 0.08, 0.19 and 0.22 mg/kg.

In two trials in the USA with 4 applications at 0.15 kg ai/ha and a 90-day PHI, the residues were 0.09 and 0.15 mg/kg. Although these results cannot be related to the reported GAP, they can be considered as supplementary supporting information.

The Meeting concluded that although the data were limited they were just sufficient to estimate a maximum residue level of 0.5 mg/kg and an STMR of 0.14 mg/kg for kiwifruit.

RECOMMENDATIONS

On the basis of data from supervised trials the Meeting concluded that the residue level listed below is suitable for establishing a maximum residue limit and the supervised trials median residue is suitable for use in dietary intake estimations.

Definition of the residue for compliance with MRLs and for the estimation of dietary intake: tebufenozide

Tebufenozide is fat-soluble

Commodity		MRL, mg/kg		PHI, days	STMR,mg/kg
CCN	Name	New	Previous		
FI 0341	Kiwifruit	0.5	-	90	0.14

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THIABENDAZOLE (65)

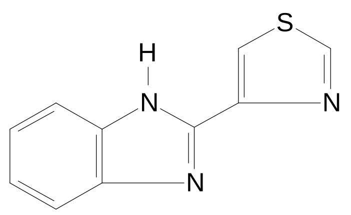
EXPLANATION

Thiabendazole was evaluated by the Joint Meeting in 1970, 1971, 1972, 1975, 1977, 1979, and 1981. MRLs were recommended for apple, banana, cereal grains, citrus fruits, edible offals, meat, milks, bulb onions, pear, potato, strawberry, sugar beet, sugar beet leaves, molasses and dried pulp, and tomato, and have now been adopted as CXLs. JECFA reviewed thiabendazole in 1992 and recommended MRLs of 0.1 mg/kg for all edible tissues of cattle, goats, sheep and pigs as well as for milk of cows and goats. The compound is now reviewed under the CCPR periodic review programme on the basis of a submission by the basic manufacturer and information provided by Codex member countries.

IDENTITY

ISO common name: thiabendazole
Chemical name
IUPAC: 2-(thiazol-4-yl)benzimidazole; 2-(1,3-thiazol-4-yl)benzimidazole
CA: 2-(4-thiazolyl)-1*H*-benzimidazole
CAS No: 148-79-8
Synonyms: Bovizole, Eprofil, Equizole, Lombristop, Mertect, Mintezol, Minzolum, Nemapan, Omnizole, Polival, Thiaben, Thibenzole

Structural formula:



Molecular formula: $C_{10}H_7N_3S$
Molecular weight: 201.26

Physical and chemical properties

Pure active ingredient

Vapour pressure:	4 x 10^{-9} mm Hg (Torr) at 25 ^oC
Melting point:	304-305 ^oC
Octanol/water Partition coefficient:	log P_{ow} = 2.2 (pH 5); 2.4 (pH 9)
Solubility, mg/ml:	water 38 at pH 2; 0.03 at pH >5
	methanol 8.7
	acetone 2.9
	ethyl acetate 2.1

Hydrolysis:	stable to strong acids and bases
Photolysis:	decomposes to benzimidazole and benzimidazole-2-carboxamide in strong UV light (half-life = 29 h in water)
Chelation:	forms stable complexes with a number of metals including iron. It does not bind calcium.

Technical material

Purity:	≥98% w/w
Melting range	296-303°C
Stability:	stable to acid or base hydrolysis and heat; unstable in strong UV light

Formulations

Thiabendazole is marketed in the following formulations:

SC: 1 g/l, 50 g/l, 100 g/l. 220 g/l, 300 g/l, 450 g/l, 485 g/l
SL: 220 g/l
TC: 985 g/kg
WP: 400 g/kg, 600 g/kg, 900 g/kg
WG: 890 g/kg

In combinations:

100 g/l+300 g tecnazene/l SL
300 g/l +100 g imazalil/l SC

METABOLISM AND ENVIRONMENTAL FATE

Farm animal metabolism

Three lactating goats (*Capra hircus*, ~1 year old, 45-60 kg each, healthy) were dosed with single gelatine capsules containing 120 mg and 564 µCi of [^{14}C]thiabendazole daily for 7 consecutive days. Two goats served as controls. The goats were slaughtered on the 8th day, within 24 hours after the final dosing. Milk was collected twice daily and tissue samples after slaughter (Chukwudebe *et al*., 1994; Halls *et al*., 1991b). An average of 74% of the administered dose was accounted for at the end of the study in the excreta (urine + faeces), tissues and milk. Nearly all the recovered radioactivity was in the urine (69%) and faeces (28%). The total residues in the excreta, tissues and milk are shown in Table 1.

The proposed metabolic pathways in goats are shown in Figure 1. As in previous studies (Rosenblum, 1965; Rosenblum *et al*., 1964) the tissue residues consisted of low levels of unmetabolized thiabendazole, unconjugated 5-hydroxythiabendazole, and benzimidazole at maximum concentrations of 0.2, 0.12 and 0.08 mg/kg as thiabendazole respectively, all in the liver. The residues in milk reached a plateau (1.13 mg/kg) on the 3rd day, with a maximum concentration of 1.24 mg/kg on day 5. The major individual residue (about 39% or 0.4 mg/kg) in milk was the *O*-sulfate conjugate of 5-hydroxythiabendazole. No other individual residue was detectable (i.e. [0.5% of the total radioactivity). Earlier fractionation studies in animal substrates (Rosenblum, 1965, Rosenblum *et al*., 1964) indicated that the unidentified residues were mainly products arising from extensive degradation of thiabendazole followed by incorporation into proteins (20-60%), lipids (12-14%) and polysaccharides (~1%). In the urine the residues consisted of unconjugated 5-hydroxythiabendazole (~7.9 mg/kg) and its *O*-sulfate conjugate (~9.5 mg/kg. The residues in faeces consisted of unconjugated 5-hydroxythiabendazole (2.1 mg/kg), together with lower levels of benzimidazole (~0.4 mg/kg) and unmetabolized thiabendazole (~0.3 mg/kg).

Table 1. Distribution of thiabendazole residues in the urine, milk, faeces and tissues of goats[1].

Sample	Total radioactive residues, mg/kg as thiabendazole		
	Average of 3 goats	Minimum	Maximum
Urine[2]	40.2		
Faeces[2]	24.3		
Liver	4.8	3.7	6.2
Kidney	1.4	1.3	1.5
Milk	1.0[4]	0.49[3]	1.24[3]
Gall bladder contents	0.85	0.37	1.49
Heart	0.22	0.19	0.24
Blood	0.19	0.17	0.21
Muscles[5]	0.10	0.08	0.12
Fat[6]	0.03	0.01	0.05

[1]Total residues in tissues were determined ~24 hr after end of dosing;
[2]Daily average of total residues
[3]Average from 3 treated goats. The minimum and maximum values were observed during the 1st and 5th day of feeding
[4]Average of 7 days
[5]Composite of semimembranosus, triceps and longissimus dorsi muscles 1:1:1 w:w:w.
[6]Composite of perirenal and omental fat 1:1 w:w

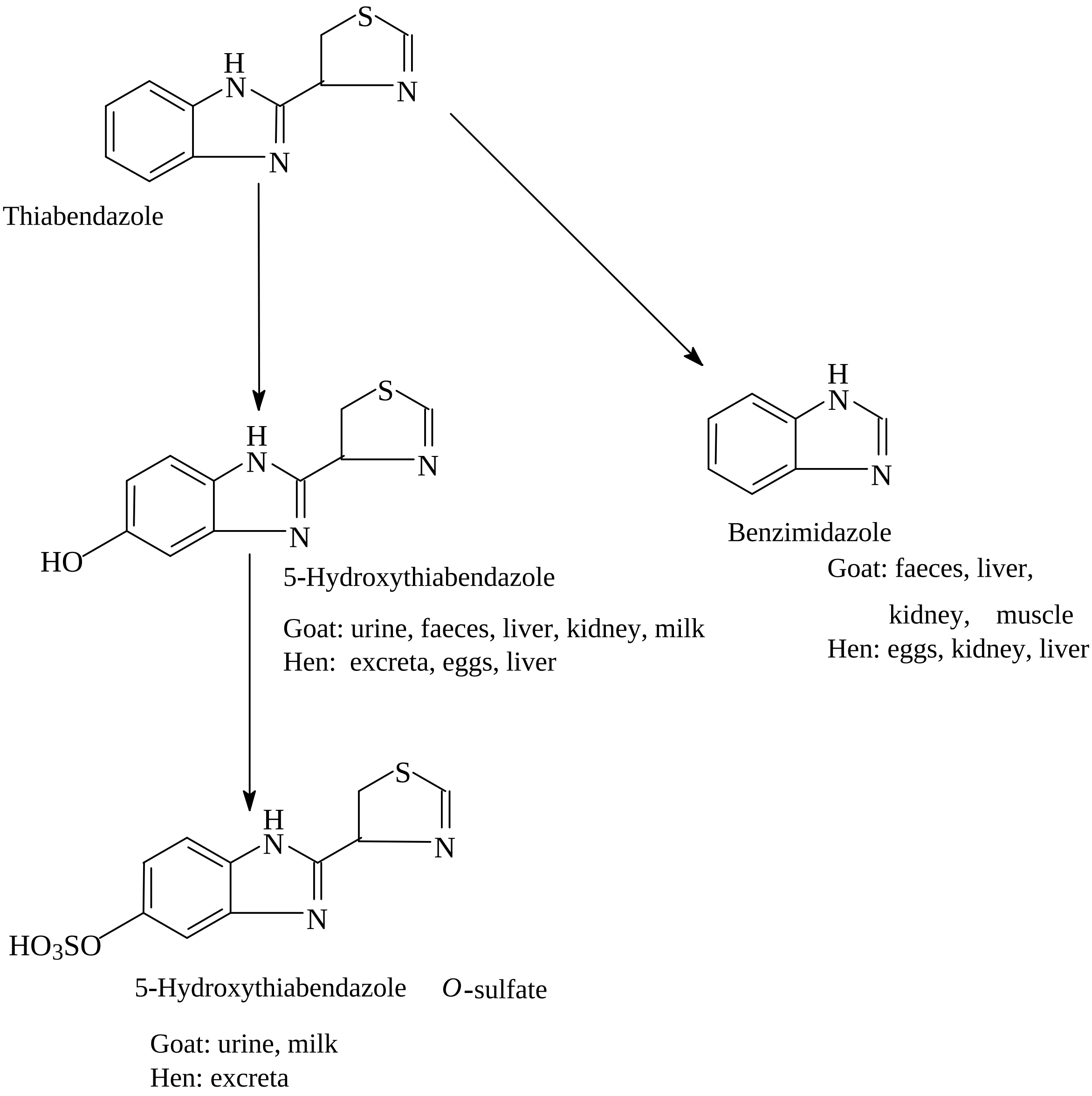
Five groups of four laying hens (*Gallus domesticus*, ~26 weeks old, 1-2 kg each, healthy) were dosed orally with single gelatine capsules, each containing 3.19 mg and ~25.2 µCi of [^{14}C]thiabendazole, daily for 10 consecutive days with one control for each group. Eggs and excreta were collected twice and once daily respectively. The hens were slaughtered and tissue samples collected for analysis on the 11th day, within 24 hours after the final dosing (Chukwudebe *et al*., 1994; Halls *et al*., 1991a). Concentrations of the total residues in the excreta, eggs and tissues are shown in Table 2. The total residues in the excreta and eggs were determined daily.

An average of 96.6% of the total administered dose was recovered, 99.6% of which was found in the excreta. Cumulatively, the total residues found in the tissues and eggs accounted for about 0.4%, or less, of the administered ^{14}C. The total residues in eggs reached a plateau of about 0.1 mg/kg as thiabendazole by day 2 and remained at about that level throughout the next 8 days of dosing.

The residues in the excreta consisted of 3.4 mg/kg of unconjugated and 4.4 mg/kg of conjugated 5-hydroxythiabendazole. The residues in the tissues and eggs were qualitatively similar and consisted mostly of unconjugated 5-hydroxythiabendazole, unmetabolized thiabendazole, and benzimidazole at maximum concentrations of 0.4, 0.11 and 0.12 mg/kg thiabendazole equivalents respectively, all in the kidneys.

None of these residues are likely to persist in the tissues or eggs in view of the low concentrations present and their rapid elimination. The proposed metabolic pathways in poultry are the same as in goats, and are also shown in Figure 1.

Figure 1. Proposed metabolic pathways of thiabendazole in dairy goats and laying hens.



These results are consistent with those reported previously in the literature on the metabolism of thiabendazole in sheep, cattle, dogs, pigs and humans. The oral administration of thiabendazole to sheep (Tocco *et al*., 1964), cattle, goats (Tocco *et al*., 1965), dogs and humans (Tocco *et al*., 1966) resulted in rapid absorption from the gastrointestinal tract. The time taken to achieve peak plasma levels varied with species and ranged from about 1 hour after dosing in dogs and humans to 7 hours in sheep, goats and cattle. In dogs, goats and cattle approximately 82% of the dose was excreted in the urine and faeces within the first 72 hours. Excretion in humans was more rapid, with approximately 80% being found in the urine within the first 24 hours. In all the species studied, hydroxylation of the benzimidazole ring to form 5-hydroxythiabendazole and subsequent conjugation to the glucuronide and sulfate were the major metabolic steps. These conjugates accounted for 70- 95% of the urinary metabolites in sheep, goats and pigs, 23% in dogs and 38% in humans. At peak plasma levels, which occurred about 6 h after dosing with 50 mg/kg of either [^{14}C] or [^{35}S]thiabendazole, the total radioactive residues retained in lamb tissues were relatively low (e.g. 2 mg/kg in muscle). By 5 days residues were undetectable ($\leq$0.06 mg/kg) in nearly all of the lamb tissues (Tocco *et al*., 1964). The total radioactive residues in calf, pig and goat tissues were also low by 24 h after dosing (e.g. 0.90 mg/kg in goat small intestines) and very low ($\leq$0.08 mg/kg) in most of the tissues by 17 days (Tocco *et al*., 1965). This rapid excretion in the urine and faeces, predominant metabolism via hydroxylation at the 5-position followed by glucuronidation or sulfation and low residue retention by the tissues has been confirmed by various investigators (e.g. Delatour and Parish, 1986; Lanusse and Prichard, 1993; McKellar and Scott, 1990; Prichard *et al*., 1981; Rosenblum, 1977; Weir and Bogan, 1985).

Table 2. Distribution of thiabendazole residues in hen excreta, eggs and tissues.

Sample	Total radioactive residues, mg/kg as thiabendazole		
	Average[1]	Minimum[2]	Maximum[2]
Excreta[3]	26.1[3]		
Liver	1.5	1.39	1.6
Kidney	1.2	1.17	1.25
Gizzard	0.3	0.25	0.34
Heart	0.3	0.31	0.34
Egg	0.1	0.13	0.18
Muscle, breast	0.07	0.06	0.76
Muscle, thigh	0.09	0.08	0.11
Fat[4]	0.02	0.013	0.022

[1]Averaged from 20 treated chickens
[2]Minima and maxima of average residues in the 4 chickens of each of the 5 groups
[3]Average of 10 days
[4]Composite from different parts of the body

Plant metabolism

The fate of [*phenyl*-^{14}C]thiabendazole was studied in wheat, soya beans and sugar beet treated at maximum recommended rates (Halls and Sanson, 1991a-c; Van den Heuvel *et al*., 1996). The results are shown in Tables 3 and 4, and the proposed metabolic pathways are shown in Figure 2.

Wheat. Actively growing wheat plants, at the 2-3 tiller stage, were sprayed once with [^{14}C]thiabendazole at a rate of about 0.80 kg ai/ha, representing the maximum recommended rate for wheat. Immature samples (foliage, forage and haylage) were taken after 2 hours, 7 days and 37 days respectively. At about 63 days after treatment, mature plants were harvested for samples of grain and straw. After extractions with acid, base and enzyme preparations, the extractable residues were characterized by a combination of GC-MS, reverse-phase HPLC and TLC.

The percentage of unextractable residue increased from about 1.8 in immature foliage to 16.8 in mature straw. Fractionation of the unextracted residues from straw by tissue solubilization followed by enzymatic and chemical hydrolyses demonstrated that most of the ^{14}C was distributed throughout several natural product fractions, including soluble polysaccharides, proteins, pectins, lignins and cellulose, but the level in any individual chromatographic fraction was less than 0.05 mg/kg as thiabendazole.

These results are consistent with findings from residue trials on wheat with unlabelled thiabendazole (Justin, 1985a,b, 1986) in which wheat seeds were treated with thiabendazole at rates up to 70% above the recommended maximum (1.4 g ai/litre of seeds) before planting. The growing wheat crops were also sprayed with thiabendazole at the maximum recommended field use rate (0.8 kg ai/ha) about 6-8 weeks before harvest. No thiabendazole (<0.05 mg/kg) was found in the grain harvested from the treated wheat.

Soya beans. The aerial portions of actively growing soya bean crops at the stage between late flowering and early pod set were sprayed twice, at a 14-day interval, with [^{14}C]thiabendazole at a combined rate of about 0.68 kg ai/ha, the maximum recommended rate on soya beans. Samples of foliage and forage were taken at intervals of 2 hours and 27 days after treatment and mature plants were harvested to provide seeds and straw about 78 days after the first spray. The extractable residues were characterized as above (Halls and Sanson, 1991b; Van den Heuvel *et al*., 1996).

Table 3. Distribution and characterization of ^{14}C-labelled thiabendazole residues in plants (percentage of total radioactive residues).

Plant, Fraction	^{14}C, % of TRR				
	Time after treatment & type of sample				
Wheat	2 hours foliage	7 days forage	37 days haylage	63 days	
				Straw	grain
Organo-extractable	97.2	79.3	46.3	60.1	41.5
Thiabendazole	97.2	79.3	36.8	33.1	23.2
Polars	ND[1]	0	3.2	19.1	18.3
Water-soluble	1.1	6.8	39.3	23.1	13.7
Polars	0	0	19.9	14.4	0
Benzimidazole related	0	0	23.1	33.5	18.3
Unextractable (tissue-associated)	1.8	14.0	11.7	16.8	17.5
Soya beans	2 hours foliage	27 day forage		78-day	
				straw	Seeds
Organo-extractable	93.3	60.8		47.3	63.2
TBZ	93.3	60.6		43.6	42.9
Polar	0	0		0	0
Aqueous soluble	1.4	35.5		41.4	33.3
Polar	0	1.4		7.3	0
BNZ-related	0	1.4		7.3	0
Unextractable (tissue associated)	5.4	3.8		11.2	1.0
Sugar beet	2 hours tops	56 days		90 days	
		tops	roots	tops	Roots
Organo-extractable	91.1	54.1	56.4	28.5	29.0
TBZ	91.0	52.2	55.6	27.1	25.8
Polar	0	<1	0	0	0
Aqueous soluble	2.1	39.2	43.3	60.4	65.0
Polar	0	11.5	6.8	14.1	10.8
BNZ related	0	11.5	6.8	14.1	10.8
Unextractable (tissue associated)	6.9	6.8	0.2	11.0	6.0

[1]Not determined

Sugar beet. Actively growing sugar beet plants were sprayed five times, at 14-day intervals, with [^{14}C]thiabendazole at a total application rate of 2.015 kg ai/ha, the maximum recommended rate for sugar beet. Immature treated and control tops and roots were taken at about 2 hours after the first and at last (56 days) treatments. At about 90 days after the initial treatment (35 days after the fifth and final treatment) mature plants were harvested and tops and roots were sampled. All samples were characterized by HPLC (Halls and Sanson, 1991c; Van den Heuvel *et al*., 1996).

Table 4. Thiabendazole residues in plants following foliar applications of [^{14}C]thiabendazole.

Crop, Sample	Application		DAT[2]	Residues, mg/kg[1]			
	kg ai/ha	No.		Total	TBZ[3]	BNZ[4]	Unextractable[5]
WHEAT	0.8	1					
Immature Foliage			0 (2 h)	67.46	65.57	<0.05	1.21
Immature Forage			7	41.20	32.67	<0.05	5.77
Immature Haylage			37	21.93	8.07	5.06	2.57
Straw			63	22.36	6.40	7.49	3.76
Grain			63	0.12	<0.05	<0.05	<0.05
SOYA BEANS	0.34	2					
Immature Foliage			0 (2 h)	14.32	13.36	<0.05	0.77
Immature Forage			27	25.45	15.12	0.36	0.97
Straw			78	10.15	4.22	0.74	1.14
Seed			78	0.88	0.38	<0.05	<0.05
SUGAR BEET	0.40	5					
Immature Tops			0 (2 h)	10.13	9.22	<0.05	0.70
Immature Tops			56	24.66	12.87	2.84	1.68
Immature Roots			56	0.86	0.48	0.06	<0.05
Mature Tops			90	10.01	2.43	1.41	1.10
Mature Roots			90	0.40	0.10	<0.05	<0.05

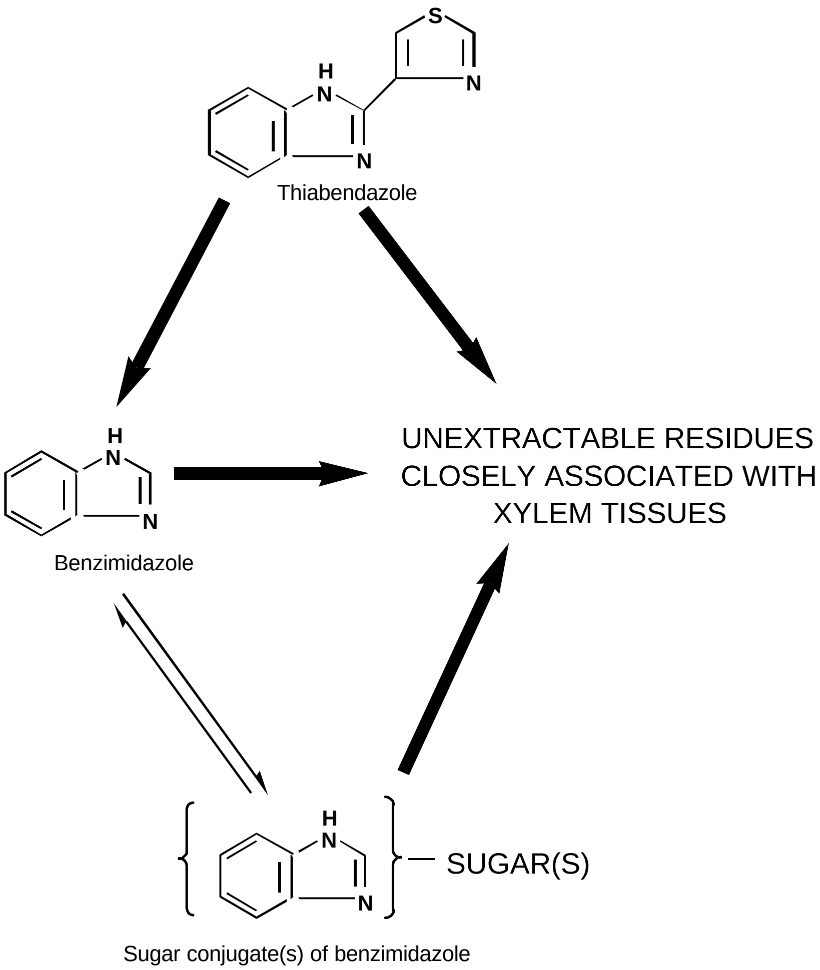
[1]Thiabendazole equivalents
[2]Days after first treatment
[3]Thiabendazole
[4]Sum of conjugated and unconjugated benzimidazole
[5]With KOH/MeOH reflux

The residue distributions in wheat, soya beans and sugar beet agree with other results showing the predominant movement of thiabendazole through plant axoplasm (Aharonson and Ben-Aziz, 1973; Allam *et al*., 1969; Ben-Aziz and Aharonson, 1974; Chatrath *et al*., 1972; Erwin *et al*., 1968, 1970, 1971; Gray and Sinclair, 1971; Hide and Cayley, 1977; Lyda and Burnett, 1970; Wang *et al*., 1971). This axoplasmic movement results in measurable levels of thiabendazole residues in shoot tissues (stems, leaves, straw) and relatively less in storage tissues (grain, seeds, roots).

Seed potatoes. The uptake, distribution and metabolism of thiabendazole by King Edward seed potatoes were examined under post-harvest storage conditions by Tisdale and Lord (1973). The potatoes were briefly immersed in solutions of [^{14}C]thiabendazole (~9.94 µCi/mg) at concentrations of 50, 100, 200 and 500 mg/l. Skin and tissue sections were subsequently processed and analysed. Metabolism and penetration were also studied by applying a pH 3 solution of [^{14}C]thiabendazole (196 mg/l) to cylindrical glass rings anchored to the surface of washed tubers by a thin layer of grease for periods of 5 and 60 minutes. The tubers were sectioned and autoradiographed at intervals of 2, 10, 21, 45, 75 and 120 days after treatment. The remaining tubers were subjected to multiple extractions with acidified methanol or sodium acetate-HCl buffer, followed by centrifugation and neutralisation of the supernatant solutions with base. The residues were partitioned into ethyl acetate, back-extracted into acid and then characterized by a combination of TLC, LSC, UV spectrometry and mass spectrometry.

Potato tubers absorbed thiabendazole from aqueous solutions at all pH levels examined (2-9) within 5 minutes. Thiabendazole penetrated only about 2 mm into the tubers in 2 weeks and a little further after 12 weeks. Autoradiography demonstrated that most of the thiabendazole (~96%) remained in the outer skin of the tubers; this is consistent with the high partition coefficient of thiabendazole between skins and water (27.7) and the coefficient of only 1.0 between the underlying tissues and water. The sorption of residues to the tubers was directly proportional to the concentration of applied thiabendazole; at the highest applied concentration of 500 mg/l ≥96% of thiabendazole in the tubers remained sorbed to the outer skin with negligible movement into the fleshy internal tissues. Even after 120 days of post-harvest storage only one radioactive component was detected, accounting for over 80% of applied [^{14}C]thiabendazole. The mass spectral characteristics of this radioactive component (M^+ 201; base peak 174, $(M\text{-}HCN)^{+\cdot}$ were identical to those of an authentic thiabendazole standard and demonstrated that the only residue in the potato tuber was thiabendazole.

Figure 2. Fate of thiabendazole in plants following foliar applications.



Post-harvest applications of [^{14}C]thiabendazole to stored potatoes did not result in detectable metabolic transformations (Tisdale and Lord, 1973). The major residual component was parent thiabendazole; benzimidazole was not detected ([0.05 mg/kg). Over 90% of the residue was confined to the potato skins and none was detected in the fleshy inner tissues. This is consistent with the results of other post-harvest studies with unlabelled thiabendazole (Briggs, 1981; Cayley *et al*., 1979; Eckert, 1970; Friar and Reynolds, 1991; Griffith and Hide, 1976; Hide and Cayley, 1977, 1983, 1989; Justin and Johnson, 1993; Lentza-Rizos, 1986). No evidence of thiabendazole metabolism was found even after 4 months storage under conditions favourable for metabolism. Even with adhering soil on the surface of stored tubers, the stability of thiabendazole in soil indicates that the only residual product will be thiabendazole itself (Daly and Williams, 1990, 1991).

In another study with unlabelled thiabendazole, over 90% of the thiabendazole residue, determined by bioassay, was found in the outer peel of post-harvest-treated pears, with negligible penetration into the inner fruit (Ben-Arie, 1975).

Oranges. The uptake, distribution and fate of [C]thiabendazole (specific activity ~0.4 μCi/mg; ≥99% pure) in oranges under typical post-harvest storage conditions were examined in Valencia oranges (Rosenblum and Meriwether, 1970). Oranges were treated in a 1-litre cylinder with 0.1% [^{14}C]thiabendazole as an aqueous wettable powder suspension. The treated oranges were shaken free of excess liquid, allowed to air-dry for 2 hours and stored under simulated commercial conditions for 28 days at temperatures of 10 ± 1°C and 21 ± 1°C. At specified intervals, samples were separated into whole peel, inner peel and fleshy pulp. The uptake and distribution of ^{14}C in these separate fractions were determined by oxidative combustion, LSC and reverse isotope dilution assay.

Irrespective of storage duration and temperature, radioactivity was taken up by the treated oranges; virtually all (~95%) of the ^{14}C was sorbed to the peel and none penetrated into the fleshy inner pulp. Assays of the orange samples over the 28-day storage period demonstrated that about 95% of the radioactivity consisted of parent thiabendazole itself in spite of the duration and conditions (28 days, 21°C) being favourable for metabolism.

Rotational crops. Thiabendazole products are used mainly for post-harvest treatments (e.g. on stored potatoes, citrus, pome fruits and bananas) where outdoor applications to agricultural soil are not involved. There are, however, some uses of thiabendazole (e.g. as cereal seed dressing, pre-plant treatment of seed potatoes and limited foliar applications) from which relatively low residues could remain in soil and potentially be transferred to rotational crops. A confined accumulation study with [^{14}C]thiabendazole applied to soil at the maximum anticipated use rates was therefore conducted on rotational crops (Halls and Sanson, 1992). The study represents a worst-case situation for the uptake of residues that might be encountered during these relatively minor commercial uses of thiabendazole. Three outdoor plots of 235 x 82.5 cm containing sandy loam soil were sprayed with [^{14}C]thiabendazole once, or twice two weeks apart, at a total application rate of 2.15 kg ai/ha. The rotational crops were planted at different times after the last spray treatment, to represent the scenarios of crop failure or premature sowing (planting 30 days after the 2nd application) and normal agronomic practice (planting after 120 and 320 days, i.e. about 6 months and one year).

Three representative crops, wheat (small grain), turnips (root crop) and lettuce (leafy vegetable) were planted in each plot. At culturally and agronomically appropriate harvest intervals, the crops were sampled for the determination of total residues. Quantitatively significant components of the residues (≥10% of the total in the sample) were also characterized. Immediately after the last thiabendazole treatments, and at appropriate harvest intervals during the study, soil cores (0 to 30 cm depth) were taken for the determination of total residues. The total residues in the soil and crop samples were quantified by oxidative combustion followed by LSC. Residues were characterized, after acid, base or enzymatic hydrolytic extractions, by a combination of GC-MS, reverse-phase HPLC and TLC radiochromatographic analyses.

Residue levels in the top 15-cm layers of the three plots were about 0.8-1.1 mg/kg and were quantitatively consistent with a total thiabendazole application rate of 2.15 kg ai/ha (Table 5). In general, no significant residues ([0.1 mg/kg) were detected below the 15-cm soil depth. The results demonstrate that neither thiabendazole nor any of its soil degradation products has the propensity to leach into ground water. About 75-94% of the total soil residue was extractable and most of it (~63-94%) was thiabendazole (Table 6.). This picture agrees with the results of previous studies of the degradation of thiabendazole in soil (Daly and Williams, 1990, 1991).

The major components of the rotational crop residues were thiabendazole and benzimidazole and its sugar conjugate(s). Lower levels of 5-hydroxythiabendazole (maximum 25-30% of the thiabendazole residue) were also observed in some plant samples. Since 5-hydroxythiabendazole is produced by degradation in soil but not in plants, it is reasonable to conclude that the 5-hydroxythiabendazole was soil-generated and subsequently taken up by the crops through axoplasmic mechanisms in the same way as thiabendazole (e.g. Gray and Sinclair, 1971; Wang *et al*., 1971). In addition to thiabendazole, benzimidazole, 5-hydroxythiabendazole and the unextractable residues, other radioactive components were observed in the HPLC radiochromatograms of various crop extracts, but all were individually at levels below 0.05 mg/kg.

Table 5. Residues of thiabendazole in soil plots planted with rotational crops.

Sample	Total residue, mg/kg as thiabendazole	Extractable, % of total	Thiabendazole, % of total
Plot A planted after 30 days			
2 h after treatment	0.79	93.8	93.8
137 days after treatment	0.98	75.3	69.6
Plot B planted after 120 days			
2 h after treatment	1.07	89.0	89.0
223 days after treatment	0.76	88.6	86.9
Plot C planted after 320 days			
2 h after treatment	0.95		
398 days after treatment	0.95	78.1	63.2

Table 6. Nature of the residues in rotated crops grown in thiabendazole treated soil.

Sample	DAT[1]	Residues, mg/kg as thiabendazole				
		Total	Thiabendazole	Total benzimidazole[2]	5-OH-TBZ[3]	Unextractable
IMMATURE LETTUCE						
30-Day Plot	75	0.37	0.07	0.05	<0.05	0.05
120-Day Plot	153	0.66	0.23	0.15	0.05	0.09
320-day Plot	357	1.56	0.29	0.81	0.10	0.16
MATURE LETTUCE						
30-Day Plot	95	0.66	0.23	0.03	<0.05	0.12
120-Day Plot	174	0.27	0.05	0.09	<0.05	<0.05
320-Day Plot	372	0.51	0.08	0.32	<0.05	<0.05
IMMATURE TURNIP TOPS						
30-Day Plot	56	0.10	0.04	<0.05	<0.05	<0.05
120-Day Plot	153	0.85	0.42	0.22	<0.05	<0.05
320-Day Plot	357	0.34	0.05	0.12	<0.05	<0.05
MATURE TURNIP TOPS						
30-Day Plot	95	0.63	0.22	0.09	<0.05	0.07
120-Day Plot	180	0.77	0.32	0.05	<0.05	0.05
320-Day Plot	398	1.05	0.11	0.43	0.05	0.06
MATURE TURNIP ROOTS						
30-Day Plot	95	0.15	0.08	<0.05	<0.05	<0.05
120-Day Plot	180	0.16	0.09	<0.05	<0.05	<0.05
320-Day Plot	398	0.15	0.11	<0.05	<0.05	<0.05

Sample	DAT[1]	Residues, mg/kg as thiabendazole				
		Total	Thiabendazole	Total benzimidazole[2]	5-OH-TBZ[3]	Unextractable
IMMATURE WHEAT FOLIAGE						
30-Day Plot	56	0.56	0.13	0.07	0.05	0.11
120-Day Plot	153	2.29	0.66	0.49	0.18	0.25
320-Day Plot	357	1.23	0.58	0.31	<0.05	0.11
Wheat Straw						
30-Day Plot	137	6.79	2.52	2.12	0.15	0.07
120-Day Plot	223	2.61	0.89	0.80	<0.05	<0.05
320-Day Plot	408	10.25	2.55	2.49	0.70	0.49
WHEAT HULLS						
30-Day Plot	137	4.65	2.42	0.57	<0.05	<0.05
120-Day Plot	223	1.13	0.64	<0.05	<0.05	0.08
320-Day Plot	408	6.58	2.01	1.87	<0.05	0.14
WHEAT GRAIN						
30-Day Plot	137	0.09	0.05	<0.05	<0.05	<0.05
120-Day Plot	223	0.05	<0.05	<0.05	<0.05	<0.05
320-Day Plot	408	0.18	0.09	<0.05	<0.05	<0.05

[1]Days after final treatment with [^{14}C]thiabendazole
[2]Sum of conjugated and unconjugated benzimidazole
[3]5-hydroxythiabendazole

Environmental fate in soil

Degradation. The fate of thiabendazole in microbially active sandy loam soil was studied under aerobic conditions at 25 ± 1°C (Daly and Williams, 1991). Thiabendazole was degraded with an aerobic half-life approximating 737 days, consistent with results from a similar study by Aharonson and Kafkafi (1975). Degradation products consisted of low levels of benzimidazole (<2.5%) and 5-hydroxythiabendazole (<0.5%). The proposed degradation pathways are shown in Figure 3. Unextractable radiocarbon increased slowly during the study, ranging from 1.24% at day 0 to 20.20% at day 120. This increase, despite the use of hydrolytic extractants such as HCl and KOH, is consistent with the strong binding of thiabendazole to soil (Aharonson and Kafkafi, 1975; Cayley and Lord, 1980; Dykes, 1989). Volatile material, 96% of which was $^{14}CO_2$, also increased slowly, attaining its highest level after 12 months and accounting for 5.8% of the applied radioactivity. These results indicate that thiabendazole, despite its relative stability in soil, would eventually be mineralized to CO_2.

Thiabendazole was stable in soil (half-life >737 days) under anaerobic conditions (Daly and Williams, 1990), but its levels in soil decreased during the preliminary aerobic phase of the study, before the attainment of anaerobic conditions, with a calculated half-life of about 211 days. Unextractable residues and $^{14}CO_2$ attained levels of 5.8 and 0.8% respectively of the applied radioactivity. The only degradation product detected was benzimidazole at a maximum of 5.9%, formed mainly during the preliminary aerobic phase of the study.

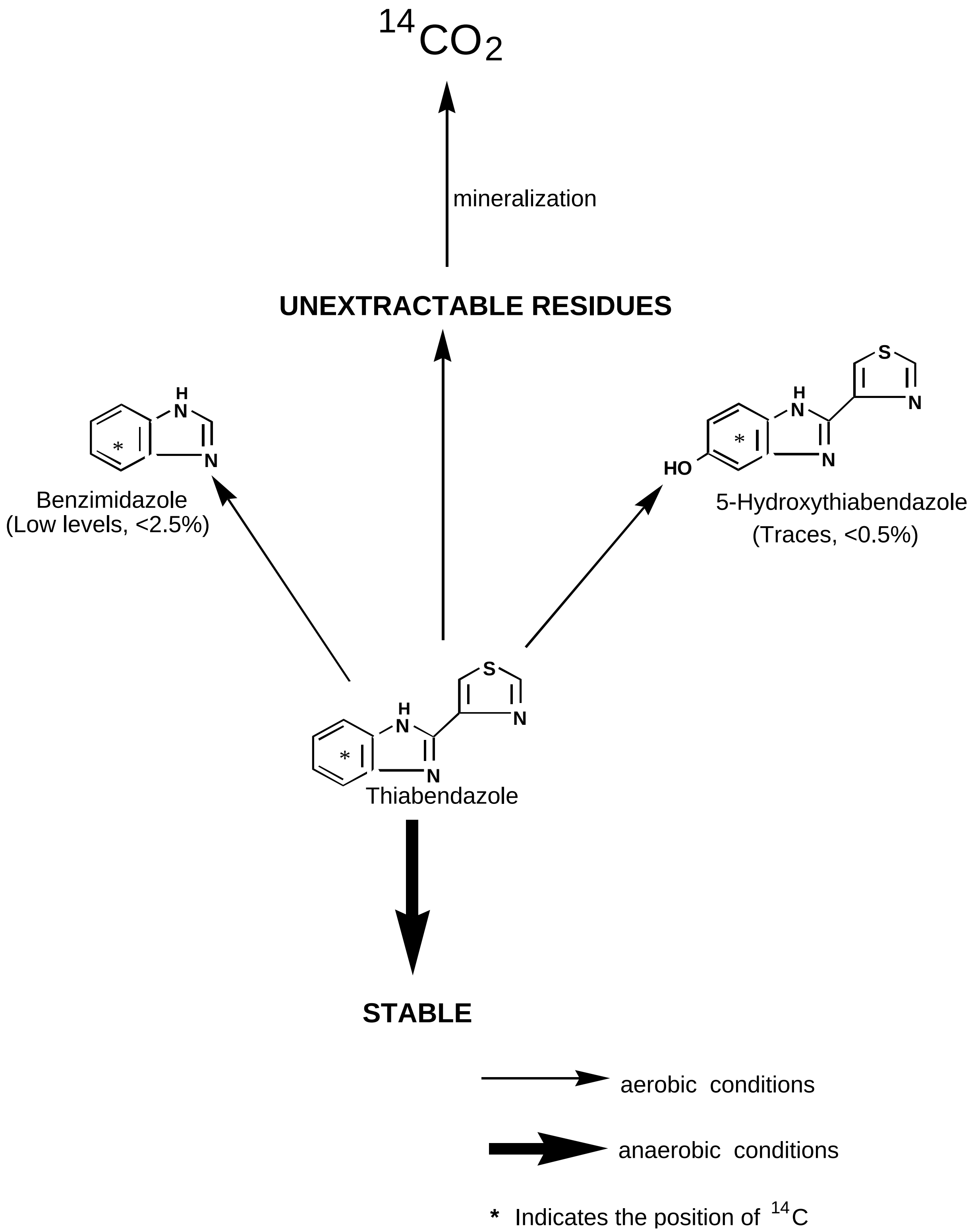
Photolysis. The degradation of [^{14}C]thiabendazole on sandy loam soil exposed to artificial sunlight was studied at 25 ± 2.5°C (Dykes and Kabler, 1990). Thiabendazole was found to be photolytically stable with a calculated half-life of 933 days. Recoveries of ^{14}C from exposed and unexposed soil samples averaged about 98 and 104% respectively, and nearly all of this radioactivity (90-100%) was due to [^{14}C]thiabendazole; no other residue was found.

Mobility. To quantify the sorption of [^{14}C]thiabendazole to soil, batch equilibrium studies were conducted with silt loam, clay, sandy loam and sandy soils (Dykes, 1989). The adsorption K_{OC} values ranged from 1,104 to 22,467, indicating that thiabendazole binds very tightly to soil, and desorption was correspondingly low with K_{OC} values from about 1,336 to 18,325. On the basis of the high K_{OC} values (>1,000) thiabendazole is considered to be immobile in soil.

Results from soil column leaching studies conducted by the WARF Institute (1976) also demonstrated that thiabendazole is immobile in soil under simulated conditions of either rapid (gravity-controlled) or slow (1.25 cm/day for 45 days) leaching. In another leaching study (Aharonson and Kafkafi, 1975), it was further demonstrated that thiabendazole is practically immobile in soil even under conditions simulating the passage of 1,000 mm of rain water through a soil column.

Results from leaching studies with aged residues (WARF Institute, 1978) demonstrated that both aerobically and photolytically aged thiabendazole residues are immobile in soil. In these studies, with about 10 physico-chemically different soils more than 98% of the recovered radioactive residues remained in the top 2.5-cm layer of the soils. The results of these batch equilibrium and leaching studies show that thiabendazole residues can be classified as being immobile in soil under both laboratory and field conditions. Pesticides that are immobile in soil are considered unlikely to leach into groundwater or travel in run-off water into streams and lakes (Kenaga, 1980).

Figure 3. Proposed degradation pathways of thiabendazole in soil.



Environmental fate in water

Photolysis. [^{14}C]Thiabendazole was shown to be degraded rapidly in water when exposed to artificial sunlight, with a half-life of approximately 29 hours (Flynn, 1994). The degradation resulted in the formation of benzimidazole-2-carboxamide (~10%), a polar fraction (8.6%) and benzimidazole (~6%). These residues were confirmed by MS. A minor degradation product with HPLC retention properties consistent with benzimidazolecarboxylic acid was also present in trace amounts. These products are not likely to have significant biological activities (Stone *et al*., 1965; Delatour and Parish, 1986). It can therefore be concluded that thiabendazole is degraded rapidly in the aquatic environment to non-toxic products: the only potential residue of concern to which non-target aquatic species might be exposed is the parent thiabendazole.

METHODS OF RESIDUE ANALYSIS

Analytical methods

The methods used for the quantification of thiabendazole and its metabolites in plant and animal commodities were validated by spiking the untreated commodity with known levels of thiabendazole and metabolites before solvent extraction. The limit of determination (LOD) is defined as the lowest fortification level that yields acceptable recoveries (>70%) of the analyte with a chromatographic signal to noise (S/N) level >10. The limit of detection is defined as the lowest concentration of analyte that can be detected with an S/N level >3.

Bananas, whole and pulp

Liquid chromatography. Extraction with ethyl acetate is followed by concentration and purification on a cation exchange solid-phase extraction (SPE) column. The purified extract is analysed for thiabendazole by liquid chromatography on a cation exchange column eluted with acetonitrile/phosphate buffer (30:70, pH 4) with fluorescence detection (excitation 305 nm, emission 380 nm). Recoveries of thiabendazole averaged 93% for whole bananas fortified with thiabendazole at 0.05-10 mg/kg and 95% for pulp fortified at 0.01-2 mg/kg, with an LOD and limit of detection in whole bananas of 0.05 and <0.01 mg/kg (Arenas & Johnson, 1994a).

Spectrofluorimetry. Extraction with ethyl acetate is followed by purification by a series of acid/base partitions. The purified extract in 0.1N HCl is analysed for thiabendazole by spectrofluorimetry. Recoveries were >85%, with an LOD and limit of detection in whole bananas of 0.05 and 0.01 mg/kg (Justin & Johnson, 1993a).

Chicory (Endive)

Spectrofluorimetry. An aqueous slurry of the sample homogenate is extracted with ethyl acetate and the extract purified by a series of acid/base partitions. The thiabendazole in the final 0.1N HCl solution is determined by fluorescence spectrofluorimetry with an excitation wavelength of 305 nm and an emission wavelength of 360 nm. The limit of detection is 0.005 mg/kg. (Johnson, 1994b).

Citrus fruit and by-products

Liquid chromatography. The method is identical to that for bananas. Recoveries of thiabendazole averaged 96% for citrus fortified at 0.05-20 mg/kg. The LOD and limit of detection were 0.05 and <0.01 mg/kg respectively (Arenas *et al*., 1996).

Spectrofluorimetry. The method is the same as for chicory. The limit of detection is 0.02 mg/kg for whole citrus fruit, 0.1 mg/kg for citrus oil, 0.02 mg/kg for citrus juice, 0.1 mg/kg for molasses and 0.3 mg/kg for dried citrus pulp (Justin & Johnson, 1992a,b).

Mushrooms

Spectrofluorimetry. As for chicory. The LOD and limit of detection for whole mushrooms are 0.1 and 0.05 mg/kg (Justin & Johnson, 1992c).

Pome fruit and by-products

Liquid chromatography. The homogenized sample is extracted with ethyl acetate after the addition of sodium sulfate and the crude extract is filtered. The filtrate is extracted with 0.1N HCl, the pH adjusted to 8, and the thiabendazole partitioned into ethyl acetate. The ethyl acetate is evaporated to dryness, the residue is dissolved in methanol, and the solution analysed by liquid chromatography on a C18 column eluted with methanol/water (60:40) containing 0.3% ammonia. The limit of detection is 0.05 mg/kg (Johnson, 1994a).

Spectrofluorimetry. As for chicory. The LOD is 0.05 mg/kg for whole fruit, 0.03 mg/kg for juice and 0.5 mg/kg for dried pomace. The limit of detection is 0.01 mg/kg for juice and 0.2 mg/kg for dried pomace (Justin & Johnson, 1992d,e).

Potato tubers

Liquid chromatography. The method is the same as that for chicory except that the buffer is 25:75 acetonitrile/phosphate, pH 3.4. Recoveries of thiabendazole averaged 100% for whole white potatoes at 0.05-20 mg/kg and 94% for sweet potatoes fortified at 0.005-0.1 mg/kg. The LODs were 0.05 and 0.005 mg/kg respectively. The limit of detection was 0.0025 mg/kg (Arenas *et al*., 1995).

Spectrofluorimetry. An aqueous slurry of the sample homogenate is hydrolysed on a steam bath with sulfuric and hydrochloric acids and the resulting mixture is digested overnight with diastase. The mixture is extracted with ethyl acetate and determination completed as with chicory. Recoveries of thiabendazole exceeded 85%. The LOD was 0.05 mg/kg for whole potatoes and 0.025 mg/kg for potato flakes, with limits of detection of 0.02 and 0.01 mg/kg respectively (Justin, 1993b).

Cereal seed, straw and processed commodities - wheat

Liquid chromatography, thiabendazole. The grain, straw or processed commodity is extracted with methanol and the residue remaining after filtration is extracted again with hot methanol/KOH. The extract is purified by a series of acid/base liquid-liquid partitions. The final solution is analysed by liquid chromatography on a C-18 column eluted with methanol/water (40:60) containing 0.1% ammonium acetate. Detection is by fluorescence (excitation 300 nm; emission 350 nm). Recoveries averaged 89% over the fortification range 0.05-2 mg/kg. The LOD was 0.05 mg/kg and the limit of detection 0.02 mg/kg. (Armstrong & Norton, 1993b).

Liquid chromatography, free and conjugated benzimidazole. The grain, straw or processed commodity is extracted with methanol and the residue remaining after filtration is extracted again with hot methanol/KOH. Concentrated HCl is added and the filtered solution is concentrated under vacuum. Beta-glucosidase is added to the residue, the pH adjusted to 5 and the solution incubated for two hours at 37°C. The solution is acidified and extracted with ethyl acetate. The aqueous solution is adjusted to pH 9, extracted 3 times with ethyl acetate and the combined extracts are evaporated to dryness. The residue is analysed for benzimidazole by liquid chromatography on a C-18 column eluted with methanol/water (25:75) containing 0.1% ammonium acetate. Detection is by fluorescence (excitation 260 nm; emission 300 nm). Recoveries averaged 84% over the fortification range 0.1-2 mg/kg, with an LOD of 0.1 and a limit of detection of 0.05 mg/kg (Fieser & Johnson, 1994a).

Fruits and vegetables

Liquid chromatography. The crop is homogenised with aqueous HCl, the filtrate adjusted to pH 8, the thiabendazole partitioned into ethyl acetate and the ethyl acetate evaporated to dryness. The residue is dissolved in methanol and the solution analysed by liquid chromatography on a C-18 column eluted with methanol/phosphate buffer (65:35), pH 8. Detection is by fluorescence. Recoveries of thiabendazole were >80%, with a limit of detection of 0.01 mg/kg (Johnson, 1994c).

Soil

Liquid chromatography. The method is used for the determination of thiabendazole and benzimidazole. The soil sample is extracted with methanolic KOH, then with dimethylformamide in HCl. The solution is extracted with ethyl acetate, the extract purified by a series of acid/base liquid-liquid partitions and the ethyl acetate evaporated to dryness. The residue is dissolved in aqueous acid and the solution is analysed by liquid chromatography, eluting with methanol/water (60:40) containing 0.1% ammonium acetate. Detection is by fluorescence (excitation 300 nm, emission 350 nm). Recoveries of thiabendazole and benzimidazole averaged 87% and 92% respectively at 0.01-1 mg/kg. The LOD was 0.01 mg/kg and the limit of detection 0.005 mg/kg (Fieser & Jacobson, 1994).

Water

Liquid chromatography. The method is used for the determination of thiabendazole in drinking water. The sample is passed through an “EMPORE” C-18 disk and the adsorbed thiabendazole is eluted with acetonitrile. The solution is analysed for thiabendazole by reverse-phase HPLC with fluorescence detection. The LOD is 0.0001 mg/kg (Johnson, 1996).

Feed

Liquid chromatography. The feed sample is extracted with methanol and the solution analysed for thiabendazole by liquid chromatography on a cation exchange column eluted with acetonitrile/phosphate buffer (25:75) at pH 3.4. Detection is by fluorescence (excitation 305 nm; emission 380 nm). Recoveries were >95%. The limit of detection is 5 mg/kg. (Cobin, 1994)

Animal tissues

Liquid chromatography. Thiabendazole and the animal metabolites 5-hydroxythiabendazole (5-OH-TBZ) and benzimidazole are released from the tissue by digesting with 6N HCl at 90-95°C for 24 hours. The solution is adjusted to pH 8, extracted with ethyl acetate and the extract purified on a cation exchange solid-phase extraction column. Quantification is by liquid chromatography on a cation exchange column eluted with acetonitrile/phosphate buffer (25:75) at pH 3.0-3.4 with fluorescence detection. Recoveries were >85%, with LODs and limits of detection of 0.1 and 0.005 mg/kg for each analyte (Arenas & Johnson, 1994b).

Spectrofluorimetry. The tissue is homogenised with phosphate buffer, pH 4.5, then incubated overnight with glusulase at 37°C. The solution is adjusted to pH 6.5, extracted with ethyl acetate and the extract purified by a series of acid/base partitions. Thiabendazole and 5-OH-TBZ are determined by fluorescence (thiabendazole, excitation 305 nm, emission 360 nm; 5-OH-TBZ, excitation 340, emission 420 nm). Recoveries were >85%, with LODs and limit of detection of 0.1 and 0.05 mg/kg respectively (Justin, 1990c).

Milk

Liquid chromatography. The sample containing residues of thiabendazole, 5-hydroxy thiabendazole, and its sulfate conjugate is heated with concentrated HCl for four hours at 85-90°C. The cooled solution is adjusted to pH 8, extracted with ethyl acetate and the extract purified on a cation exchange solid-phase extraction column. The solution is analysed for thiabendazole and 5-OH-TBZ by liquid chromatography on a cation exchange column eluted with acetonitrile/phosphate buffer (20:80) at pH 3.8, with fluorescence detection. Recoveries were >85%, with an LOD and limit of detection of 0.05 and 0.005 mg/kg respectively (Arenas & Johnson, 1995).

Spectrofluorimetry. The method is the same as that for animal tissues. Recoveries were >80%, with an LOD and limit of detection of 0.05 and 0.02 mg/kg respectively (Justin, 1990c).

Eggs

Liquid chromatography. The method is the same as for tissues. Recoveries were >85%. The LOD was 0.05 mg/kg and the limit of detection was 0.01 mg/kg for each analyte (Arenas & Johnson, 1994c).

Spectrofluorimetry. As for tissues. Recoveries were >85%, with an LOD and limit of detection of 0.1 and 0.05 mg/kg respectively (Justin, 1990b).

Stability of pesticide residues in stored analytical samples

The stability of residues was tested in apples, apple juice, apple pomace, bananas, citrus, mushrooms, potatoes, wheat, milk, eggs, and chicken liver, muscle and kidney. The homogenised samples were fortified with known amounts of residues and stored in sealed high-density polyethylene bottles in the dark at –20 ± 10°C. The residues measured at various intervals after fortification are shown in Table 7.

Definition of the residue

On the evidence of the 5 studies carried out with labelled thiabendazole and other related studies using unlabelled material the only detectable residue (≥0.05 mg/kg) in edible crop commodities is likely to be thiabendazole.

The animal metabolism studies and transfer studies with cows and poultry indicate that thiabendazole and 5-hydroxythiabendazole are the main residue components in meat and eggs, while the sulfate conjugate of 5-hydroxythiabendazole is the main component in milk.

The Meeting concluded that the following residue definitions are appropriate.

For compliance with MRLs

For plant products: thiabendazole.
For animal products: sum of thiabendazole and 5-hydroxythiabendazole.

For estimations of dietary intake

For plant products: thiabendazole.
For animal products: sum of thiabendazole, 5-hydroxythiabendazole and its sulfate conjugate.

Table 7. Stability of thiabendazole and metabolites in stored analytical samples.

Sample	Residues, mg/kg, after storage periods, months							Ref.
	0	1	2	3	6	9	12	
	TBZ	TBZ	TBZ	TBZ	TBZ	TBZ	TBZ	
Apple, whole	0.093			0.084		0.093		Norton, 1992c
Apple juice	0.083			0.097		0.094		
Dried pomace	0.85			0.84		0.77		
Banana, whole	0.49	0.49	0.48	0.48				Norton, 1993
Banana pulp	0.097	0.097	0.097	0.096				
Citrus, whole	0.42			0.47		0.48		Norton, 1992a
Citrus oil	2.0			1.9		2.1		
Molasses	1.8			1.8		1.8		
Dried peel	1.9			2.0		1.7		
	0	3	6	9	12	27	28	
	TBZ	TBZ	TBZ	TBZ	TBZ	TBZ	TBZ	
Mushroom	0.0175	0.024	0.02					Johnson, 1995
	0.094	0.10	0.089		0.097		0.097	
	47.2	50.1	51.7		50.6		49.9	
Potato, whole	0.20	0.20				0.22		Norton, 1995c
Potato peel	0.54	0.52				0.47		
	0	1	3	6	12	18	23	
	TBZ /BNZ	TBZ/ BNZ	TBZ/BNZ	TBZ/BNZ	TBZ/BNZ	TBZ/ BNZ	TBZ/BNZ	
Wheat grain	0.22/0.21		0.20/0.18	0.21/0.20		0.21/0.19	0.19/0.18	Armstrong and Norton, 1993b
Wheat bran	0.22/0.18	0.20/0.20	0.20/0.18	0.18/0.18	0.18/0.19	0.20/0.18		
Wheat flour	0.20/0.19	0.19/0.21	0.17/0.21	0.19/0.20	0.19/0.19	0.18/0.20		
Wheat straw	0.17/0.21	0.19/0.16	0.20/0.14	0.26/0.14	0.22/0.18	0.19/0.20	0.20/0.19	
	0			2				
	TBZ	5-OH-TBZ	5-$NaSO_4$-TBZ	TBZ	5-OH-TBZ	5-$NaSO_4$-TBZ		
Milk	0.38	0.30	0.14	0.36	0.34	0.16		Arenas,1994a
Egg yolk	0.084	0.47		0.095	0.47			Dahmen, 1990
Egg white	0.086	0.48		0.094	0.49			
Chicken liver	0.42	0.38		0.44	0.33			Arenas, 1994b
Chicken muscle	0.43	0.42		0.45	0.40			
Beef kidney	0.44	0.37		0.46	0.34			

TBZ: thiabendazole
BNZ: benzimidazole
5-OH-TBZ: 5-hydroxythiabendazole
5-$NaSO_4$-TBZ: sulfate conjugate of 5-hydroxythiabendazole

USE PATTERNS

The major registered or approved uses of thiabendazole on food crops are shown in Tables 8 and 9.

Table 8. Major approved post-harvest and pre-planting uses of thiabendazole.

Crop	Country	APPLICATION			
		Form., concn.	Method	g ai/100 l	Rate (ai)
Apples, pears	Argentina	SC, 450 G/L	Spray	50-90	0.90 g /t
	Australia	SC, 900G/KG	Dip	100	
	Canada	SC, 450 G/L	Dip, drench	45	
	France	SC, 450 G/L	Spray	45;	
		SC, 450 G/L	Dip	225	
	Italy	SC, 450 G/L	Dip	45 – 110	
	Mexico	WP, 600 G/KG	Dip, drench	200-850	
	Netherlands	SC, 450 G/L	Spray	135[a]	
	South Africa	SC, 450 G/L	Dip	100	
	Spain	SL, 220 G/L	Dip	110 – 130	
	USA	SC, 450 G/L	Dip, spray	61	
Avocado	South Africa	SC, 450 G/L	Dip	135	
Bananas	Argentina	SC, 450 G/L	Dip	20-40	
	Australia	SC, 900G/KG	Dip	20-40	
	Brazil	SC, 485 G/L	Dip, spray	20-45	
	Colombia	SC, 450 G/L SL, 220 G/L	Dip, spray Dip, spray	20-45 20-40	
	Costa Rica	SL, 220 G/L SC, 450 G/L	Dip, spray Dip, spray	20-40 20-40	
	Ecuador	SL, 220 G/L	Dip, spray	20-40	
	France	SL, 220 G/L	Dip, spray	45	
	French Antilles	SC, 450 G/L	Dip, spray	22-45	
	Guatemala	SC, 450 G/L SL, 220 G/L	Dip, spray Dip, spray	20-40 20-40	
	Honduras	SC, 450 G/L SL, 220 G/L	Dip, spray Dip, spray	20-40 20-40	
	Israel	SC, 450 G/L	Dip, spray	60	
	Mexico	WP, 600 G/KG	Dip, spray	40-80	
	Nicaragua	SC, 450 G/L SL, 220 G/L	Dip, spray Dip, spray	20-40 20-40	
	Panama	SC, 450 G/L SL, 220 G/L	Dip, spray Dip, spray	20-40 20-40	
	South Africa	SC, 450 G/L	Dip	20	
	USA	SC, 50 G/L	Dip	20	
		TC, 985 G/KG	Dip	20	
		SC, 450 G/L	Dip, spray	20	
Cabbages	Germany	SC, 450 G/L	Spray	68	0.034 g/100 kg
Carrots	USA	SC, 450 G/L	Dip	560	
Celery	Israel	SC, 450 G/L	Dip	200	
Citrus fruit	Argentina	SC, 450 G/L	Dip, spray	50-500	
	Australia	SC, 900G/KG	Dip	100	
	Brazil	SC, 485 G/L	Dip, spray	50-500	
	Columbia	SC, 450 G/L	Spray	45-90	
	Israel	SC, 450 G/L	Dip	110	
	Mexico	WP, 600 G/KG	Drench, spray	150-180	
	South Africa	TC, 985 G/KG SC, 450 G/L	Dip Dip	400 100-200	
	Spain	SL, 220 G/L	Dip	130-220	
	USA	SC, 500 G/L	Spray	100	
		TC, 985 G/KG	Spray	100-500	0.88-2.5 g/t
Endive	France	SC, 450 G/L	Dip	100-135	
	Germany	SC, 450 G/L	Dip	100-135	

Crop	Country	APPLICATION			
		Form., concn.	Method	g ai/100 l	Rate (ai)
Papayas	USA	TC, 985 G/KG	Spray	100-200	2.0 g/t
Potatoes	Argentina	SC, 450 G/L	Spray	2100	42 g/t
	Australia	SC, 450 G/L	Spray	2250	45 g/t
	Canada	SC, 450 G/L	Spray	2100	42 g/t
	Columbia	SC, 450 G/L SC, 450 G/L	Dip Spray	180 225	 4.5 g/t
	France	SC, 450 G/L	Spray	3150	63 g/t
	Germany	SC, 450 G/L	Spray	3150	63 g/t
	Italy	SC, 450 G/L	Spray	2000-4000	40 g/t
	Mexico	WP, 600 G/KG	Spray	5000	50-100 g/t
	Netherlands	SC, 209 G/L	Spray		30 g/t, PHI 60 days
		DP, 20 G/KG	Dusting		30 g/t, PHI 90 days
	New Zealand	SC, 450 G/L	Spray	2115	42 g/t
	South Africa	SC, 450 G/L	Spray	160-315	6.4 –12.8 g/t
			Dip	200	
(ware potatoes)	UK	SC, 450 G/L SL, 100 G/L SL, 220 G/L	Spray Spray Spray		40 g/t 40 g/t 44 g/t
	USA	SC, 450 G/L	Spray, dip	139	5.6 g/t
Potato seed roots	Argentina	SC, 450 G/L	Dip	180	
	USA	SC, 450 G/L	Dip	375	
Soya beans (seed)	Brazil	SC, 485 G/L	Spray		10-20 g/100 kg
(Pre-plant)	Mexico	WP, 600 G/KG	Spray	50-100	50-200 g/100 kg
Squash	Mexico	WP, 600 G/KG	Spray	150-250	
Wheat (seed)	Italy	SC, 450 G/L	Spray	120	120 g/100 kg
(Pre-plant)	Mexico	WP, 600 G/KG	Spray	100-300	100-300 g/100 kg
	USA	SC, 300 G/L	Spray	30000	67-200 g/100 kg

Table 9. Major approved pre-harvest uses of thiabendazole.

Crop	Country	Application			PHI, days
		Form., concn.	kg ai/ha	g ai/100 l	
Apples, pears	Argentina	SC, 450 g/l	0.45-0.90		0
	Italy	SC, 450 g/l		60	15
	Mexico	WP, 600 g/kg	0.50-1.0	500-1000	
	Japan	WP, 75 g/kg		7.5	14
	Spain	SC, 450 g/l	0.45-0.90	45	30
	Spain	WP, 600 g/kg	0.42-0.84		
Asparagus	UK	LS, 220 g/l	Dip, high vol. drench	100	180
Avocados	Mexico	WP, 600 g/kg	0.50-0.75	500-750	15
Bananas	Spain	SC, 450 g/l	0.45		15
	Spain	WP, 600 g/kg	0.42		15
Broccoli	Spain	SC, 450 g/l	0.45 – 0.90	68-90	7
	Spain	WP, 600 g/kg	0.30-0.85	45-85	7
Celery	Israel	SC, 450 g/l	0.40		3
	Spain	SC, 450 g/l	0.45 – 0.90	68-90	7
	Spain	WP, 600 g/kg	0.30-0.85	45-85	7
Cherries	Spain	SC, 450 g/l	0.36	45-68	15
	Spain	WP, 600 g/kg	0.42-0.44		
Citrus fruit	Japan	WP, 600 g/kg		50	1
	Mexico	WP, 600 g/kg	0.5-1.0	150-180	0
Cucumbers	Spain	SC, 450 g/l	0.45 – 0.90	68-90	3
Garlic	Italy	SC, 450 g/l	1.4 g ai/kg	135	
Grapes	Mexico	WP, 600 g/kg	0.50-1.0	500-1000	
	Japan	WP, 75 g/kg		30	45
Green beans	Spain	SC, 450 g/l	0.45 – 0.90	68-90	3
	Spain	WP, 600 g/kg	0.30-0.85	45-85	3

Crop	Country	Application			PHI, days
		Form., concn.	kg ai/ha	g ai/100 l	
Lettuce	Spain	SC, 450 g/l	0.45-0.90	68-90	7
	Spain	WP, 600 g/kg	0.30-0.85	45-85	7
Mangoes	Mexico	WP, 600 g/kg	0.50-0.70	500-700	15
Melons	Spain	SC, 450 g/l	0.45-0.90	68-90	3
Mushrooms	Australia	SC, 450 g/l	225 g ai/kg moss		
	Japan	WP, 600 g/kg		60	
	Japan	SL, 100 g/l		50-100	
	South Africa	SC, 450 g/l	120-150 g ai/80 m^2	120-150	
	UK	WP, 600 g/kg		69	1
	USA	SC, 450 g/l	265 g ai/93 m^2		0.5
Onions	Italy	SC, 450 g/l	1.4 g ai/kg	135	
	UK	SC, 450 g/l	0.293		
Peppers	Spain	SC, 450 g/l	0.45 – 0.90	68-90	3
		WP, 600 g/kg	0.30-0.85	45-85	3
Potatoes (seed)	Argentina	SC, 450 g/l	1.2		
	Italy	SC, 450 g/l		180	
	Mexico	WP, 600 g/kg		2000-3000	
	South Africa	SC, 450 g/l	dip	200-400	
			spray	1000-2000	
Potatoes (foliar)	Mexico	WP, 600 g/kg	0.50-1.0	500-1000	
Soya beans	Argentina	SC, 450 g/l	0.22-0.32		21
	Mexico	WP, 600 g/kg	0.35-0.70	350-700	
	UK	SC, 450 g/l	0.225-0.315		
Strawberries	Netherlands	FT, 120 g/kg	43 g ai/m^2		3[a]
	Spain	SC, 450 g/l	0.45-0.90	68-90	3
	Spain	WP, 600 g/kg	0.30-0.85	45-85	3
Tomatoes	Netherlands	FT, 120 g/kg	43 g ai/m^2		3[a]
	Spain	SC, 450 g/l	0.45-0.90	68-90	3
	Spain	WP, 600 g/kg	0.30-0.85	45-85	3
Vines	Spain	SC, 450 g/l	0.68-0.90	90	7
		WP, 600 g/kg		90-135	7
Rice	Mexico	WP, 600 g/kg	0.50-0.75	500-750	10
Wheat (foliar)	Argentina	SC, 450 g/l	0.22-0.32		14
	USA	WG, 890 g/kg	0.52-0.78	1600	

[a] Indoor use

RESIDUES RESULTING FROM SUPERVISED TRIALS

Residue data from supervised trials with thiabendazole on apples, bananas, chicory, citrus, mushrooms, pears, potatoes, strawberries, tomatoes and wheat are shown in Tables 10 to 17.

Table 10. Residues from post-harvest application to citrus
Table 11. Residues from pre- and post-harvest application to apples and pears
Table 12. Residues from pre-harvest applications to strawberries
Table 13. Residues from post-harvest application to bananas
Table 14. Residues from pre-harvest applications to tomatoes
Table 15. Residues from post-harvest application to chicory
Table 16. Residues from post-harvest application to potatoes
Table 17. Residues from pre-harvest irrigation and direct spray application to mushrooms

Citrus fruits. Post-harvest trials were conducted in the USA (8) and Spain (10) from 1990 to 1994 on oranges, lemons, grapefruit and tangerines. In the Spanish trials oranges were treated with single post-harvest drench applications of 66 or 110 g ai/hl. The US trials were with initial post-harvest dips at

100 or 500 g ai/hl followed by mist applications in wax at 350 or 500 g ai/hl. Benzimidazole residues were not detectable (<0.01 mg/kg). Samples were stored frozen and analysed within nine months of treatment.

Table 10. Residues of thiabendazole in or on whole unwashed citrus fruits from post-harvest applications of SC, SL, WP or TC formulations of thiabendazole in supervised trials.

Fruit, Country, Year, Variety	Application					Residues, mg/kg	Remarks	Ref.
	Form.	kg ai/ha	kg ai/hl	l/t	Type			
Orange USA, 1990 Navel	FMC 555, Mertect Fungicide	12	0.50 0.50	2.4	dip (3 min) plus spray	1.8, 1.8	(2/1)[1]	Norton, 1995b
Orange USA, 1991 Valencia	FMC 555, Mertect Fungicide	12	0.50 0.50	2.4	dip (3 min) plus spray	1.2, 1.2	(2/1)	
Grapefruit USA, 1991	FMC 555, Mertect Fungicide	12	0.50 0.50	2.4	dip (3 min) plus spray	2.9, 2.9	(2/1)	
Lemon USA, 1991	FMC 555, Mertect Fungicide	12	0.50 0.50	2.4	dip (3 min) plus spray	5.1, 5.4	(2/1)	
Orange USA, 1994 Navel	FMC 555, Mertect Fungicide	8.4	0.10 0.35	2.4	dip (3 min) plus spray	4.4, 4.8, 4.7, 4.4, 4.4,4.6	(3/2)	
Orange USA, 1994 Hamlin	FMC 555, Mertect Fungicide	8.4	0.10 0.35	2.4	dip (3 min) plus spray	3.0, 3.1, 3.0, 2.8, 2.7, 2.7	(3/2)	
Tangerine USA, 1994 Sunburst	FMC 555, Mertect Fungicide	8.4	0.10 0.35	2.4	dip (3 min) plus spray	3.9, 3.5, 3.3, 3.1, 3.5, 3.4	(3/2)	
Grapefruit USA, 1994 Marsh	FMC 555, Mertect Fungicide	8.4	0.10 0.35	2.4	dip (3 min) plus spray	3.0, 2.8, 2.5, 3.6, 3.8, 3.5	(3/2)	
Orange Spain, 1990 Clementino de nules	Tecto 20S		0.066 0.11		drench	<0.1, 0.89, 0.81 <0.1, 1.1, 1.1	(1/3) 8 DAT (1/3) 8-11 DAT	Tecnidex, 1992a[2]
Spain, 1991 Clementino de fina	Tecto 20S		0.066 0.11		drench	1.3, 3.9, 1.2, 8.5	(1/2) 28-42 DAT (1/2) 28-42 DAT	
Spain, 1991 Clementina Hernandina	Tecto 20S		0.11		drench	1.7, 2.5	(1/2) 40 DAT	
Spain, 1990 Navel	Tecto 20S		0.066 0.11		drench	0.72, 0.69, 0.79 0.64, 0.68, 0.68	(1/3) 10-12 DAT (1/3) 10-12 DAT	
Spain, 1991 Navel	Tecto 20S		0.066 0.11		drench	3.5, 0.69, 0.98 3.8, 0.52, 2.7	(1/3) 20-20 DAT (1/3 20-29 DAT	

[1]Number of analyses per sample/number of samples analysed
[2]Summary reports only
DAT: days after last treatment

Pome fruits. Post-harvest residue trials were conducted in the USA and Spain in 1990-1991 on apples and pears. An initial post-harvest dip at 60 g ai/hl was followed by a mist application in wax at 200 mg ai/hl 30 days after cold storage in ten US trials. Five trials were also conducted in Spain at 110 g ai/hl. Samples were stored frozen and analysed within nine months of treatment.

Four pre-harvest residue trials were conducted in Japan on apples and pears with WP formulations of thiabendazole (600 g ai/kg). Nine sprays at 30 g ai/hl, 1.8 kg ai/ha, were applied to apples and five at 30 g ai/hl, 1.2 kg ai/ha, to pears. Samples were analysed after PHIs of 7, 14 and 21 days.

Table 11. Residues of thiabendazole in or on whole apples and pears from applications of SC, SL, TC and WP formulations of thiabendazole in supervised trials.

Fruit, country, year	Application					Residues, mg/kg	Remarks[1]	Reference
	Form.	kg ai/ha	kg ai/hl	l/t	Type			
Apple Golden USA, 1990	Mertect 340F & Mertect Fungicide	8.4	0.060 0.20	 4.2	Dip plus spray	3.0, 2.7	(2/1)	Norton , 1992c
Red Delicious						3.0, 3.4	(2/1)	
Red Delicious						3.1, 3.4	(2/1)	
Non Delicious						3.2, 3.2	(2/1)	
Non Delicious						3.2, 3.4	(2/1)	
Apple[2] Golden Spain, 1991	Tecto 20S		0.11		Drench	1.8, 2.1 1.9, 2.2 1.8, 1.9	DAT 32 DAT 61 DAT 147	Tecnidex, 1992b
Apple[3] Japan, 1974 (Pre-harvest)	WP 9 applications		0.03		Spray	0.45, 0.47 0.42, 0.30 0.19, 0.22	PHI 7 PHI 14 PHI 21	Japan 1997
						1.1, 1.1 1.1, 1.1 0.68, 0.77	PHI 7 PHI 14 PHI 21	
						0.40, 0.43 0.38, 0.38 0.36, 0.33	PHI 7 PHI 14 PHI 21	
						1.3, 1.3 0.90, 0.89 0.41, 0.41	PHI 7 PHI 14 PHI 21	
Pear[3], 1990 USA, CA Bartlett	Mertect 340F, Mertect Fungicide	8.4	0.060 0.20	4.2	Dip + Spray	1.1, 1.1	(2/3)	Norton, 1992b
WA						3.0, 3.2,		
WA						0.89, 0.87		
Bosc NY	Mertect 340F, Mertect Fungicide	8.4	0.060 0.20	4.2	dip plus spray	3.6, 3.7	(2/2)	
NY						4.8, 5.1	(2/2)	
Pear[2], Blanquilla Spain 1991						1.7, 1.8 2.0, 2.1	DAT 75 DAT 160	Tecnidex, 1992b
Pear Japan, 1974 (Pre-harvest)	WP 5 applications		0.03		spray	0.12, 0.11 0.083, 0.083 0.075, 0.072	PHI 7 PHI 14 PHI 21	Japan, 1997
						0.50, 0.50 0.52, 0.51 0.28, 0.27	PHI 7 PHI 14 PHI 21	
						0.10, 0.093 0.087, 0.081 0.077, 0.072	PHI 7 PHI 14 PHI 21	

[1]Number of analyses per sample/number of samples analysed
[2]Average residues from triplicate analyses of samples from two trials. Summary reports only submitted
[3]Results of duplicate analyses
DAT: days after last treatment
PHI: pre-harvest interval in days

Strawberries. Pre-harvest trials were conducted on strawberries in Mexico and Spain in 1989-1992 with ground spray foliar applications of SC and WP formulations. In Mexico four applications were made 7 days apart, at rates of 0.50-2.0 kg ai/ha. In Spain there were single applications at 1.2 kg ai/ha.

Table 12. Residues of thiabendazole in strawberries from pre-harvest applications of SC and WP formulations of thiabendazole in supervised trials.

Country, Year	Application				PHI, days	Residues , mg/kg	Reference
	Form.	kg ai/ha	kg ai/hl	Type			
Mexico[1], 1992	Tecto 60	0.50	0.083	Ground spray (4)	0 1 3 7 14	0.84, 0.56 1.2, 0.67 1.6, 0.73 1.6, 0.70 0.83, 0.29	Unduraga, 1992a
Mexico[1] 1992	Tecto 60	1.0	0.17	Ground spray (4)	0 1 3 7 14	1.5, 0.69 4.4, 2.2 2.7, 1.7 1.8, 1.6 1.8, 1.7	
Mexico[1] 1992	Tecto 60	2.0	0.34	Ground spray (4)	0 1 3 7 14	4.3, 2.4 6.4, 3.5 5.9, 2.9 5.0, 2.0 1.8, 1.1	
Mexico[1] 1992	Tecto 60	0.50	0.083	Ground spray (4)	0 1 3 7 14	1.1, 0.77 2.1, 1.3 4.3, 3.2 1.4, 0.89 0.71, 0.33	Unduraga, 1992b
Mexico[1] 1992	Tecto 60	1.0	0.17	Ground spray (4)	0 1 3 7 14	2.1, 1.2 5.8, 3.7 4.4, 2.0 3.4, 1.7 0.83, 0.52	
Mexico[1] 1992	Tecto 60	2.0	0.34	Ground spray (4)	0 1 3 7 14	3.1, 2.1 9.7, 4.9 9.3, 4.3 6.4, 3.5 3.6, 1.2	
Mexico 1992	Tecto 60	0.50	0.093	Ground spray (4)	0 1 3 7 14	1.4 1.2 0.66 0.41 0.31	
Mexico 1992	Tecto 60	1.0	0.19	Ground spray (4)	0 1 3 7 14	1.9 1.8 1.2 0.87 0.53	
Mexico 1992	Tecto 60	2.0	0.38	Ground spray (4)	0 1 3 7 14	1.8 4.5 2.6 1.8 0.90	
Spain[2] 1989	Tecto 45	1.2	0.030	Ground spray (1)	0 3 7 14	1.7 1.6 1.4 1.1	Ag Vet, 1991

Country, Year	Application				PHI, days	Residues , mg/kg	Reference
	Form.	kg ai/ha	kg ai/hl	Type			
Spain[2] 1989	Tecto 45	1.2	0.030	Ground spray (1)	0 3 7 14	0.78 0.33 0.43 0.1	

[1]Samples were taken from duplicate plots
[2]Residues are the averages of quadruplicate analyses. The trials were conducted under plastic

Bananas. Post-harvest trials were conducted in the USA, Honduras and Guadeloupe in 1992-1995 with single dips or sprays to run-off of SL and SC formulations of thiabendazole: four dip trials in Guadeloupe in 1992, two dip trials in Hawaii, USA, in 1995 and four spray trials in Honduras in 1992. The fruit were ripened to the green to yellow tip stage (the stage preferred by customers) according to normal commercial practice. At least 10 fingers taken from two clusters were composited for one sample. The samples were stored frozen and analysed within three months of treatment. Benzimdazole residues could not be detected in any of the samples

Table 13. Residues of thiabendazole in or on whole bananas and banana pulp from single post-harvest applications of SC and SL formulations of thiabendazole in supervised trials.

Country Year	Application					Sample	Residues, mg/kg	Remarks	Ref.
	Form.	kg ai/ha	kg ai/hl	l/t	Type				
Hawaii USA, 1995	Mertect 340F	4.2	0.040	10.5	Dip (15 sec)	Green banana	1.4, 1.3, 1.2, 1.6, 1.4, 1.2, 1.7 1.5, 1.7, 1.4	(1/10)[1]	Norton, 1995a
					Dip (60 sec)		1.8, 1.6, 1.6, 1.6, 1.9, 2.3, 1.5, 2.0, 1.6, 1.3	1/10	
Hawaii USA, 1995	Mertect 20S	4.2	0.040	10.5	Dip (15 sec)	Green banana	0.94, 1.1, 1.1, 1.2 1.0, 0.97, 1.4, 1.0, 1.0, 0.97	(1/10)	
					Dip (60 sec)		1.3, 1.4, 1.4, 1.0, 1.6, 1.3, 1.1, 1.4, 1.5, 0.96	(1/10)	
Honduras 1992 Los Flores	Mertect 20S	4.2	0.040	10.5	Spray	Green banana Ripe banana pulp	0.96, 0.92, 1.1, 1.0, 0.91, 0.95, 0.95, 0.98, 1.2, 0.96 mean: 0.99 0.023, 0.014, 0.023, 0.020, 0.024, 0.005, 0.012, 0.024, 0.029, 0.022, mean: 0.019	(2/10)	Norton, 1993a
Corozal						Green banana Ripe banana pulp	0.89, 0.88, 0.80, 1.0, 0.79, 0.60, 0.67, 1.0, 0.90, 0.72 mean: 0.83 0.010, 0.009, 0.008, 0.016, 0.014, 0.012, 0.010, 0.016, 0.008, 0.006, mean: 0.011		
Honduras 1992 Los Flores	Mertect 340-F	4.2	0.040	10.5	Spray	Green banana Ripe banana pulp	0.67,0.67, 0.79, 0.78, 0.70, 0.65, 0.75, 0.68, 0.64, 0.85 mean: 0.72 0.018, 0.028, 0.008, 0.003, 0.031, 0.025, 0.025, 0.030, 0.031, 0.015, mean: 0.021	(1/10)[a]	Norton, 1993a
Corozal						Green banana Ripe banana	0.63, 0.76, 0.88, 0.62, 0.59, 0.84, 1.0, 0.76, 0.63, 0.72 mean: 0.74 0.018, 0.020, 0.026,	(1/10)	

Country Year	Application					Sample	Residues, mg/kg	Remarks	Ref.
	Form.	kg ai/ha	kg ai/hl	l/t	Type				
						pulp	0.025, 0.016, 0.019, 0.014, 0.017, 0.016, 0.028 mean: 0.02		
Guadeloupe, 1992	Mertect 20S		0.045		dip (2 min)	Green banana	1.8, 1.6, 1.5 1.1, 1.3, 0.96 1.4, 1.1, 1.6	(3/3)	Sing, 1992
Guadeloupe, 1992	Mertect SC		0.045		dip (2 min)	Green banana	1.4, 1.4, 1.9 2.6, 2.1, 1.7 3.3, 2.8, 3.5	(3/3)	
Guadeloupe, 1992	Mertect 20S		0.090		dip (2 min)	Green banana	1.8, 1.8, 1.1 1.0, 2.1, 1.6 2.1, 2.6, 2.3	(3/3)	
Guadeloupe, 1992	Mertect SC		0.090		dip (2 min)	Green banana	2.3, 2.6, 1.8 3.9, 7.3, 5.3 4.7, 4.3, 3.9	(3/3)	

[1]Number of assays per sample/number of samples analysed

Tomatoes. Four pre-harvest trials were conducted on tomatoes grown under plastic in Spain in 1990-1991 with ground spray foliar application of thiabendazole SC and WP formulations. Two trials in 1990 were with two applications, 7 days apart, at 0.50 kg ai/ha, and two in 1991 with single applications at 3.1 kg ai/ha (AgVet, 1991).

Table 14. Residues of thiabendazole in tomatoes grown under plastic in Spain from pre-harvest applications of SC and WP formulations of thiabendazole in supervised trials

Application				PHI, days	Residues, mg/kg	Remarks[1]
Form.	kg ai/ha	kg ai/hl	Type			
Tecto 45	3.1	0.090	Ground spray (1)	0 4 7 11 14 21	1.6, 2.3, 2.4 1.7, 1.7, 1.8 1.6, 1.8, 2.0 1.4, 1.6, 1.6 1.3, 1.5, 2.2 1.0, 1. 2,1.4	(3/1)[1]
Tecto 45	3.1	0.090	Ground spray (1)	0 4 7 11 14 21	2.0, 2.3, 1.9 1.9, 2.1, 1.7 1.6, 2.0, 1.5 1.4, 1.9, 1.1 1.3, 1.5, 0.83 1.3, 1.3, 0.84	(3/1)
Tecto 60	0.50	0.050	Ground spray (2)	3 7 10	0.26, 0.32, 0.18, 0.30 0.25, 0.36, 0.50, 0.37 0.44, 0.41, 0.80, 0.73	(4/1)
Tecto 60	0.50	0.050	Ground spray (2)	3 7 10	0.28, 0.32, 0.40, 0.25 0.35, 0.30, 0.43, 0.40 0.52, 0.32, 0.72, 0.68	(4/1)

[1]Number of analyses per sample/number of samples

Chicory. Pre-planting trials were conducted in France between 1979 and 1982 with single dip or spray applications of SC and SL formulations of thiabendazole to chicory roots which were grown to harvest. Eleven trials in 1979, 1980 and 1982 were with Flowable SC and 9 trials in 1979 and 1980 with the 20-S formulation at 67-630 g ai/hl. The endive leaves and roots were analysed separately. Residues of thiabendazole in the edible endive leaves did not exceed 0.05 mg thiabendazole/kg.

Table 15. Residues of thiabendazole in or on whole unwashed chicory from single pre-planting applications of SC and SL formulations of thiabendazole in supervised trials (Schreur, 1992).

Country, Year, Variety	Application					Residues, mg/kg	
	Form	kg ai/ha	kg ai/hl	l/t	Type	Leaf	Root
France, 1979 Witloof	Mertect Flowable		0.067 0.10 0.20		dip (2 min)	<0.005 (2) <0.005 <0.005	<0.065 0.038 <0.015
France, 1979 Witloof	Mertect 20S		0.067 0.10 0.20		dip (2 min)	<0.005 (2)	- <0.005, (2) <0.005 0.036
France, 1980 Witloof	Mertect Flowable	 20 40 50	0.10 0.20 0.40 0.25 0.50 0.63	 8 8 8	dip (3-5 min) spray	<0.05 <0.05 <0.05 <0.05 <0.05 <0.05	9.4 12 4.4 3.7 12 3.7
France, 1980 Witloof	Mertect 20S	 20 40 50	0.099 0.20 0.40 0.25 0.50 0.63	 8 8 8	dip (3-5 min) spray	<0.05 <0.05 1.2* <0.05 <0.05 <0.05	13 23 37 55 7.3 12
France, 1982 Witloof	Mertect Flowable	 60	0.10 0.60	 10	dip (2 min) spray	<0.05 (2) <0.05 (2)	10, 10 10, 10

*Probable contamination

Potatoes. Post-harvest residue trials were conducted in the UK and the USA from 1975 to 1990. Seven trials in the UK were with single spray mist applications of a Flowable formulation at 30-80 g ai/t on whole potatoes. Potatoes in the US trials were subjected to an initial seed treatment at 2400 g ai/hl before cutting and planting, followed by an application at 6.2 g ai/t immediately after harvest and before storage, and a second application at 6.2 g ai/t approximately 30 days later. Samples were stored frozen and analysed within 19 months of treatment.

Table 16. Residues of thiabendazole in or on whole potatoes from post-harvest applications of SC formulations of thiabendazole in supervised trials.

Country, Year	Application					Residues, mg/kg	Remarks	Reference
	Form.	g ai/t	kg ai/hl	l/t	Type			
USA, ID, 1990 Russet	Mertect 340-F	 6.2 6.2	2.4	 0.26 0.26	dip spray spray	 1.8, 1.9, 1.6, 1.6, 1.7, 1.7, 1.2, 1.2,	(2/4) [1] 3 applications[2]	Norton, 1993b
WA Burbank		 6.2 6.2	2.4	 0.26 0.26		 7.0, 7.1, 6.0, 6.3, 7.0, 7.1, 7.0, 7.3	(2/4)	
USA, MI, 1990 Yukon gold		 6.2 6.2	2.4	 0.26 0.26	dip spray spray	 4.9, 5.1, 4.1, 4.3, 2.8, 3.8, 5.1, 5.5	(2/4) 3 applications[2]	
USA, ME, 1990 Superior		 6.2 6.2	2.4	 0.26 0.26	dip spray spray	 3.3, 3.4, 2.6, 3.4, 4.0, 4.2, 3.4, 3.6	(2/4) 3 applications[2]	
UK, 1990 Estima[3]	Storite FL	40			spray	0.6, 1.3, 1.0 1.3, 1.9, 1.9 2.0, 2.0, 2.0	DAT 0, DAT 42 DAT 84	McKenzie, 1991
		80				1.9, 1.6, 1.9 2.5, 2.9, 3.5 3.1, 3.0, 2.8	(3/1) DAT 0 DAT 42 DAT 84	
Obelix[3]		40			spray	2.0, 2.6, 1.4	DAT 0, 42, 84	
		80				3.3, 2.8, 2.6	DAT 0, 42, 84	

Country, Year	Application					Residues, mg/kg	Remarks	Reference
	Form.	g ai/t	kg ai/hl	l/t	Type			
Manfora[3]		40			spray	1.7, 1.8, 2.2	DAT 0, 42, 84	
		80				2.0, 3.2, 2.1	DAT 0, 42, 84	
Record	Extractect	30	30		spray	1.2, 2.6 1.7, 2.4, 1.5	(1/2) DAT 0 DAT 42	Agriserch, 1991
Desiree	Exractect	30	30		spray	4.4 5.4	DAT 0 DAT 42	
		60	30		spray	6.6, 7.3 8.2, 8.7	(1/2) DAT 0 DAT 42	
Cara	Extractect	30	30		spray	12 11	DAT 0 DAT 42	

[1]Number of analyses per sample/number of samples analysed
[2]Seed treatment plus 2 post-harvest sprays approximately 30 days apart
[3]Samples were lightly washed before analyses to remove adhering soil
DAT: days after last treatment

Sugar beet. Pre-harvest residue trials were conducted on sugar beet grown in Spain in 1996 with ground spray applications of a thiabendazole SC formulation. Eight trials were with single applications, or 2 applications approximately 33 days apart, at 480 g ai/ha (0.12 kg ai/hl) after development of 4-8 leaves and the crops were grown to harvest (Valcarcel, 1977). Residues of thiabendazole were <0.01 mg/kg in all 16 root samples taken from 0 to 91 days after the last application. The leaves contained the residues shown below:

DAT	Residues in leaves, mg/kg
0	0.07
29-36	0.07, 0.36, 0.05, 0.07
59 –65	<0.01, <0.01, <0.01, 0.41, 0.12
>71	<0.01, 0.019

Mushrooms. Six trials were conducted in the USA in 1990-1991 on mushrooms treated with four applications of an aqueous solution by irrigation at 54 or 108 g ai/100 m^2 or by direct spray at 9.5 or 19 g ai/hl. Applications were made after pinning or after the first harvest break and then after the second, third and fourth breaks according to label instructions. The maximum residues of thiabendazole on mushrooms collected 12 hours after the last application ranged from 2.4 to 13 mg/kg for irrigation and from 30 to 41 mg/kg for spray applications. The residues of benzimidazole were <0.01 mg/kg in all samples. Samples were stored frozen and analysed within 18 months of treatment.

In four trials in Japan in 1988 and two in 1993 mushrooms were treated with a WP formulation using single applications to the bed medium at a rate of 0.120 g ai/kg. The residues of thiabendazole did not exceed 0.25 mg/kg.

Table 17. Residues of thiabendazole in or on whole mushrooms from pre-harvest applications of WP and SC formulations of thiabendazole.

Country, Year	Application				Residues, mg/kg[1]	Replication; PHI, days	Reference
	Form.	g ai/100 m^2	kg ai/hl	Type			
USA, 1990	MERTECT 340F	108 54 54 54	0.019 0.0095 0.0095 0.0095	Irrigation	3.1, 3.2 (1 & 2) 3.1, 3.1 (3) 3.8, 3.9 (4)	(2/1)[2] (2/1) (2/1)	Norton, 1992d
USA, 1990	MERTECT 340F	108 54 54 54	0.019 0.0095 0.0095 0.0095	Irrigation	1.9, 1.9 (1 & 2) 2.0, 2.2 (3) 2.4, 2.5 (4)	(2/1) (2/1) (2/1)	

Country, Year	Application				Residues, mg/kg[1]	Replication; PHI, days	Reference
	Form.	g ai/100 m[2]	kg ai/hl	Type			
USA, 1990-91	MERTECT 340F	108 54 54 54	0.019 0.0095 0.0095 0.0095	Irrigation	9.3, 9.6 (1) 7.0, 7.3 (2) 13, 13 (3) 12, 12 (4)	(2/1) (2/1) (2/1) (2/1)	
USA, 1990-91	MERTECT 340F	108 54 54 54	0.019 0.0095 0.0095 0.0095	Irrigation	5.8, 6.0 (1) 3.9, 3.9 (2) 5.9, 6.1 (3) 7.6, 8.0 (4)	(2/1) (2/1) (2/1) (2/1)	
USA, 1990	MERTECT 340F	108 54 54 54	0.019 0.0095 0.0095 0.0095	Spray	37, 38 (1 & 2) 19, 21 (3) 30, 31 (4)	(2/1) (2/1) (2/1)	
USA, 1990-1991	MERTECT 340F	108 54 54 54	0.019 0.0095 0.0095 0.0095	Spray	48, 50, 50, 52 (1) 25, 26, 27, 26 (2) 33, 34, 35, 36 (3) 37, 39, 40, 41 (4)	(2/2) (2/2) (2/2) (2/2)	
Japan, 1988	PANMUSH WP	120 mg/kg of bed			0.092, 0.089 0.12, 0.11 0.018 0.008	PHI 125[3] PHI 115 PHI 125 PHI 115	Japan, 1997
Japan, 1993	PANMUSH WP	120 mg/kg of bed			0.25, 0.25 0.19, 0.19	PHI 196 PHI 188	

[1]Figures in parentheses are breaks at which thiabendazole was applied
[2]Number of analyses per sample/number of samples
[3]Results of 4 replicate trials at the same site

Wheat. Fourteen pre-harvest trials were conducted according to GAP on wheat in the USA in 1990 using single ground or aerial sprays of thiabendazole WG formulation at 620 g ai/ha after development of 2 to 3 tillers but before the first node. The wheat was grown to harvest (Armstrong and Norton, 1993b). The residues of thiabendazole and the metabolite benzimidazole were <0.05 mg/kg in all 14 grain samples. The thiabendazole residues in the straw were in rank order <0.05 (11), 0.11, 0.07 and 0.13 mg/kg. Samples were stored frozen and analysed within 23 months of treatment.

Animal feeding studies

Cows. Dairy cattle (3 per group) were dosed once daily by capsule for 28 days with thiabendazole at levels corresponding to 25, 75 and 250 ppm in the feed. The level of 25 ppm represents a likely maximum intake, based on a diet of 70% maize grain and 30% citrus pulp or 50% maize grain, 25% apple pomace and 25% potato waste. There were two control cows. Morning and evening milk samples were collected from all cows on days -1, 1, 2, 4, 7, 14, 21, 28, 29, 35, 42 and 56, and a 500-ml sub-sample from each primary sample was retained. The two sub-samples from each day's milking of the cows in each group were combined and duplicate 500-ml samples were analysed for thiabendazole and 5-hydroxythiabendazole (5-OH-TBZ). The tissues and organs from two of the three cows in each dosed group were collected on day 29 of the study. The remaining cow from each group was slaughtered on day 57. Tissues from each cow were analysed for thiabendazole and 5-OH-TBZ.

The total residues of thiabendazole and 5-OH-TBZ in the milk and tissues increased with increasing dose but not necessarily proportionally. Residues in the milk reached a plateau two days after treatment, but they were ≤0.01 mg/kg higher than the control value in the low-dose group. This difference is not considered to be significant. The total residues of thiabendazole plus 5-OH-TBZ in the cows dosed at the 25 ppm level were <0.05 mg/kg in milk and tissues except a single value of 0.05 mg/kg in kidney (Justin, 1990a). No difference was observed between the thiabendazole residues measured in various muscles. The residues decreased rapidly to control levels when the animals were returned to a thiabendazole-free diet.

Table 18. Residues of thiabendazole and 5-hydroxythiabendazole in milk (daily averages of 3 cows) from cows dosed with thiabendazole.

Sampling day	Residues, mg/kg, at dose equivalent to					
	25 ppm		75 ppm		250 ppm	
	TBZ	5-OH-TBZ	TBZ	5-OH-TBZ	TBZ	5-OH-TBZ
-1	0.013	0.003	0.012	0.004	0.013	0.004
1	0.014	0.009	0.014	0.059	0.014	0.072
2	0.014	0.012	0.014	0.081	0.017	0.110
4	0.015	0.013	0.014	0.091	0.015	0.110
7	0.014	0.013	0.015	0.083	0.014	0.115
14	0.014	0.013	0.015	0.073	0.016	0.111
21	0.013	0.012	0.015	0.091	0.017	0.127
28	0.013	0.013	0.014	0.108	0.016	0.134
29	0.016	0.004	0.013	0.008	0.015	0.067
35	0.014	0.003	0.013	0.004	0.012	0.004
42	0.010	0.002	0.014	0.006	0.014	0.004
49	0.015	0.004	0.014	0.005	0.018	0.002
56	0.013	0.004	0.014	0.004	0.018	0.002

Animals fed a thiabendazole-free diet on days 29-56
5-OH-TBZ: 5-hydroxythiabendazole

Table 19. Residues of thiabendazole and 5-hydroxythiabendazole in tissues from cows dosed with thiabendazole.

Sample (Sampling day)	Residues, mg/kg, at dose equivalent to					
	25 ppm		75 ppm		250 ppm	
	TBZ	5-OH-TBZ	TBZ	5-OH-TBZ	TBZ	5-OH-TBZ
Fat (29)	0.016 0.018	0.004 0.002	0.013 0.017	0.009 0.012	0.014 0.015	0.007 0.010
Fat (57)	0.016	0.003	0.006	0.002	0.017	0.002
Kidney (29)	0.012	0.038 0.049	0.016 0.017	0.079 0.42	0.024 0.030	0.33 0.55
Kidney (57)	0.020	0.010	0.020	0.008	0.022	0.014
Liver (29)	0.022	0.026 0.028	0.036 0.060	0.041 0.130	0.056 0.080	0.12 0.16
Liver (57)	0.018	0.016	0.018	0.015	0.020	0.017
Muscle (29)	0.012 0.014	0.002 0.003	0.013 0.014	0.004 0.006	0.015 0.017	0.004 0.005
Muscle (57)	0.014	<0.01	0.012	0.002	0.014	0.002

Animals fed a thiabendazole-free diet on days 29-57

Chickens. Ten groups of birds (25 per group, males and females) were treated continuously for 7 weeks with thiabendazole at levels corresponding to 0, 2, 20, 200 and 2000 ppm in the feed. Four males and four females at each treatment level were killed within four hours of the last dose and the liver, kidney, fat and muscle analysed for thiabendazole and 5-hydroxy-thiabendazole, as were the eggs from the three highest treatment groups. The total residues in the tissues and eggs of birds dosed at the 20 ppm feed level (the expected maximum intake based on a poultry diet of 70% maize grain, 20% potatoes and waste and 10% wheat grain) were <0.1 mg/kg, except a single value of 0.12 mg/kg in kidney (Justin, 1990b).

Table 20. Residues[1] of thiabendazole and 5-hydroxythiabendazole in tissues and eggs from chickens dosed with thiabendazole.

Sample	Residue, mg/kg, at dose equivalent to									
	0		2 ppm		20 ppm		200 ppm		2000 ppm	
	TBZ	5-OH-T[2]	TBZ	5-OH-T	TBZ	5-OH-T	TBZ	5-OH-T	TBZ	5-OH-T
Fat/skin	0.009 0.013	0.009 0.013	0.010 0.012	0.009 0.013	0.010 0.015	0.010 0.013	0.024 0.060	0.029 0.055	0.16 0.41	0.20 0.63
Kidney	0.01 0.026	0.013 0.02	0.018 0.040	0.022 0.041	0.018 0.029	0.050 0.093	0.038 0.057	0.24 0.79	0.19 0.54	1.5 5.7
Liver	0.005 0.008	0.012 0.019	0.006 0.012	0.014 0.029	0.010 0.014	0.046 0.067	0.027 0.051	0.16 0.58	0.29 0.60	1.8 5.2
Muscle	0.007 0.008	0.005 0.007	0.007 0.009	0.006 0.008	0.009 0.013	0.008 0.010	0.019 0.035	0.016 0.036	0.081 0.26	0.17 0.64
Egg yolk			-	-	0.007 0.020	0.016 0.031	0.038 0.063	0.39 1.3	0.53 0.67	1.2 1.9
Egg white			-	-	0.003 0.011	0.004 0.012	0.017 0.027	0.032 0.048	0.18 0.21	0.24 0.36

[1]Average residues from two groups at each level

FATE OF RESIDUES IN STORAGE AND PROCESSING

In storage

Potatoes. The effect of cold storage on the residues of thiabendazole in or on potatoes was studied in Greece in 1985. One hundred kg of potatoes were sprayed with a flowable formulation at 30 or 60 g ai/tonne and stored in the dark at 5°C and 100% relative humidity. Random samples of six whole tubers or the separated pulp and peel from six tubers were analysed for thiabendazole over a 6-month period. The initial (day 0) samples were analysed after the spray deposit had dried. Unwashed tubers showed an initial loss (probably mechanical) of thiabendazole residues between day 0 and day 14 but thereafter the surface residues did not decline significantly. The results are shown in Table 21 (Lentza-Rizos, 1986).

Table 21. Effect of cold storage on residues of thiabendazole in or on potatoes treated with single post-harvest applications of SC formulations of thiabendazole.

Sample	Thiabendazole, mean of 2 analyses, mg/kg				
	Storage period, days	Treatment with 30 kg ai/ha			Treatment with 60 kg ai/ha
		Pulp	Peel	Whole[1]	Whole[2]
Unwashed tubers	0	0.9	108	27	52
	14	1.3	61	15	33
	28	1.2	48	12	32
	56	1.3	56	14	34
	84	1.6	66	16	34
	161	2.2	49	13	35
	189	2.3	69	17	36
Washed tubers	0	0.07	6.2	1.4	3.7
	14	0.07	6.6	1.6	3.4
	28	0.07	5.5	1.4	3.3
	56	0.1	7.6	2.0	5.5
	84	0.1	8.7	2.5	7.0
	161	0.1	8.6	2.1	5.2
	189	0.1	11.5	2.9	5.2

[1]Calculated from the residues in the peel and pulp
[2]Whole potatoes analysed

Apples. The effect of cold storage on the residues of thiabendazole was studied in Spain in 1987. Apples were dipped for 35 seconds in aqueous suspensions of Tecto 60 at an application rate of 100 g ai/hl and the treated fruit stored at 0-2°C and 85-90% relative humidity. The residues of thiabendazole were determined 24 hours after treatment and at monthly intervals during storage. The results are shown in Table 22 (Cano *et al.*, 1987).

Table 22. Effect of cold storage on the residues of thiabendazole in or on unwashed apples treated with single post-harvest dips of WP formulations of thiabendazole. Spain, 1987. Single samples analysed.

Variety	Application			Storage period, days	Residues, mg/kg		
	Form	kg ai/hl	Type		Whole[1]	Peel	Pulp
Golden Delicious	Tecto 60 WP	0.10	Dip	0	3.3	4.5	0.54
				37	2.9	4.2	0.11
				62	2.3	3.7	0.19
				90	2.3	3.0	0.27
				125	1.9	2.1	0.12
				146	2.0	1.9	0.10
				174	1.8	1.8	0.16
Starking	Tecto 60 WP	0.10	Dip	0	3.5	4.6	0.72
				37	3.0	3.9	0.17
				62	2.6	3.8	0.18
				90	2.1	2.6	0.27
				125	1.9	1.6	0.10
				146	1.4	1.3	0.13
				174	0.82	0.83	0.15

[1]Residues in whole apples were calculated from the residues in the peel and pulp

In processing

Citrus fruit. Two processing trials were conducted in the USA in 1990. Oranges and grapefruit were treated with a post-harvest dip at 12 g ai/t followed by a spray mist application in wax at 500 g ai/hl. Whole, washed fruit were processed.

The effects of simulated home processing on residues of thiabendazole in the preparation of marmalade, both in an open preserving pan and in a microwave oven were studied in the UK in 1993.

The results of all three trials are shown in Table 23.

Table 23. Residues of thiabendazole in processed fractions of washed and unwashed citrus fruit treated post-harvest.

Ref.	Fruit, Country Year	Sample	Mean residue, mg/kg	Processing factor	Replications[1]
Norton, 1992a	Orange, USA, 1990	Washed fruit	1.1		4/1
		Juice	0.07	0.064	2/1
		Molasses	5.4	4.9	2/1
		Oil	14	12.7	2/1
		Finisher pulp	0.16	0.15	2/1
		Dried pulp	8.7	7.9	2/1
	Grapefruit USA, 1990	Washed fruit	1.55		2/1
		Juice	0.06	0.038	2/1
		Molasses	9.15	5.9	2/1
		Oil	14	9.0	2/1

		Finisher pulp Dried pulp	0.15 12.5	0.097 8.1	2/1 2/1
Friar and Reynolds, 1994	Orange UK, 1993	Unwashed fruit Peel Marmalade (preserving pan) Marmalade (microwave)	2.41[2] 8.64[3] 0.78 0.90	 3.6 0.32 0.37	5/1 1/3 1/3

[1] Number of analyses per sample/number of samples analysed
[2] About 3.5 kg of oranges were processed
[3] Residues in the peel were calculated from the peel/whole orange mass ratio and the residues in the whole oranges. It was assumed that the pulp did not contain any residue.

Apples. Apples were treated with a post-harvest dip at 60 g ai/hl followed by a spray mist application in wax at 8.4 g ai/t approximately 30 days after cold storage in the USA in 1990. Whole unwashed fruit were processed into juice, wet pomace and dried pomace (Norton, 1992c). The thiabendazole residues were 3.85 mg/kg in washed whole apples, 1.05 mg/kg in apple juice, 13.5 mg/kg in wet pomace and 45 mg/kg in dried pomace.

Potatoes. Potatoes were processed to chips in the USA in 1990. Seed potatoes were dipped in an aqueous suspension at 2400 g ai/hl before cutting and planting, and the daughter tubers were sprayed at 6.2 g ai/t immediately after harvest and before cold storage. A second application at 6.2 g ai/t followed approximately 30 days later. The whole washed potatoes were processed by successive washing, abrasive peeling, washing, slicing, washing, frying in vegetable oil at 178-182^{o}C, de-oiling, and salting. The results are shown in Table 24 (Norton, 1993b).

The effect of microwave and oven cooking on the residues of thiabendazole in or on potatoes was studied in the UK in 1990 (Friar and Reynolds, 1991). Potatoes treated post-harvest with a single application of 40 g ai/t were stored for 182 days and the raw peel, raw pulp and unpeeled raw potatoes were subjected to microwave and oven cooking. The residues found in the processed fractions of individual potatoes are shown in Table 25.

Table 24. Residues of thiabendazole in processed fractions of potatoes.

Sample	Residues, mg/kg	Processing factor
Unwashed whole potatoes	6.5	
Wash water	2.2	
Washed potatoes	2.2	0.34
Wet peel	6.4	0.98
Dried peel	109	17
Chips	<0.5	<0.08
Flakes	0.2	0.03

Table 25. Residues of thiabendazole in processed potato fractions

Sample	Residues, mg/kg		Processing factor[1]
	Individual potatoes	Mean	
whole potato before cooking	1.7, 1.9, 2.4, 2.7, 3.0	2.36	
whole potato, microwave cooked	4.1, 2.9, 2.6, 4.0, 2.9, 3.9	2.84	1.2
whole potato, oven cooked	2.8, 3.2, 2.9, 3.8	3.17	1.34

Sample	Residues, mg/kg		Processing factor[1]
	Individual potatoes	Mean	
raw peel before cooking	9.1, 10, 10, 12, 12, 12, 10, 20, 14	12.2	
peel, microwave cooked	17, 14, 17, 25, 16, 16, 12, 15	16.4	1.34
peel, oven cooked	13, 13, 12, 16 15, 16, 11, 16	14	1.15
raw pulp before cooking	0.05, 0.05, 0.15 0.06, 0.14	0.09	
pulp, microwave cooked	0.05, 0.06, 0.03 0.04	0.05	0.56
pulp, oven cooked	0.04, 0.04, 0.07, 0.09	0.06	0.67

[1]From raw to cooked whole potato, raw to cooked peel, and raw to cooked pulp

Four trials on the effects of washing, boiling, baking and crisping on the residues of thiabendazole in or on potatoes were conducted in the UK in 1976. Potatoes were treated post-harvest with single applications at 40 or 80 g ai/t. The tubers were processed after storage for 1 and 21 days after treatment and the residues of thiabendazole determined (AgVet, 1976). The results are shown in Table 26.

Table 26. The effect of washing, boiling, baking and crisping on the residues in or on potatoes treated post-harvest with an SC formulation of thiabendazole.

Process	Storage, days	Residues, mg/kg[1]			Processing factor from	
		40 kg ai/t	80 kg ai/t	Untreated	Raw potatoes	Washed potatoes
Unwashed	1	16.95	24.6	0.095		
	21	20.3	28.4	0.08		
Washed	1	1.5	2.7	0.1	0.088, 0.11	
	21	5.36	7.26	0.075	0.26	
Washed, peeled, chipped	1	0.11	0.15	0.08	0.006	0.073, 0.055
	21	0.24	0.47	0.09	0.012, 0.017	0.044, 0.065
Washed, peeled, crisped	1	0.17	0.17	0.5	0.010, [0.007	
	21	No valid results		0.28		
Washed, peeled, boiled	1	0.22	0.45	0.09	0.013, 0.018	0.15, 0.16
	21	0.25	0.36	0.09	0.012, 0.013	0.047, 0.05
Washed, baked	1	1.8	3.13	0.13	0.18, 0.13	1.2, 1.16
	21	3.0	8.25	0.35	0.15, 0.29	0.55, 1.13

[1]Mean residues from processing Record and Pentland Dell varieties

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

Monitoring data. The results of retail monitoring carried out in the UK by the Working Party on Pesticide Residues (WPPR) from samples obtained in 1991 are shown in Table 27.

Table 27. Residues of thiabendazole in retail commodities.

Commodity	Source	No. of samples	Residues, mg/kg	No. of samples in range
Main crop potato	UK	176	<0.1 0.1-0.9 1.2-2.5	149 20 7
Cooking apples	UK	24	<0.1	24
Dessert apples	UK	25	<0.2 0.2-4.5	24 1
Dessert apple	imported	36	<0.2 0.2-4.5	28 8
Green cabbage	UK	17	<0.05	17
Green cabbage	imported	5	<0.05	5
Chicory	UK	8	<0.2	8
Chicory	imported	3	<0.2	3
Chicory	unknown	1	<0.2	1
Grapefruit	imported	25	<0.5 0.6-6.6	8 17
Lemon	imported	12	<0.5 0.6-2.1	9 3
Strawberry	UK	19	<0.2	19
Strawberry	imported	20	<0.2 0.3-0.8	18 2
Strawberry	unknown	4	<0.2	4

NATIONAL MAXIMUM RESIDUE LIMITS

The following national MRLs were reported to the Meeting.

Country, commodity	MRL, mg/kg
Argentina	
citrus fruit (pre-harvest and post-harvest)	10
apple (pre-harvest and post-harvest), banana with peel, pear, quince-(post-harvest)	3
onion, garlic	1
potato-(post-harvest), banana without peel (post-harvest)	0.4
rice	0.2
Austria	
citrus fruit	6
bananas, Chinese cabbage, potatoes, pome fruit, turnip-rooted celery	3
other vegetables	1.5
rape seed	1
cereals	0.5
other	0.1
Belgium	
pome fruit	10
strawberries, potatoes	3
grains	0.2
bulb vegetables, tomatoes	0.1
chicory	0.05
other	0.1*
Brazil	
apples, citrus fruit, pears	10
potato	5

Country, commodity	MRL, mg/kg
banana (unpeeled)	3
banana (without peel)	0.4
cereals (unprocessed)	0.2
mango, papaya, peaches, honeydew melons, squash, cotton seed, soya beans, grasses	0.1* P
Canada	
apples, citrus, pears	10
potatoes	4
bananas (edible pulp)	0.4
sugar beet	0.1*
Chile	
citrus fruits, apples, pears	10
potatoes, sugar beet	5
cereals (raw)	0.2
tomatoes	0.1
milk, carcasses	0.1*
Denmark	
pome and stone fruits, parsley	10
citrus fruits	6
berries and small fruits, other fruits	3
other vegetables	2
potatoes	0.5
European Union	
banana	3
citrus fruit	6
pome fruit	5
potato, ware	5
Strawberry	**5**
Provisional MRLs set at LOD, generally 0.05 or 0.1 mg/kg, for various tree nuts, peaches, grapes, berries, root, bulb, and fruiting vegetables, cucurbits, brassicas, leafy and stem vegetables, legumes, pulses, oil seeds, early potatoes, tea, and hops established under Council Directive 95/38/EC. MRLs to be incorporated into national legislation by Member States.	
Finland	
citrus fruits	6
apples, pears	3
potatoes (industrial use)	1
bananas	0.4
cereal grains	0.2
other potatoes	0.1
Others	2
France	
citrus fruits	6
bananas	3
endive	05
Germany	
potatoes (washed)	4
pome fruits	3
cabbage, rape	1
cereals	0.2
other foods of plant origin (except bananas & citrus fruits)	0.1
Hungary	
lemon, grapefruit, orange, mandarin (Imports)	10
bananas (Imports)	3
bananas without peel (Imports)	0.4
Israel	
citrus fruit, pears, apples	10
celery	5
bananas, strawberries	3
Italy	
potatoes (washed)	4

Country, commodity	MRL, mg/kg
apples, pears	3
wheat, rice	0.2
onions, garlic	0.1
Japan*	
fruits (except citrus and banana)	3
vegetables	2
sugar beet	0.1
Kenya	
citrus fruit	6
bananas	3
bananas (pulp)	0.4
Mexico	
citrus (dry pulp)	35 (FA)
apples (post-harvest), Citrus fruit, pears (pre-harvest and post-harvest)	10
potatoes (pre-harvest and post-harvest)	3
rice	1
wheat straw	0.2
soya beans, wheat	0.1
sweet potatoes	02*
Netherlands	
pome fruit	10
potato	5
strawberry	3
tomato	2
cereal	0.2
onion	0.1
milk, meat	<0.1
other	<0.1
citrus fruit, banana *see* Preservatives Decision Commodities Act	
marmalade *see* Jam & Preservatives Decision Act	
New Zealand	
potatoes	10
bananas, citrus	3
meat	0.1
Romania	
eggs (without shells), whole milk, milk products, meat	0.1
South Africa	
potatoes, pineapples	10
apples, citrus, pears	6
avocados	5
bananas, muskmelon	3
mushrooms	1
(all tolerances in commodities for local use)	
Spain	
citrus fruit	6
washed potatoes	4
bananas, pome fruits, strawberries	3
cherries, tomatoes	2
all other plant products	0.1
Sweden	
apple, citrus fruits, pears	10
fruit & vegetables	6
potatoes	0.5
bananas (without peel)	0.4
Switzerland	
citrus fruits (whole)	10
bananas (whole)	3
bananas (pulp)	0.4
potatoes	01*
NOTE: Values for citrus fruit and bananas are not tolerances but are upper limits for	

Country, commodity	MRL, mg/kg
the food to be considered fit for human consumption.	
Taiwan	
citrus fruits (peel)	10
mushrooms, tropical fruits (peel)	5
root vegetables	3
rice	2
citrus fruits (pulp)	1
tropical fruits (pulp)	0.5
UK	
potatoes	5
USA-Raw Agricultural Commodities	
apple (post-harvest)	10
avocado	10
banana (Pre-harvest and post-harvest)	3
banana pulp (pre-harvest and post-harvest)	0.4
beans, dry	0.1
beets, sugar-without tops	0.25
beets, sugar-tops	10
cantaloupe	15
carrot (post-harvest)	10
citrus fruits	10
grape	10
mango	10
mushroom	40
papaya (post-harvest)	5
pear (post-harvest)	10
potato (pre-harvest and post-harvest)	10
rice, rough	3
rice, straw	10
soya bean	0.1
strawberry	5
sweet potato (post-harvest for seed use)	02
squash, Hubbard	1
wheat grain	1
wheat straw	1
fat of cattle, goats, hogs, horses, sheep	0.1
meat by products of cattle, goats, hogs, horses, poultry, sheep	0.1
meat of cattle, goats, hogs, horses, poultry, sheep	0.1
eggs	0.1
poultry	0.1
milk	0.4
wheat milled fractions (except flour)	3
apple pomace, dried (post-harvest)	33
beets, sugar, pulp (dried and/or dehydrated)	3.5
citrus molasses	20
citrus pulp, dried (post-harvest)	35
grape, pomace (dry or wet)	150
potato processing waste (Pre-harvest and post-harvest)	30
rice, hulls	8
wheat milled fractions (except flour)	3

NS = Not specified
FA = Feed Additive
P = Provisional
*At or about the limit of determination

Residues are usually defined as thiabendazole except in products of animal origin where they are usually defined as combined residues of thiabendazole and 5-hydroxythiabendazole.

APPRAISAL

Thiabendazole was evaluated by the Joint Meeting several times from 1970 to 1981, when MRLs were recommended for a number of commodities. The compound was evaluated by the present Meeting under the periodic review programme of the CCPR.

At its 1992 meeting JECFA noted that total residues of thiabendazole and 5-hydroxythiabendazole were below 0.1 mg/kg in all analysed tissues and milk within a few days of withdrawal and therefore adopted the definition of the residue and the MRLs of 0.1 mg/kg recommended by the 1975 JMPR for animal commodities and milk.

Thiabendazole is registered in many countries for use as a post-harvest and pre-harvest fungicide, veterinary drug and human medicine. The major use for plant protection is the post-harvest application.

The disposition of thiabendazole and its metabolites in humans and farm animals has been extensively studied. Many of the studies have also been published in the open literature. The oral administration of thiabendazole to sheep, cattle, goats, dogs and humans resulted in rapid absorption from the gastrointestinal tract. The time to achieve peak plasma levels varied with species and ranged from about 1 hour in dogs to 7 hours in sheep, goats and cattle. In dogs, goats and cattle, approximately 82% of the dose was excreted in the urine and faeces within the first 72 hours after oral administration. In all the species studied, almost all the recovered ^{14}C (97-99.6%) was in the urine and faeces. The hydroxylation of the benzimidazole ring at the 5-position to form 5-hydroxythiabendazole and subsequent conjugation to form the glucuronide and sulfate are the major metabolic steps. A minor metabolic pathway found in faeces and tissues involves loss of the thiazolyl group to form benzimidazole (BNZ). None of these residues are likely to persist in edible tissues in view of their relatively low concentrations and rapid elimination. Although the magnitude and profile of the residues differ slightly among different animal species (rats, lactating goats and laying hens), and samples (tissues, milk, eggs and excreta) the major metabolic steps and metabolites are the same.

Single gelatine capsules, each containing 120 mg of [^{14}C]thiabendazole, were administered daily to lactating goats for 7 consecutive days. Milk was collected twice daily and tissue samples after slaughter on the 8th day, within 24 hours after the final dose. An average of 74% of the administered dose was accounted for at the end of the study in the excreta (urine + faeces), tissues and milk, nearly all of it in the urine (69%) and faeces (28%). In urine, the residues, expressed as thiabendazole, consisted of unconjugated 5-hydroxythiabendazole (~7.9 mg/kg) and its *O*-sulfate conjugate (~9.5 mg/kg). The residues in the faeces consisted of unconjugated 5-hydroxythiabendazole (2.1 mg/kg), together with lower levels of benzimidazole (~0.4 mg/kg) and unmetabolized thiabendazole (~0.3 mg/kg). About 1% of the dose was found in the tissues. The highest tissue residues were in the liver and consisted of low levels of unmetabolized thiabendazole, unconjugated 5-hydroxythiabendazole and benzimidazole, at maximum concentrations of 0.2, 0.12 and 0.08 mg/kg respectively. Total residues in milk reached a steady state in 3 days and averaged about 1% of the orally administered dose (~1 mg/kg) after the final (7-day) dose. In milk the *O*-sulfate conjugate of 5-hydroxythiabendazole accounted for about 39% of the ^{14}C (0.4 mg/kg). No other individual residue was detectable (≤0.5% of the total radioactivity). Fractionation studies indicated that the unidentified residues were mainly products arising from the extensive degradation of thiabendazole followed by incorporation into proteins (20-60%), lipids (12-14%) and polysaccharides (~1%).

Single gelatine capsules, each containing 3.19 mg of [^{14}C]thiabendazole were orally administered daily to laying hens for 10 consecutive days; eggs and excreta were collected twice and once daily respectively. The hens were killed on the 11th day, within 24 hours after the final dose.

An average of 96.6% of the total administered dose was recovered. About 99.6% of this recovered dose was found in the excreta, and consisted of unconjugated (3.4 mg/kg) and conjugated (4.4 mg/kg) 5-hydroxythiabendazole. Cumulatively, the total residues found in the tissues and eggs accounted for about 0.4% or less of the ^{14}C. The total residues in eggs attained a level of about 0.1 mg/kg by day 2 and remained relatively unchanged throughout the next 8 days. The residues in tissues and eggs consisted mainly of unconjugated 5-hydroxythiabendazole, unmetabolized thiabendazole and benzimidazole at maximum concentrations, in the kidneys, of 0.4, 0.11 and 0.12 mg/kg respectively. The proposed metabolic pathway in poultry is the same as in goats.

Neither thiabendazole nor its related residues are likely to persist in milk, eggs or edible tissues because of their relatively low concentrations and rapid elimination.

The fate of [*phenyl*-^{14}C]thiabendazole was studied in actively growing wheat (2-3 tiller stage), soya beans (late flowering to early pod set) and sugar beet treated at maximum recommended rates (0.8, 0.68 and 2.015 kg ai/ha respectively). Residues were characterized after a combination of solvent (MeOH, $MeOH/H_2O$) and hydrolytic (KOH/MeOH) extractions, by reversed-phase HPLC and electron-impact GC-MS analyses. The same pattern of metabolites was seen in all three crops.

The total residues were about 0.12 mg/kg in wheat grain, 22 mg/kg in the straw, and 67.5 mg/kg in the foliage. Neither thiabendazole nor any individual metabolite was detectable in grain (≤0.05 mg/kg). The major individual residue found in the shoots was thiabendazole and the highest level, 65.6 mg/kg, was detected in early foliage. In all wheat tissues examined, only low proportions of the applied thiabendazole were converted to benzimidazole, which was subsequently conjugated with sugars. The benzimidazole could be released from the conjugate(s) by treatment with glucosidase. Benzimidazole was detected only in shoot tissues (<0.05 mg/kg in forage and 7.49 mg/kg in straw), either free or as the sugar conjugate(s). The highest level of unextractable residues was found in immature wheat forage (5.77 mg/kg), constituting about 14% of the total radioactive residue. The unextractable residues were distributed in very small amounts throughout several fractions of natural products, all of which were individually at or below the limit of detection (0.05 mg/kg). These results are consistent with findings in residue trials on wheat, including seed dressing and foliar treatments at or higher than the recommended rates with unlabelled thiabendazole, in which no residue (<0.05 mg/kg) was detectable in the grain at harvest. Since thiabendazole was present at higher levels than benzimidazole in growing wheat plants, the expected levels of benzimidazole in grain will also be undetectable (i.e. <0.05 mg/kg).

The aerial parts of actively growing soya bean crops were sprayed twice, at a 14-day interval, with [^{14}C]thiabendazole at a total rate of about 0.68 kg ai/ha. Immature samples (foliage and forage) were taken at intervals of 2 h and 27 days after treatment and mature samples were harvested and separated into grain and straw about 78 days after the first spray. The extractable residues were characterized by both reversed-phase HPLC and GC-MS. The total residues in the seed (~0.9 mg/kg) were less than 10% of those in the straw (~10 mg/kg). At day 27, thiabendazole was the single major residue (59% or 15.12 mg/kg) found in the shoots and benzimidazole-related compounds were present in smaller amounts (1.4% or 0.36 mg/kg). Benzimidazole was released from the conjugate(s) by glucosidase treatment. Thiabendazole (42.9% of the TRR) was the only individual residue detected (≥0.05 mg/kg) in the grain.

The foliage of actively growing sugar beet plants was sprayed five times, at 14-day intervals, with [^{14}C]thiabendazole at a total application rate of about 2.02 kg ai/ha. Immature top and root samples were taken about 2 h after the first and last treatments. About 90 days after the first treatment (35 days after the fifth and final spray) mature samples were harvested and separated into tops and roots. The residues were characterized by HPLC. At day 56 the organo-extractable residue in the roots was about 90% thiabendazole, amounting to 55.8% of the TRR. In the mature roots the total residues (~0.40 mg/kg) were about 4% of those in the tops (~10 mg/kg). The main component was the parent thiabendazole, at about 0.10 mg/kg; no other individual component was detectable (<0.05 mg/kg). A level of 2.7 mg/kg of thiabendazole was present in mature tops, where benzimidazole (1.4 mg/kg) was also present.

The distribution of the residues in wheat, soya beans and sugar beet is consistent with other results showing the predominantly axoplasmic movement of thiabendazole which results in measurable levels of thiabendazole residues in shoot tissues such as leaves and straw, and relatively less in storage tissues (grains and roots). It can be concluded that the profile and distribution of residues of thiabendazole in three representative actively growing crops (small grain, legume and root crops), following foliar applications, are the same.

The uptake, distribution and metabolism of thiabendazole by seed potatoes were studied under post-harvest storage conditions. Potatoes were briefly immersed in solutions of [^{14}C]thiabendazole at concentrations of 50, 100, 200 and 500 mg/kg and pH levels of 2-9. Skin and tissue sections were subsequently analysed. Potato tubers sorbed thiabendazole from aqueous solutions rapidly (within 5 minutes) at all pH levels. Thiabendazole penetrated only about 2 mm into the tubers in 2 weeks and a little more after 12 weeks, most of it (~96%) remaining on the outer skin. Even after 120 days of post-harvest storage, the only radioactive component detected was thiabendazole, accounting for over 80% of the applied ^{14}C. These results are supported by several additional studies indicating that thiabendazole does not penetrate into the fleshy tissues and does not undergo metabolic transformation. Benzimidazole was not detected (<0.05 mg/kg).

The uptake, distribution and residual fate of [^{14}C]thiabendazole under typical post-harvest storage conditions were also examined in Valencia oranges. Virtually all (~95%) of the radioactivity was sorbed by the peel and none penetrated into the inner pulp. Radiometric assays of the orange samples over the 28-day storage period demonstrated that practically all (~95%) of the radioactivity was due to thiabendazole itself although the conditions, at 21°C, were favourable for metabolism.

The post-harvest treatment studies on oranges, potatoes and pears gave similar results, showing sorption of thiabendazole by the outer surface of storage tissues without penetration into the fleshy interior.

The uptake of soil residues was studied in three representative crops: wheat (small grain), turnips (root) and lettuce (leafy vegetable). Three sandy loam plots were sprayed with [^{14}C]thiabendazole once, or twice two weeks apart, at a total application rate of 2.15 kg ai/ha representing the worst case that might occur in practice. The crops were harvested at maturity. After 137, 223 and 398 days, the extractable residues in the soil amounted to 75.3, 88.6, and 78.1% of the TRR respectively and thiabendazole accounted for 69.6, 86.9 and 63.2% of the TRR at these times. The residues were present in the upper 0 15 cm of the soil; no significant residues were found at 15-30 cm. The major components of the residues in the crops were thiabendazole (0.08 0.23 mg/kg in mature lettuce, 0.08-0.11 mg/kg in turnip roots, 0.63-1.0 mg/kg in turnip tops, <0.05-0.09 mg/kg in wheat grain, 2.61-10.25 mg/kg in wheat straw) and benzimidazole (0.03 mg/kg in mature lettuce, <0.05 mg/kg in turnip roots, 0.05-0.43 in turnip tops, <0.05 mg/kg in wheat grain, and 0.8-2.5 mg/kg in wheat straw), with the benzimidazole both free and as sugar conjugate(s). Lower levels of 5-hydroxythiabendazole (maximum 25-30% of the thiabendazole) were also observed in immature lettuce and wheat forage. Since 5-hydroxythiabendazole is a degradation product in soil, but not a plant metabolite, it is reasonable to conclude that it was produced in the soil and subsequently taken up by the crops. In addition to thiabendazole, benzimidazole, 5-hydroxythiabendazole and the unextractable residues, other radioactive components were also observed in the HPLC radio-chromatograms of various crop extracts, but all of them individually at levels below 0.05 mg/kg. The results demonstrate that the profile and distribution of thiabendazole residues in three representative crops (leafy vegetables, small grains and root crops) planted in treated soil are the same, but the composition of the residue is different from that in actively growing crops following foliar applications.

The fate of thiabendazole in microbially active sandy loam soil was studied under aerobic conditions at 25 ± 1°C. Thiabendazole was degraded with an aerobic half-life of about 737 days. The products consisted of low levels of benzimidazole (<2.5%) and 5-hydroxythiabendazole (<0.5%). Unextractable radiocarbon increased slowly during the study from 1.24% at day 0 to 20.2% at day 120. This increase is consistent with the strong binding of thiabendazole to soil. Volatile material, 96% of which was $^{14}CO_2$, also increased slowly, attaining its highest level after 12 months and accounting for 5.8% of the applied radioactivity. These results indicate that thiabendazole is fairly stable in soil but will eventually be mineralized under aerobic conditions to CO_2. Practically no degradation was observed under anaerobic conditions.

Thiabendazole was found to be photolytically stable on the surface of soil, with a calculated half-life of 933 days. Recoveries of ^{14}C from irradiated and unirradiated soil samples averaged about 98 and 104% respectively, and 90-100% of the radioactivity was due to thiabendazole; no other residue was found.

The adsorption of thiabendazole to soil was studied with silt loam, clay, sandy loam and sand. The results (K_{oc} values ranged from 1,104 to 22,467) indicate that thiabendazole is bound very tightly to soil. Similarly, the desorption of thiabendazole from these soils was also low, with K_{oc} values from about 1,336 to 18,325. Column leaching studies with the parent compound and residues aged on soil surfaces indicated that about 98% of the applied radioactivity remained in the top 2.5 cm of the column. On the basis of the high K_{oc} values and the column leaching studies, thiabendazole is considered to be immobile in soil.

[^{14}C]Thiabendazole was shown to be degraded rapidly in water when exposed to artificial sunlight, with a half-life of approximately 29 hours. The degradation resulted in the formation of benzimidazole-2-carboxamide (~10%), a polar fraction (8.6%) and relatively low levels (~6%) of benzimidazole. A minor degradation product, with HPLC retention properties consistent with a carboxybenzimidazole, was also present in trace amounts.

Analytical methods for determining residues from supervised trials have been validated with all the crops reported in this review. Validated methods are also available for analysing animal tissues and milk, as well as soil and water. The recoveries in food commodities were above 70% and the typical limits of detection and determination were 0.01-0.05 mg/kg and 0.05-0.1 mg/kg respectively.

Thiabendazole, free and conjugated 5-hydroxythiabendazole, and benzimidazole were found to be stable during frozen storage in crops for periods of 12 to 28 months, and in animal commodities for at least 2 months.

<u>Definition of the residue</u>

The studies carried out with labelled thiabendazole and related studies with the unlabelled material show that the only individual detectable residue (≥0.05 mg/kg) in edible crop commodities is likely to be the parent thiabendazole.

The animal metabolism and transfer studies indicate that thiabendazole and 5-hydroxythiabendazole are the major residue components in meat and eggs, while the sulfate conjugate, which was determined in all reported studies, is the major component in milk. The parent thiabendazole occurred at much lower concentrations in all commodities.

The Meeting concluded that the following definitions of the residue are appropriate.

<u>For compliance with MRLs</u>

For plant products: thiabendazole.
For animal products: sum of thiabendazole and 5-hydroxythiabendazole.

<u>For estimations of dietary intake</u>

For plant products: thiabendazole.
For animal products: sum of thiabendazole, 5-hydroxythiabendazole and its sulfate conjugate.

Post-harvest trials were conducted in the USA and Spain from 1990 to 1994 on oranges, lemons, grapefruit and tangerines. Ten trials were carried out on oranges in Spain with single post-harvest drench applications at 66 g ai/hl and 110 g ai/hl, and eight in the USA on citrus fruit with initial dip applications at

100 g ai/hl, followed by mist applications in wax with 350 or 500 g ai/hl at rates of 8.4 or 12 g ai/t fruit, much higher than the rates of 0.8 5.5 g ai/t specified on the labels. Residues of thiabendazole on unwashed whole fruit from the US trials in rank order were 1.2, 1.8, 2.9, 3.0, 3.8, 3.9, 4.8 and 5.4 mg/kg. The Spanish trials were reported in a summarized form which did not contain essential details and could not be used to estimate maximum residue levels.

Since there were no residue data from treatments according to GAP, the Meeting recommended the withdrawal of the existing CXL of 10 mg/kg.

Post-harvest residue trials were conducted in the USA (10) and Spain (5) in 1990-1991 on apples and pears. In the US trials, initial dip applications at 60 g ai/hl were followed by mist applications at 200 g ai/hl in wax (about twice the GAP concentration). The US labels provided do not include application in wax for pome fruits, however, in contrast to citrus fruits for which application in wax is specified. The residues on apples and pears (*) in the US trials were 0.89, 1.1* 3.0, 3.2, 3.2, 3.4, 3.4, 3.4, 3.7* and 5.1* mg/kg whole fruit. The trials in Spain were at 110 g ai/hl, the maximum GAP concentration, but were reported in a summarized form which did not contain essential details and they could not be used for the estimation of maximum residue levels.

Pre-harvest foliar applications on apples at four times the Japanese GAP rate gave rise to residues in the range 0.08-0.52 mg/kg.

As the trials were not according to national GAP, the Meeting recommended the withdrawal of the existing CXLs for apples and pears.

Pre-harvest residue trials on strawberries in Mexico, where there is no GAP, and Spain in 1989-1992 were with ground foliar applications of SC and WP formulations. In Mexico four applications were made 7 days apart, at rates of 0.50-2.0 kg ai/ha. In Spain a single application was carried out at 1.2 kg ai/ha (approx. 1.3 times GAP). The residues from the Spanish trials were 0.33 and 1.6 mg/kg at 3 days PHI. The data were insufficient to estimate a maximum residue level.

Residues following the post-harvest treatment of bananas were determined in a number of trials in Hawaii, Honduras and Guadeloupe. Residues in 10-20 replicate samples taken from individual treated lots indicated that the treatments were fairly uniform. The highest residues of the parent thiabendazole in each trial with 0.04 kg ai/hl in rank order were 0.79, 0.88, 1.0, 1.2, 1.4, 1.6, 1.7, 1.8, 2.3 and 3.3 mg/kg. Benzimidazole residues could not be detected in any samples. The dip treatments in Hawaii and Guadeloupe gave higher residues than the spray applications in Honduras. The pulp of ripened bananas from four trials contained average residues in the range 0.011-0.021 mg/kg which amounted to 1.3-2.9% of the residues measured in whole green bananas. The highest residues in individual samples from each trial in rank order were 0.016, 0.028, 0.029 and 0.031 mg/kg.

Since the use patterns (20-40 g ai/100 l) for post-harvest applications are very similar in a number of countries, the Meeting estimated a maximum residue level of 5 mg/kg for banana to replace the current CXL (3 mg/kg) and an STMR level of 0.029 mg/kg for banana pulp.

No information was provided on residues in onions. The Meeting therefore recommended the withdrawal of the CXL for bulb onions.

Four pre-harvest residue trials were conducted on tomatoes grown under plastic in Spain in 1990-1991 with ground spray foliar applications of SC and WP formulations. Two trials in 1990 were with two applications 7 days apart, at 0.50 kg ai/ha, and two trials in 1991 were with single applications at 3.1 kg ai/ha (approximately 3 times the GAP rate). The data were insufficient to estimate a maximum residue level and the Meeting recommended the withdrawal of the CXL for tomatoes.

Single dip or spray applications of SC and SL formulations of thiabendazole were used on chicory roots. Twenty trials were conducted with flowable SC and 20-S formulations at 67-630 g ai/hl. The chicory leaves, hearts and roots were all analysed for thiabendazole residues. Residues in the edible witloof chicory sprouts did not exceed 0.05 mg/kg even when the roots were treated at a sixfold rate.

The Meeting estimated a maximum residue level, at or about the limit of determination, of 0.05 mg/kg, and an STMR level of 0.05 mg/kg for witloof chicory (sprouts).

Post-harvest residue trials were conducted on potatoes. In seven trials in the UK whole potatoes were treated with a single spray mist application of a flowable formulation at 30-80 g ai/tonne. Potatoes in the US trials were subjected to an initial seed treatment at 2400 g ai/hl (approximately twice the GAP concentration) before cutting and planting, followed by an application of thiabendazole at 6.2 g ai/t (1.1 times the GAP rate) immediately after harvest and before storage, and a similar application about 30 days later. The residues of thiabendazole on unwashed potatoes from both sets of trials in rank order were 1.9, 2.0, 2.2, 2.4, <u>2.6</u>, <u>4.2</u>, 5.4, 5.5, 7.3 and 11 mg/kg.

The Meeting estimated a maximum residue level of 15 mg/kg and an STMR of 3.4 mg/kg for potato (adhering soil may be removed by rinsing or gentle brushing, to conform to the commodity to which Codex MRLs apply).

Pre-harvest residue trials on sugar beet were reported from Spain. One or two ground sprays were applied at 480 g ai/ha after development of 4-8 leaves. The residues of thiabendazole were <0.01 mg/kg in all 16 root samples taken from 0 to 91 days after the last application. The leaves and tops contained residues up to 0.41 mg/kg after 59-65 days. Since no GAP or processing studies were reported, the Meeting could not estimate maximum residue levels for sugar beet, sugar beet leaves or tops, molasses or dry pulp, and consequently recommended the withdrawal of the CXLs.

Mushrooms were treated with four applications of an aqueous solution by irrigation at 54-108 g ai/100 m^2 or by direct spray at 9.5-19 g ai/hl. Applications were made after pinning or after the first harvest break and then after the second, third and fourth breaks according to US label instructions. The maximum residues of thiabendazole on mushrooms collected 12 hours after the last application were 1.9, 2.2, 2.4, 2.5, 3.1, 3.2, 3.9, 3.9, 6.0, 6.1, 7.3, 8.0, 9.6, 12 and 13 mg/kg for irrigation and 21, 27, 30, 31, 36, 41 and 52 mg/kg for spray applications. The residues of benzimidazole were <0.01 mg/kg in all samples.

In four residue trials in Japan a WP formulation was applied once to the bed medium at a rate of 0.120 g ai/kg. The residues of thiabendazole were ≤0.25 mg/kg.

The Meeting evaluated the residues from direct spray applications according to US GAP and estimated a maximum residue level of 60 mg/kg and an STMR of 31 mg/kg for mushrooms.

In fourteen pre-harvest trials on wheat a single ground or aerial spray was applied at 620 g ai/ha (US GAP) after development of 2 to 3 tillers but before the first node, and the wheat was grown to harvest. The residues of thiabendazole and benzimidazole were <0.05 mg/kg in all 14 grain samples. The thiabendazole residues in the straw in rank order were <0.05 (11), 0.07, 0.11 and 0.13 mg/kg.

The Meeting noted that wheat readily takes up thiabendazole residues from soil (<0.05-0.09 mg/kg in wheat grain and 2.61-10.25 mg/kg in wheat straw grown in soil treated at 2.15 kg/ha). Although the pre-harvest use is limited and the application rates (up to 1 kg/ha except onion and garlic 1.4 kg ai/ha) are relatively low, the Meeting concluded that further field-scale rotational crop studies would be required before the pre-harvest use of the compound could be recommended and accordingly recommended the withdrawal of the CXL for cereal grains.

Animal transfer studies were conducted with poultry and cows. Ten groups of chickens (25 per group, males and females) were treated continuously for 7 weeks with thiabendazole at levels corresponding to 2, 20, 200 and 2000 ppm in the feed. Four males and 4 females at each treatment level were killed within four hours after the last dose and the liver, kidney, fat and muscle analysed for thiabendazole and 5-hydroxythiabendazole, as were eggs from the three highest treatment levels. The sum of thiabendazole and 5-hydroxythiabendazole, including its conjugate released by acid hydrolysis, was 0.02-0.028 mg/kg in fat (taken from different parts of the birds), 0.017-0.023 mg/kg in a 1:1 mixture of breast and leg meat, and 0.06-0.08 mg/kg in liver at the 20 mg/kg feed level (the expected level based on a poultry diet of 70% corn grain, 20% potatoes and waste and 10% wheat grain). At the same feeding level the average residues were 0.023-0.05 mg/kg in egg yolk and 0.007-0.023 mg/kg in egg white.

The Meeting noted the 3.4 mg/kg STMR for potatoes and the processing factor of 17 for processing potatoes to dry potato peel, and concluded that the 20 mg/kg feeding level appropriately covered the residues likely to occur in poultry feed. The Meeting estimated maximum residue and STMR levels of 0.05 mg/kg for poultry meat and 0.1 mg/kg for eggs.

Dairy cattle were treated once daily by capsule for 28 days with thiabendazole at levels corresponding to 25, 75 and 250 ppm in the feed. Milk samples were collected from all cows on days -1, 1, 2, 4, 7, 14, 21, 28, 29, 35, 42 and 56. Tissues and organs from two of the three cows in each treatment group were collected on day 29, and the remaining cow from each group was slaughtered on day 57. All the samples were analysed for thiabendazole and 5-hydroxythiabendazole. The residues in the milk reached plateaus two days after treatment of 0.014 mg/kg thiabendazole and 0.012 mg/kg 5-hydroxythiabendazole in the 25 ppm group and 0.017 mg/kg thiabendazole and 0.11 mg/kg 5-hydroxythiabendazole in the 250 ppm group, but these levels were less than 0.01 mg/kg higher than the control value at the 25 ppm feeding level, and below the limit of determination of the analytical procedure (0.05 mg/kg). The total residues of thiabendazole plus 5-hydroxythiabendazole in the cows of the 25 ppm group were <0.05 mg/kg in the milk and tissues except a single value of 0.05 mg/kg in kidney. At the 250 ppm level the residues were highest in kidney (0.024-0.03 mg/kg thiabendazole, 0.33-0.55 mg/kg 5-hydroxythiabendazole) and liver (0.056-0.08 thiabendazole, 0.12-0.16 mg/kg 5-hydroxythiabendazole), with much lower residues in the muscle and fat (0.014-0.017 mg/kg thiabendazole, 0.004-0.01 mg/kg 5-hydroxythiabendazole). No difference was observed between the thiabendazole residues in various meat tissues. The residues decreased rapidly to control levels when the animals were returned to a thiabendazole-free diet. The level of 25 ppm is a likely maximum rate, based on a diet of 50% maize grain, 25% apple pomace and 25% potato waste.

On the basis of the likely maximum residues in feed items the Meeting estimated maximum residue levels of 0.05 mg/kg for cattle meat and milk and 0.1 mg/kg for cattle edible offal, and STMRs of 0.05 mg/kg for all three commodities.

The metabolism study in goats at a level corresponding to approximately 20 ppm in the feed indicated much higher total residues of 1.1 mg/kg in milk (0.4 mg/kg 5-hydroxythiabendazole), 4.8 mg/kg in liver, 1.4 mg/kg in kidney and 0.1 mg/kg in meat. The Meeting concluded that further feeding studies would be required to estimate maximum residue levels in the meat, milk and edible offals of other animals, and recommended the withdrawal of the CXLs for milks and the meat and edible offals of goats, horses and sheep.

The effect of cold storage was studied with apples and potatoes after post-harvest treatment. The residues decreased during the first 24 hours but then remained relatively constant for 5 to 6 months.

The effects of processing were studied with post-harvest-treated apples, oranges and potatoes. Apples were treated with a post-harvest dip at 60 g ai/hl followed by a spray mist application in wax at 8.4 g ai/t approximately 30 days after cold storage. Whole fruits were processed into juice, wet pomace and dried pomace. The study could not be used to estimate processing factors, because a wax treatment is not specified on the label and the residues on whole unwashed apples were lower than on washed fruit, which cast doubt on the reliability of the results. Properly planned and executed processing studies representing typical industrial processes would be required before maximum residue levels could be estimated.

Oranges and grapefruit were treated with a post-harvest dip at 12 g ai/t followed by a spray mist application of thiabendazole in wax at 500 g ai/hl. The whole, washed fruits were processed into various fractions. The processing factors were 0.05 for juice and 8 for dried pomace.

The effect of home-processing on residues of thiabendazole in home-made marmalade was studied in the UK in 1993. The processing factors for home-made marmalade prepared in a preserving pan and in a microwave oven were 0.32 and 0.37 respectively.

Since no maximum residue level or STMR could be estimated for citrus fruits, no STMR-P levels could be estimated.

The effect of washing on the thiabendazole residues in potatoes was studied in several trials. The reduction of residues depended mainly on the time which elapsed between treatment and washing, and probably on the efficiency of washing which was not quantified. The processing (i.e. washing) factors calculated from the experiments in rank order were 0.05, 0.09, 0.11, 0.11, 0.12, <u>0.14</u>, <u>0.16</u>, 0.16, 0.17, 0.26 and 0.34 with a median of 0.15 and a mean of 0.13. Peeling removed a further substantial proportion of the residues in washed potatoes. The Meeting noted that residues are transferred from the peel to the peeled potatoes during peeling as potatoes peeled before washing contained average residues of 1.54 mg/kg and after washing 0.08 mg/kg. During industrial processing potatoes are always washed before peeling, and in a kitchen operation either before or after peeling or both. The Meeting therefore concluded that it is more appropriate to estimate the effect of peeling washed potatoes. The average ratio of the residue in pulp to that in washed potatoes was 0.045.

Since washing reduced the residues in raw potatoes by an average factor of 0.13 the Meeting estimated STMR-P levels of 0.44 mg/kg (3.4 x 0.13) for washed potatoes, and 0.02 mg/kg (0.44 x 0.045) for washed and peeled potatoes.

In processing trials in the USA seed potatoes were dipped in an aqueous suspension of thiabendazole containing 2400 g ai/hl before cutting and planting followed by a spray application to the daughter tubers at 6.2 g ai/t immediately after harvest and before cold storage, followed by a second application of 6.2 g ai/t approximately 30 days later. The processing of the potatoes involved washing, abrasive peeling, washing, slicing, washing, frying in vegetable oil at 178-182°C, de-oiling, and salting.

The effect of microwave and oven cooking on the residues of thiabendazole in or on potatoes was studied in the UK in 1990. Potatoes were treated post-harvest with a single application at 40 g ai/t. The tubers were stored for 182 days and the raw peel, raw pulp and unpeeled raw potatoes subjected to microwave and oven cooking.

In four trials on the effects of washing, boiling, baking and crisping in the UK in 1976 potatoes were treated post-harvest with single applications at 40 and 80 g ai/t. The tubers were stored for 4 and 21 days after treatment before processing.

The processing factors found in the US and UK trials were 1.13, 1.16, 1.2 (2) and 1.34, mean 1.2, for baked whole potatoes; 0.044, 0.055 and 0.073, mean 0.06, for potato chips; 0.03 for potato flakes and 17 for dried potato peel.

Since baking and frying do not change the residue content substantially, baked potatoes may be consumed with or without peel, and cooked or fried potatoes may be prepared in widely varying ways, the Meeting recommended the use of STMR-Ps for washed potato (0.44 mg/kg) and washed and peeled potato (0.02 mg/kg) for the assessment of dietary intake.

RECOMMENDATIONS

The studies carried out with labelled thiabendazole and other related studies with the unlabelled material show that the only residue detectable individually 0.05 mg/kg in edible crop commodities is likely to be the parent thiabendazole.

The animal metabolism and transfer studies indicate that thiabendazole and 5-hydroxythiabendazole are the major residue components in meat and eggs, while the sulfate conjugate, which was determined in all studies reported, is the major residue component in milk. The parent thiabendazole occurred at much lower concentration in all commodities.

The Meeting concluded that the following definitions of the residue are appropriate.

For animal products: sum of thiabendazole and 5-hydroxythiabendazole

<u>For estimations of dietary intake for compliance with MRLs</u>
For plant products: thiabendazole
For animal products: sum of thiabendazole, 5-hydroxythiabendazole and its sulfate conjugate

The Meeting estimated the following maximum residues and STMR levels. The maximum residue levels are recommended for use as MRLs.

Commodity		Recommended MRL, STMR mg/kg			PHI[1]
CCN	Name	New	Previous	STMR	
FP 0226	Apple	w	10		
FI 0327	Banana	5 PO	3	0.029[2]	
GC 0080	Cereal grains	w	0.2		
FC 0001	Citrus fruits	w	10 Po		
ML 0812	Cattle milk	0.05		0.05	
MM 0812	Cattle meat	0.05		0.05	
MO 0812	Cattle, edible offal of	0.1			
MO 0096	Edible offal of cattle, goats, horses, pigs & sheep	w	0.1*		
MM 0096	Meat of cattle, goats, horses, pigs & sheep	w	0.1*		
ML 0106	Milks		0.1*		
VO 0450	Mushroom	60		31	12 hrs
VA 0385	Onion, bulb	w	0.1		
FP 0230	Pear	w	10		
VR 0589	Potato	15	5 Po[3]	3.3 0.43 (P)[4] 0.02 (P)[5]	
PM 0110	Poultry meat	0.05	0.1	0.05	
PE 0112	Eggs	0.1	0.1	0.1	
FB 0275	Strawberry	w	3		
VR 0596	Sugar beet	w	5		
AV 0596	Sugar beet leaves and tops	w	10		
DM 0596	Sugar beet molasses	w	1		
AB 0596	Sugar beet pulp, dry	w	5		
VO 0448	Tomato	w	2		
VS0469	Witloof chicory (sprouts)	0.05*		0.05	Root treatment

PHI on which the recommendations are based
[1]STMR for banana pulp
[2]Washed before analysis

[3]STMR-P for washed potato
[4]STMR-P for washed and peeled potato

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ANNEX I

ACCEPTABLE DAILY INTAKES, ACUTE REFERENCE DOSES, RECOMMENDED MRLs, STMR LEVELS AND GLs RECORDED BY THE 1997 MEETING

The Table of recommendations is in two parts. Part 1 includes maximum Acceptable Daily Intakes (ADIs), Acute Reference Doses (RfDs), recommendations for Maximum Residue Limits (MRLs) and Supervised Trials Median Residue (STMR) levels. Part 2 comprises Guideline Levels (GLs) recorded for guazatine These were estimates of maximum residue levels, but cannot be recommended for use as MRLs because the Meeting withdrew the ADI for guazatine.

Some ADIs may be 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.)

In general, the MRLs recommended for compounds which have been reviewed previously are additional to, or amend, those recorded in the reports of earlier Meetings. If a recommended MRL is an amendment the previous value is also recorded. All recommendations for compounds re-evaluated in the CCPR periodic review programme are listed however (even if identical to existing CXLs or draft MRLs) because such re-evaluations replace the original evaluation rather than supplement it.

STMR levels were introduced in 1996 in response to recommendations of a Joint FAO/WHO Consultation on Guidelines for Predicting the Dietary Intake of Pesticide Residues held in York, UK, in 1995. The 1996 JMPR report explains the reasons for their introduction and gives details of the procedures used in their calculation (Sections 2.2.1, 2.2.3, Annex IV and the introduction to Annex I).

The Table includes the Codex reference numbers of the compounds and the Codex Classification Numbers (CCNs) of the commodities, to facilitate reference to the Guide to Codex Maximum Limits for Pesticide Residues and other Codex documents.

Commodities are listed in alphabetical order. This is a change from earlier 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.

The following qualifications and abbreviations are used. Some of the abbreviations are included in the list on pp. xi-xiv, but are repeated here for convenience.

* following recommended MRL	At or about the limit of determination
* following name of pesticide	New compound
** following name of pesticide	Compound reviewed in CCPR periodic review programme
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 meat	The recommendation applies to the fat of the meat.
P following an STMR level	An STMR for a processed commodity calculated by applying the mean processing factor for the process to the STMR calculated for the raw agricultural commodity.
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.
STMR	Supervised Trials Median Residue.
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	The recommendation accommodates veterinary uses.

recommendations for commodities of animal origin	
W in place of an MRL	The previous recommendation is withdrawn, or withdrawal of a CXL or draft MRL is recommended.

PART 1. ACCEPTABLE DAILY INTAKES (ADIs), RECOMMENDED MRLs AND SUPERVISED TRIALS MEDIAN RESIDUES (STMRs)

Pesticide (Codex ref. No.)	ADI (mg/kg bw)	Commodity		Recommended MRL or ERL (mg/kg)		STMR (mg/kg)
		CCN	Name	New	Previous	
Abamectin (177)	0.002[1]	TN 0660	Almond	0.01*	-	0
		AM 0660	Almond hulls	0.1	-	0.040
		FP 0226	Apple	0.02	-	0.003
		JF 0226	Apple juice			0.00019 P
		MO 0812	Cattle, Edible offal of	W	0.05	
		MF 0812	Cattle fat	0.1 V	-	
		MO 1289	Cattle kidney	0.05 V	Note[2]	
		MO 1281	Cattle liver	0.1 V	Note[2]	
		VC 0424	Cucumber	0.01	0.05	0.005
		DH 1100	Hops, dry	0.1	-	0.016
		VL 0482	Lettuce, Head	0.05	-	0.020
		VC 0046	Melons, except Watermelon	0.01*	-	0.002
		FP 0230	Pear	0.02	0.01*	0.005
		VR 0589	Potato	0.01*	-	0
		VC 0431	Squash, Summer	0.01*	-	0.002
		VO 0448	Tomato	0.02	0.02	0.0085
		TN 0678	Walnut	0.01*	-	0
		VC 0432	Watermelon	0.01*	-	0.002
			Apple sauce			0.00036 P
			Canned pears			0.00023 P
			Pear purée			0.00024 P
		Residue	(for MRLs and STMRs): sum of avermectin B_{1a}, avermectin B_{1b},, 8,9-Z-avermectin B_{1a} and 8,9-Z- avermectin B_{1b}			
		Notes	[1]For sum of abamectin and 8,9-*Z*- isomer. Previous ADIs were 0.001 mg/kg bw for Abamectin alone and 0.0002 mg/kg bw for the sum of abamectin and the 8,9-*Z*- isomer [2]Previous recommendation for cattle edible offal is replaced by recommendation for cattle kidney and cattle liver to accommodate JECFA recommendations arising from veterinary uses of abamectin			
Aminomethylphosphonic acid (AMPA) (198)	0.3[1]	GC 0645	Maize	2		
		AS 0645	Maize fodder	5		
		AF 0645	Maize forage	2		
		Residue	for MRLs: aminomethylphosphonic acid (AMPA) for STMRs: see glyphosate			
		Notes	[1]for sum of glyphosate and aminomethylphosphonic acid See also recommendations for glyphosate. AMPA is the main residue resulting from the treatment of genetically-modified maize with glyphosate.			
Amitrole	0.002	Note	Previous Temporary ADI 0.0005 mg/kg bw			

Pesticide (Codex ref. No.)	ADI (mg/kg bw)	Commodity CCN	Commodity Name	Recommended MRL or ERL (mg/kg) New	Recommended MRL or ERL (mg/kg) Previous	STMR (mg/kg)
(079)						
Captan	0.1	FP 0226	Apple	20	10	4.05
(007)		AB 0226	Apple pomace, dry	2	-	0.26 P
		FS 0013	Cherries	40	20	15
		DF 0269	Dried grapes (currants, raisins and sultanas)	50	-	10.4 P
		FB 0269	Grapes	25	20	6.1
		FB 0275	Strawberry	30	15	4.8
			Apple juice (unheated)			1.2 P
			Apple juice (heated)			0 P
			Apple sauce			0 P
			Grape juice			7.3 P
		Residue (for MRLs and STMRs): captan				
Carbofuran**	0.002	AL 1020	Alfalfa fodder	10	20	1.6
(096)		AL 1021	Alfalfa forage (green)	10	5	0.93
		FI 0327	Banana	0.1*	0.1*	0.1
		GC 0640	Barley	W	0.1*	
		VB 0402	Brussels sprouts	W	2	
		VB 0041	Cabbages, Head	W	0.5	
		VC 4199	Cantaloupe	0.2	-	0.02
		VR 0577	Carrot	W	0.5	
		MF 0812	Cattle fat	0.05*	0.05*	0.05
		VB 0404	Cauliflower	W	0.2	
		DM 0001	Citrus molasses[1]			0.11 P
		AB 0001	Citrus pulp, dry[1]	2	-	0.29
		SB 0716	Coffee beans	1	0.1*	0.1
			Coffee, Instant			0.005 P
		SM 0716	Coffee, Roast			0.005 P
		VC 0424	Cucumber	0.3	-	0.05
		MO 0096	Edible offal of cattle, goats, horses, pigs and sheep	0.05*	0.05*	0.05
		VO 0440	Egg plant	W	0.1*	
		MF 0814	Goat fat	0.05*	0.05*	0.05
		DH 1100	Hops, dry	W	5	
		MF 0816	Horse fat	0.05*	0.05*	0.05
		VB 0405	Kohlrabi	W	0.1*	
		VL 0482	Lettuce, Head	W	0.1*	
		GC 0645	Maize	W	0.1*	
		AS 0645	Maize fodder	W	5	
		MM 0096	Meat of cattle, goats, horses, pigs and sheep	0.05*	0.05*	0.05
		ML 0106	Milks	0.05*	0.05*	0.05
		SO 0090	Mustard seed	W	0.1*	
		GC 0647	Oats	W	0.1*	
		SO 0088	Oilseed	W	0.1*	
		VA 0385	Onion, Bulb	W	0.1*	
		FC 0004	Oranges, Sweet, Sour[1]	0.5	-	0.1
		JF 0004	Orange juice[1]			0.001
		FS 0247	Peach	W	0.1*	
		FP 0230	Pear	W	0.1*	
		MF 0818	Pig fat	0.05*	0.05*	0.05
		VR 0589	Potato	0.1	0.5	0.03
		CM 0649	Rice, Husked	W	0.2	
		MF 0822	Sheep fat	0.05*	0.05*	0.05
		GC 0651	Sorghum	0.1*	0.1*	0.01
		AF 0651	Sorghum forage (green)	2	-	0.065
		AS 0651	Sorghum straw and fodder, dry	0.5	-	0.055

Pesticide (Codex ref. No.)	ADI (mg/kg bw)	Commodity CCN	Commodity Name	Recommended MRL or ERL (mg/kg) New	Previous	STMR (mg/kg)
		VD 0541	Soya bean, dry	W	0.2	
		VC 0431	Squash, Summer	0.3	-	0.05
		FB 0275	Strawberry	W	0.1*	
		VR 0596	Sugar beet	W	0.1*	
		AV 0596	Sugar beet leaves or tops	W	0.2	
		GS 0659	Sugar cane	0.1*	0.1*	0.1
		SO 0702	Sunflower seed	0.1*	0.1*[2]	0.1
		VO 1275	Sweet corn (kernels)	W	0.1*	
		VO 0447	Sweet corn (corn-on-the -cob)	0.1	-	0.03
		VO 0448	Tomato	W	0.1*	
		GC 0654	Wheat	W	0.1*	
		Residue	for MRLs: sum of carbofuran and 3-hydroxy-carbofuran expressed as carbofuran for STMRs: sum of carbofuran and 3-hydroxy-carbofuran, free and conjugated, expressed as carbofuran Notes [1]Residues are from the use of carbosulfan [3]Previously included with Oilseeds (SO 0088) Periodic review was for residues only			
Carbosulfan**	0.01	DM 0001	Citrus molasses			0.0012 P
(145)		AB 0001	Citrus pulp, dry	0.1	-	0.0082 P
		JF 0004	Orange juice			0 P
		FC 0004	Oranges, Sweet, Sour	0.1	-	0.01
		Residue	(for MRLs and STMRs): carbosulfan Note Periodic review was for residues only			
Chlormequat	0.05	GC 0640	Barley	0.5	-	
(015)		AS O640	Barley straw and fodder, dry	20	50	
		SO 0691	Cotton seed	0.5	-	
		DF 0269	Dried grapes	W	1	
		FB 0269	Grapes	W	1	
		ML 0107	Milk of cattle, goats and sheep	W	0.1*	
			Milk products	W	0.1*	
		GC 0647	Oats	10	10	
		AF 0647	Oat forage (green)	20	-	
		AS 0647	Oat straw and fodder, dry	20	50	
		FP 0230	Pear	10	3	
		SO 0495	Rape seed	5	-	
		OC 0495	Rape seed oil, crude	0.1*	-	
		GC 0650	Rye	3	5	
		CM 0650	Rye bran, unprocessed	10	-	
		AF 0650	Rye forage (green)	20	-	
		AS 0650	Rye straw and fodder, dry	20	50	
		CF 1251	Rye wholemeal	3	-	
		GC 0654	Wheat	2	5	
		CM 0654	Wheat bran, unprocessed	5	-	
		CF 1211	Wheat flour	0.5	-	
		AS 0654	Wheat straw and fodder, dry	20	50	
		CF 1212	Wheat wholemeal	2	-	
		Residue:	chlormequat cation (usually used as the chloride) Notes Chlormequat was reviewed in the periodic review programme in 1994. The 1994 Meeting estimated the maximum residue levels listed above, but they were recorded only as GLs because the ADI was withdrawn. As an ADI has been allocated by the present Meeting the 1994 estimates are now recommended for use as MRLs.			
Chlorothalonil	0.03	FI 0327	Banana	0.01*[1]	W[2]	0
(081)		VD 0071	Beans, dry	0.2	-	0.02
		VB 0400	Broccoli	5	W[2]	2.25

Pesticide (Codex ref. No.)	ADI (mg/kg bw)	Commodity		Recommended MRL or ERL (mg/kg)		STMR (mg/kg)
		CCN	Name	New	Previous	
		HH 0624	Celery leaves	3	-	1.95
		FB 0021	Currants, Black, Red and White	5	W[2]	1.7
		HH 0740	Parsley	3	-	1.95
		FS 0247	Peach	0.2	1	0.01
		VO 0051	Peppers, Sweet	7	W[2]	1.5
		VO 0447	Sweet corn (corn-on-the-cob)	0.01*	W[2]	0.01
		Residue	for MRLs: chlorothalonil for STMRs, plant products: chlorothalonil for STMRs, animal products: sum of chlorothalonil and 4-hydroxy-2,5,6-trichloroisophthalonitrile, expressed as chlorothalonil			
		Notes	[1]Based on trials with bagged bananas [2]Withdrawal of existing MRL or CXL was recommended by 1993 JMPR Note changed definition of residue for STMRs for animal products			
Clethodim	0.01	AL 1020	Alfalfa fodder	10	-	1.6
(187)		VD 0071	Beans (dry)	W	0.1	0.05
		VP 0061	Beans, except broad bean and soya bean	0.5*	-	0.05
		MO 1280	Cattle kidney	0.2*	0.1	-
		MO 1281	Cattle liver	0.2*	0.1	-
		MM 0812	Cattle meat	0.5*	0.05*	-
		ML 0812	Cattle milk	0.1*	0.05*	-
		PE 0840	Chicken eggs	0.5*	0.05*	-
		PM 0840	Chicken meat	0.5*	0.05*	-
		OC 0691	Cotton seed oil, crude	0.5*	0.1	-
		OR 0691	Cotton seed oil, edible	0.5*	0 05	-
		VD 0561	Field pea (dry)	2	0.1	0.08
		AM 1051	Fodder beet	0.1*	-	0.03
		VA 0381	Garlic	0.5	-	0.1
		VA 0385	Onion, Bulb	0.5	-	0.1
		SO 0697	Peanut	5	-	1.3
		OC 0495	Rape seed oil, crude	0.5*	0.05	-
		OR 0495	Rape seed oil, edible	0.5*	0.05	-
		OR 0541	Soya bean oil, refined	0.5*	0.1	-
		VR 0596	Sugar beet	0.1	0.2	-
		SO 0702	Sunflower seed	W	0.2	-
		OC 0702	Sunflower seed oil, crude	W	0.05	-
		OR 5702	Sunflower seed oil, edible	W	0.05	-
		VO 0448	Tomato	1	-	0.35
		Residue	(for MRLs and STMRs): sum of clethodim and its metabolites containing the 5-(2-ethylthiopropyl)cyclohexene-3-one and 5-(2-ethylthiopropyl)-5-hydroxycyclohexene-3-one moieties and their sulfoxides and sulfones, expressed as clethodim			
Ethephon (106)	0.05	Note	ADI unchanged			
Fenamiphos** (085)	0.0008	Notes	Previous ADI 0.0005 mg/kg bw Periodic review was for toxicology only			
Fenbuconazole*	0.03	FS 0240	Apricot	0.5	-	0.25
(197)		FI 0327	Banana	0.05	-	0.01
		GC 0640	Barley	0.2	-	0.03
		AS 0640	Barley straw and fodder, dry	3	-	0.62
		MF 0812	Cattle fat	0.05*	-	0
		MO 1280	Cattle kidney	0.05*	-	0
		MO 1281	Cattle liver	0.05	-	0.01

Pesticide (Codex ref. No.)	ADI (mg/kg bw)	Commodity CCN	Name	Recommended MRL or ERL (mg/kg) New	Previous	STMR (mg/kg)
		MM 0812	Cattle meat	0.05*	-	0.01
		ML 0812	Cattle milk	0.05*	-	0.01
		FS 0013	Cherries	1	-	0.36
		VC 0424	Cucumber	0.2	-	0.02
		PE 0112	Eggs	0.05*	-	0
		JF 0269	Grape juice	-	-	0.03 P
		FB 0269	Grapes	1	-	0.3
		VC 0046	Melons, except watermelon	0.2	-	0.025
		FS 0247	Peach	0.5	-	0.25
		TN 0672	Pecan	0.05*	-	0.01
		FP 0009	Pome fruits	0.1	-	0.025
		PM 0111	Poultry, Edible offal of	0.05*	-	0
		PF 0111	Poultry fats	0.05*	-	0
		PM 0110	Poultry meat	0.05*	-	0
		SO 0495	Rape seed	0.05*	-	0.05
		GC 0650	Rye	0.1	-	0.02
		VC 0431	Squash, Summer	0.05	-	0.02
		SO 0702	Sunflower seed	0.05*	-	0.02
		GC 0654	Wheat	0.1	-	0.02
		CM 0654	Wheat bran, unprocessed	-	-	0.052 P
		CF 1211	Wheat flour	-	-	0.005 P
		AS 0654	Wheat straw and fodder, dry	3	-	0.79
		CP 1211	White bread	-	-	0.0092 P
			Wine	-	-	0.018 P
		Residue	(for MRLs and STMRs): fenbuconazole (fat-soluble residue)			
Fenthion (039)	0.007[1]	Notes	Acute RfD: 0.01mg/kg bw. Previous acute RfD was 0.007 mg/kg bw			
Fipronil*	0.0002	Notes	Acute RfD 0.003 mg/kg bw Evaluation was for toxicology only			
Fipronil-desulfinyl	0.00003 T (2000)	Note	Acute RfD 0.003 mg/kg bw			
Folpet	0.1	FP 0226	Apple	10	-	1.8
		JF 0226	Apple juice			0.063 P
(041)		DF 0269	Dried grapes (currants, raisins and sultanas)	40	-	7.0 P[1]
		JF 0269	Grape juice			0.0066 P
		FB 0269	Grapes	10	2	2.2
		VC 0046	Melons (except Watermelon)	3	-	0.41
		VO 0448	Tomato	3	-	1.15
			Apple pomace, wet			4.7 P
			Must			0.64 P
			Raisins, hydrated			4.2 P
			Tomato paste			0.032 P
			Tomato purée			0.032 P
			Wine			0 P
		Residue	(for MRLs and STMRs): folpet			
Glyphosate	0.3	SO 0691	Cotton seed	10	0.5	2.0
(158)			Cotton seed, delinted			0.38 P
			Cotton seed, kernels			0.17 P
			Cotton seed hulls			0.68 P
			Cotton seed meal			0.24 P
		OC 0691	Cotton seed oil, crude	0.05*		0 P
		OR 0691	Cotton seed oil, edible	0.05*		0 P
		GC 0645	Maize	1	0.1*	0.47
		AF 0645	Maize forage	1		0.81

Pesticide (Codex ref. No.)	ADI (mg/kg bw)	Commodity		Recommended MRL or ERL (mg/kg)		STMR (mg/kg)
		CCN	Name	New	Previous	
		GC 0651	Sorghum	20	0.1*	5.8
			Sorghum, cleaned			7.0 P
			Sorghum bran			27 P
			Sorghum flour			2.1 P
			Sorghum grain dust			27 P
			Sorghum grits (medium)			2.8 P
			Sorghum germ			0 P
			Sorghum starch			0 P
		Residue	for MRLs: glyphosate for STMRs: sum of glyphosate and aminomethylphosphonic acid (AMPA), expressed as glyphosate			
		Note	See also recommendations for aminomethylphosphonic acid. AMPA is the main residue resulting from the treatment of genetically-modified maize with glyphosate.			
Guazatine** (114)	**ADI Withdrawn**	Note	Maximum residue levels estimated by the present Meeting are recorded as GLs. See Part 2 of the Table			
Lindane (048)	0.001 T (200)	Note	Previous ADI 0.008 mg/kg bw			
Malathion** (049)	0.3	Notes	Previous ADI 0.02 mg/kg bw Periodic review was for toxicology only			
Methamidophos	0.004	FP 0009	Pome fruits	0.5	0.5	0.18
(100)		Residue	(for MRLs and STMRs): methamidophos			
		Note	methamidophos is a metabolite of acephate for which separate MRLs are recommended			
Methidathion (051)	0.001	Note	Acute RfD 0.01 mg/kg bw			
Mevinphos**	0.0008	FP 0226	Apple	W	0.5	
(053)		FS 0240	Apricot	W	0.2	
		VB 0400	Broccoli	W	1	
		VB 0402	Brussels sprouts	W	1	
		VB 0041	Cabbages, Head	0.05	1	0.02
		VR 0577	Carrot	W	0.1	
		VB 0404	Cauliflower	W	1	
		FS 0013	Cherries	W	1	
		FC 0001	Citrus fruits	W	0.2	
		VP 0526	Common bean (pods and/or immature seeds)	0.05	0.1	0.02
		VC 0424	Cucumber	W	0.2	
		FB 0269	Grapes	W	0.5	
		VL 0480	Kale	W	1	
		VA 0384	Leek	0.02*	-	0.02
		VL 0482	Lettuce, Head	W	0.5	
		VC 0046	Melons, except watermelon	W	0.05	
		VA 0385	Onion, Bulb	W	0.1	
		FS 0247	Peach	W	0.5	
		FP 0230	Pear	W	0.2	
		VP 0063	Peas (pods and succulent = immature seeds)	W	0.1	
		VR 0589	Potato	W	0.1	
		VL 0502	Spinach	W	0.5	
		FB 0275	Strawberry	W	1	
		VO 0448	Tomato	W	0.2	
		VR 0506	Turnip, Garden	W	0.1	
		Residue	(for MRLs and STMRs): sum of (*E*)- and (Z)-mevinphos			

Pesticide (Codex ref. No.)	ADI (mg/kg bw)	Commodity		Recommended MRL or ERL (mg/kg)		STMR (mg/kg)
		CCN	Name	New	Previous	
		Note	Periodic review was for residues only			
Myclobutanil	0.03	FS 0240	Apricot	W	0.2	
(181)		FB 0278	Blackcurrant	0.5	-	0.26
		JF 1140	Blackcurrant juice			0.09 P
		FS 0013	Cherries	W	1	
		FS 0247	Peach	W	0.5	
		FS 0012	Stone fruits, except plums	2	-	0.62
		FB 0275	Strawberry	1	-	0.18
		VO 0448	Tomato	0.3	-	0.05
		JF 0448	Tomato juice			0.05 P
			Strawberry jam			0.09 P
			Strawberry preserve			0.15 P
			Tomato, canned			0.05 P
			Tomato, dry pomace			0.78 P
			Tomato paste			0.2 P
			Tomato purée			0.08 P
		Residue	(for MRLs and STMRs): myclobutanil			
Phosalone (060)	0.02	Notes	Previous ADI 0.001 mg/kg bw			
Phosmet**	0.01	AL 1020	Alfalfa fodder	W	40	
(103)		AL 1021	Alfalfa forage, green	W	40	
		FP 0226	Apple	10	10	3.4
		FS 0240	Apricot	10	5	2.9
		FB 0020	Blueberries	W	10	
		MM 0812	Cattle meat	W	1 (fat) V	
		FC 0001	Citrus fruits	W	5	
		SO0691	Cotton seed	0.05	-	0
		FI 0335	Feijoa	W	2	
		FB 0269	Grapes	10	10	3.1
		FI 0341	Kiwifruit	W	15	
		GC 0645	Maize	W	0.05	
		AS 0645	Maize fodder	W	10	
		AF O645	Maize forage	W	10	
		ML 0106	Milks	W	0.02 V	
		FS 0245	Nectarine	W	5	
		AL 0072	Pea hay or fodder (dry)	W	10	
		AL 0528	Pea vines (green)	W	10	
		FS 0247	Peach	10	10	2.9
		FP 0230	Pear	W	10	
		VP 0063	Peas (pods and succulent = immature seeds)	W	0.2	
		VD 0063	Peas (dry)	W	0.02*	
		VR 0589	Potato	0.05*	0.05	0.05
		VO 0447	Sweet corn (corn-on-the-cob	W	0.05	
		VR 0508	Sweet potato	W	10 Po	
		TN 0085	Tree nuts	W	0.1	
		Residue	(for MRLs and STMRs): phosmet			
		Notes	Changed definition of residue Residue is not fat-soluble, although it was previously so described Periodic review was for residues only			
Tebuconazole	0.003	FI 0327	Banana	0.05	-	0.01[1]
(189)		FS 0013	Cherries	5	-	0.76
		VC 0424	Cucumber	0.2	-	0.035
		DF 0269	Dried grapes (currants, raisins and sultanas)	3	-	

Pesticide (Codex ref. No.)	ADI (mg/kg bw)	Commodity		Recommended MRL or ERL (mg/kg)		STMR (mg/kg)
		CCN	Name	New	Previous	
		GC 0647	Oats	0.05*	-	0
		FS 0247	Peach	1	-	0.21
		VO 0445	Peppers, Sweet	0.5	-	0.14
		FP 0009	Pome fruits	0.5	-	0.12
	0.003	Residue	(for MRLs and STMRs): tebuconazole Note [1]In the edible portion			
Tebufenozide	0.02	FI 0341	Kiwifruit	0.5	-	0.14
(196)		Residue	(for MRLs and STMRs): tebufenozide (fat-soluble residue)			
Thiabendazole**	0.1	FP 0226	Apple	w	10	
(065)		FI 0327	Banana	5 Po	3	0.029[1]
		GC 0080	Cereal grains	w	0.2	
		FC 0001	Citrus fruits	w	10 Po	
		MO 0812	Cattle, Edible offal of	0.1		0.05
		MM 0812	Cattle meat	0.05		0.05
		ML 0812	Cattle milk	0.05		0.05
		MO 0096	Edible offal of cattle, goats, horses, pigs & sheep	w	0.1*	
		PE 0112	Eggs	0.1	0.1	0.1
		MM 0096	Meat of cattle, goats, horses, pigs & sheep	w	0.1*	
		ML 0106	Milks	W	0.1*	
		VO 0450	Mushroom	60		31
		VA 0385	Onion, Bulb	w	0.1	
		FP 0230	Pear	w	10	
		VR 0589	Potato	15	5 Po[2]	3.4
		PM 0110	Poultry meat	0.05	0.1	0.05
		FB 0275	Strawberry	w	3	
		VR 0596	Sugar beet	w	5	
		AV 0596	Sugar beet leaves and tops	w	10	
		DM 0596	Sugar beet molasses	w	1	
		AB 0596	Sugar beet pulp, dry	w	5	
		VO 0448	Tomato	w	2	
		VS0469	Witloof chicory (sprouts)	0.05*		0.05
			Potato, washed			0.44 P
			Potato, washed and peeled			0.02 P
		Residue	for MRLs, plant products: thiabendazole for MRLs, animal products: sum of thiabendazole and 5-hydroxythiabendazole for STMRs, plant products: thiabendazole for STMRs, animal products: sum of thiabendazole, 5-hydroxythiabendazole and 5-hydroxythiabendazole sulfate Notes [1]for banana pulp [2]washed before analysis Periodic review was for residues only			
Triforine** (116)	0.02	Notes	ADI unchanged Periodic review was for toxicology only			

PART 2. GUIDELINE LEVELS RECORDED FOR COMPOUNDS WITHOUT ADIs

Pesticide (Codex ref. no.)	Commodity		GL, mg/kg	Previous MRL, mg/kg	STMR, mg/kg
	CCN	Name			
Guazatine	FC 0001	Citrus fruits	W	5 Po	
(114)	FI 0353	Pineapple	W	0.1*	
	GC 0080	Cereal grains	0.05*	0.1*	0
	GS 0659	Sugar cane	W	0.1*	
	VC 0046	Melons (except Watermelon)	W	5 Po	
	VR 0589	Potato	W	0.1*	
	Residue	for GLs: octane-1,8-diyldiguanidine ("GG"), expressed as octane-1,8-diyldiguanidine for STMRs: guazatine			

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. 1969 Evaluation of some pesticide residues in food.

1970b 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.

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