ISSN 2070--2515

FAO
PLANT
PRODUCTION
AND PROTECTION
PAPER

142

Food and Agriculture
Organization of the
United Nations

World Health
Organization

Pesticide residues in food 1996

Joint FAO/WHO Meeting on Pesticide Residues

EVALUATIONS

1996

PART I - RESIDUES

Rome, 1997

CONTENTS

[1] * First evaluation
** Evaluation in CCPR periodic review programme

1996 JOINT MEETING OF THE FAO PANEL OF EXPERTS ON PESTICIDE RESIDUES IN FOOD AND THE ENVIRONMENT AND THE WHO CORE ASSESSMENT GROUP

Rome, 16-25 September 1996

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ABBREVIATIONS WHICH MAY BE USED

Ache	acetylcholinesterase
ADI	acceptable daily intake
AFI(D)	alkali flame-ionization (detector)
ai	active ingredient
ALAT	alanine aminotransferase
approx.	approximate
ASAT	aspartate aminotransferase
BBA	Biologische Bundesanstalt für Land- und Forstwirtschaft
bw (not b.w.)	body weight
c	centi- (x 10^{-2})
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
ChE	cholinesterase
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
EBDC	ethylenebis(dithiocarbamate)
EC	(1) emulsifiable concentrate (2) electron-capture [chromatographic detector]
ECD	electron-capture detector
EMDI	estimated maximum daily intake
EPA	Environmental Protection Agency
ERL	extraneous residue limit
ETU	ethylenethiourea
F_1	filial generation, first
F_2	filial generation, second
f.p.	freezing point
FAO	Food and Agriculture Organization of the United Nations
FDA	Food and Drug Administration
FID	flame-ionization detector
FPD	flame-photometric detector
g (not gm)	gram

□g	microgram
GAP	good agricultural practice(s)
GC-MS	gas chromatography - mass spectrometry
G.I.	gastrointestinal
GL	guideline level
GLC	gas-liquid chromatography
GLP	Good Laboratory Practice
GPC	gel-permeation 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
IBT	Industrial Bio-Test Laboratories
i.d.	internal diameter
i.m.	intramuscular
i.p.	intraperitoneal
IPCS	International Programme on Chemical Safety
IR	infrared
IRDC	International Research and Development Corporation (Mattawan, Michigan, USA)
i.v.	intravenous
JMPR	Joint FAO/WHO Meeting on Pesticide Residues (Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and 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
□m	micrometre (micron)
min	minute(s)
MLD	minimum lethal dose
M	molar
mo	month(s)

(not mth.)	
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
MTD	maximum tolerated dose
n	normal (defining isomeric configuration)
NCI	National Cancer Institute (United States)
NMR	nuclear magnetic resonance
NOAEL	no-observed-adverse-effect level
NOEL	no-observed-effect level
NP(D)	nitrogen-phosphorus (detector)
NTE	neuropathy target esterase
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
s.c.	subcutaneous
SC	suspension concentrate (= flowable concentrate)
SD	standard deviation
SE	standard error
SG	water-soluble granule
SL	soluble concentrate
SP	water-soluble powder
sp./spp.	species (only after a generic name)
sp gr	specific gravity
(not sp. gr.)	
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
v/v	volume ratio (volume per volume)
WG	water-dispersible granule
WHO	World Health Organization
WP	wettable powder
wt/vol	weight per volume
w/w	weight ratio (weight per weight)
<	less than
≤	less than or equal to
>	greater than
≥	greater than or equal to
*	(following residue levels, e.g. 0.01* mg/kg): level at or about the limit of determination

ESTIMATION OF THE DIETARY INTAKE OF PESTICIDE RESIDUES

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 Joint Meeting extended its estimations of residues to include calculations of the median residue levels found in supervised trials (STMR levels) in order to provide a basis for the estimation of the dietary intake of the pesticides reviewed.

The estimated STMRs are included in the Tables of Recommendations in the Appraisals of the compounds reviewed and in the Table of ADIs and MRLs in Annex I. Further details of the response of the 1996 JMPR to the York Consultation and information about an informal workshop held in The Hague, The Netherlands, in April 1996 to consider the implementation of the York recommendations by the JMPR are given in Sections 2.2.1 and 2.2.3 of the report of the Meeting.

The report of the Hague Workshop is reproduced as Annex III to these evaluations. It explains in detail the procedures used in the calculation of STMRs and directs attention to the associated changes in the Tables of residues resulting from supervised trials and in the presentation of the Appraisals.

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 Rome, 16-25 September 1996, contains a summary of the evaluations of residues in foods of the various pesticides considered as well as information on the general principles followed by the Meeting. The present document contains summaries of the residues data considered, together with the recommendations made.

The Evaluations are issued in two parts:

Part I: Residues (by FAO)

Part II: Toxicology (by WHO)

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

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

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

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

Acknowledgements

The monographs in these Evaluations were prepared by the following participants in the 1996 JMPR for the FAO Panel of Experts on Pesticide Residues in Food and the Environment:
Dr. A. Ambrus, Dr. U. Banasiak, Mr. S. Crossley, Mr. D.J. Hamilton, Mr. N.F. Ives, Mr. A.F. Machin, Ms. E. Masoller and Mr. T. Sakamoto.

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

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Viale delle Terme di Caracalla
00100 Rome, Italy

ACEPHATE (095)

[see also METHAMIDOPHOS]

EXPLANATION

Acephate was first evaluated in 1976, with further reviews of residue aspects in 1979, 1981, 1984, 1990 and 1994. The 1994 JMPR withdrew the previous recommendations for the MRLs for broccoli, Brussels sprouts, head cabbages, cauliflower, citrus fruits and tomato which had been held at Step 7B by the 1989 CCPR (ALINORM 89/24A, para 126). The manufacturer has indicated that information on GAP and residue data would be available to support new MRLs for these commodities. This information was provided to the Meeting, together with information on analytical methods and residues in food in commerce or at consumption.

METHODS OF RESIDUE ANALYSIS

Analytical methods

In the supervised trials homogenized samples were extracted with ethyl acetate, cleaned up on a silica gel column and determined by GLC. If necessary, additional clean-up was by acetonitrile-hexane partitioning. Acephate and methamidophos were determined separately in the same sample (Lai and Fowler, 1989). Recoveries of both acephate and its metabolite methamidophos were generally >70%, and the limit of determination was 0.01-0.02 mg/kg.

Stability of pesticide residues in stored analytical samples

The stability of acephate and methamidophos was studied in vegetables, pulses, oilseed, animal products, cereals and grasses for periods ranging from 28 days to more than a year at -20°C. All samples except pinto beans and eggs were taken from crops which had been treated with acephate or from animals which had been dosed with acephate. Pinto beans and eggs were fortified with acephate and methamidophos (Lai, 1987, 1988, 1989a).

In general, acephate was stable in a wide range of macerated or ground commodities when stored at -20°C, even for periods exceeding a year. The results are summarized in Table 1.

Table 1. Stability of acephate and methamidophos in samples stored at -20°C.

Commodity	Compound[1]	Storage period, days	Initial concentration[2], mg/kg	% of initial residue remaining	Reference
Celery	A	364	0.26-4.40	87-97	35
	M	364	0.02-0.29	243-300	
Celery	A	94	4.16-4.40	106-116	34
	M	94	0.23-0.29	93-148	
Snap beans	A	69	0.30-0.39	73.3-84.6	
	M	69	0.12-0.15	75.0-86.7	
Snap beans	A	548	0.30-0.39	76.7-82.1	36
	M	548	0.12-0.15	75.0-80.0	
Pinto beans (dry)	A	461	0.23-0.24[3]	95.0-95.0	
	M	461	0.09-0.10[3]	80.0-90.0	
Pigeon peas	A	418	8.11-9.74	104-110	34
	M	418	0.94-1.07	108-111	
Bell peppers	A	386	3.67-3.83	103-112	
	M	386	0.51-0.53	131-136	
Brussels sprouts	A	272	1.61-2.06	84-88	
	M	272	0.03-0.03	100-100	
Lettuce	A	28	0.29-0.31	84-93	
	M	28	0.02-0.02	50-100	
Cotton seed	A	48	0.38-0.82	73.2-86.8	
	M	48	0.02-0.03	0.0-0.0	
Grass	A	269	0.52-0.70	78.6-100	
	M	269	0.10-0.14	78.6-90	
Bermuda grass	A	61	0.62-0.72	108-122	
	M	61	0.11-0.11	109-117	
Bermuda grass	A	60	1.88-2.85	98-102	
	M	60	0.31-0.44	102-106.5	
Fresh hay	A	58	6.95-7.36	72.0-85.8	
	M	58	0.49-0.54	75.5-83.3	
Spent hay	A	58	2.81-2.91	96.2-96.4	
	M	58	0.33-0.36	90.9-91.7	
Rice grain	A	506	1.09-1.19	81-126	
	M	506	0.21-0.23	96-124	
Rice straw	A	507	0.17-0.21	90-94	
	M	507	0.06-0.06	83-83	
Eggs	A	175	0.15-0.16[3]	96.8-103	
	M	175	0.07-0.08[3]	93.3-93.3	
Cow milk	A	202	0.04-0.79	98.7-150	
	M	202	0.02-0.12	58.3-100	
Cow kidneys	A	172	0.26-0.73	71.2-73.1	
	M	172	0.02-0.07	50.0-60.0	
Cow muscle	A	193	0.11-0.40	90.5-112	
	M	193	0.01-0.03	100-100	

[1] A: acephate M: methamidophos

[2] Initial concentrations were the residues found in the commodity at harvest or collection, except pinto beans and eggs which were added to untreated commodities

[3] Fortified separately with acephate and methamidophos

USE PATTERN

Information on use patterns was provided by the governments of The Netherlands and Poland and the manufacturers. The use patterns for citrus fruits, broccoli, Brussels sprouts, head cabbages, cauliflowers and tomatoes are summarized in Table 2.

Table 2. Registered uses of acephate on citrus fruits, broccoli, Brussels sprouts, head cabbages, cauliflowers and tomatoes.

Crop	Country	Form	Application					PHI, days
			Type	kg ai/ha	kg ai/hl	No.	Interval, days	
Citrus fruits	Italy	SP	Spray		0.024-0.036			21
	Japan	WP	Spray		0.025-0.05	3		30
	New Zealand	SP	Spray		0.075			14
	Venezuela	SP	Spray		0.06-0.12			5
Broccoli	Australia	SP	Spray	0.98	0.098	-	10-14	14
	Brazil	SP	Spray	0.38-0.75	0.075			14
	Italy	SP	Spray		0.034-0.064			21
	Japan	WP	Spray		0.05	3		14
	South Africa	SP	Spray	0.26-0.38			7-10	3
	Venezuela	SP	Spray	0.38-0.75				5
Brussels sprouts	Australia	SP	Spray	0.98	0.098	-	10-14	3
	Netherlands	WP	Spray	0.75		6	7	28
	South Africa	SP	Spray	0.26-0.38			7-10	3
	USA	SP	Spray	0.56-1.1				14
Cabbages	Australia	SP	Spray	0.98	0.098	-	10-14	3
	Brazil	SP	Spray	0.38-0.75	0.075			14
	France	WP	Spray		0.075			7
	Japan	WP	Spray		0.025-0.05	3		7
	Netherlands	WP	Spray	0.75		6	7	14
	New Zealand	SP	Spray	0.75-1.1	0.075		7-10	7
	Poland	SP	Spray	0.50-0.75		2		14
	South Africa	SP	Spray	0.26-0.38			7-10	3
	Venezuela	SP	Spray	0.38-0.75				5
Cauliflowers	Australia	SP	Spray	0.98	0.098	-	10-14	3
	Brazil	SP	Spray	0.38-0.75	0.075			14
	Italy	SP	Spray		0.034-0.064			21
	Japan	WP	Spray		0.05	3		14
	Netherlands	WP	Spray	0.75		6	7	14
	New Zealand	SP	Spray	0.75-1.1	0.075		7-10	7
	South Africa	SP	Spray	0.26-0.38			7-10	3
	USA	SP	Spray	0.56-1.1				14
	Venezuela	SP	Spray	0.38-0.75				5
Tomatoes	Australia	SP	Spray	0.98	0.098	-	10-14	3
	Brazil	SP	Spray	0.38-0.75	0.075			7

Crop	Country	Form	Application					PHI, days
			Type	kg ai/ha	kg ai/hl	No.	Interval, days	
	Canada	SP	*	0.9	0.045			*
	Japan	WP	Spray		0.025-0.05	3		1
Tomatoes	New Zealand	SP	Spray	0.75	0.075		14	3
	Poland	SP	Spray	0.75		1		14
	Poland (Glasshouse)	SP	Spray		0.075	1		14
	South Africa	SP	Spray	0.56-1.5	0.056-0.075		10	3
	Spain	SP	Spray		0.038-0.11			14
	Venezuela	SP	Spray	0.38-0.75				5

* Transplant water treatment: no PHI specified

RESIDUES RESULTING FROM SUPERVISED TRIALS

Data from many supervised trials on citrus fruits, broccoli, Brussels sprouts, head cabbages, cauliflowers and tomatoes were submitted or resubmitted to the Meeting. However some reports lacked important information such as recovery data or representative chromatograms. The Meeting did not evaluate trials which lacked data on analytical recoveries or in which recoveries were below 70%, or trials with abnormally high residues in control samples and for which no representative chromatograms were supplied. In such cases it was not clear whether the control samples were contaminated or the analysis was at fault.

Trials which were unsuitable for evaluation are shown shaded in the Tables.

Residues in crops

Citrus fruits. Thirteen supervised trials were carried out in Argentina (lemons), Brazil (sweet oranges), Greece (sweet oranges), Japan (Satsuma mandarins, sour oranges, Yuzu (i.e. lemons and limes), and Natsudaidai) and New Zealand (clementines). The results are shown in Table 3.

Table 3. Residues of acephate in citrus fruits.

Crop, country, year	Application				PHI, days	Residues[1]		Reference and Remarks
	Form.	No.	kg ai/ha	kg ai/hl		Acephate	Methamidophos	
Lemon Argentina 1995	SP	1	0.88	0.13	10 31	<0.1 <0.1		66[2]
Orange Brazil 1994	SP	2	1.23	0.056	14 21 28	0.2 0.2 0.1		70
		2	2.46	0.11	14	0.5		

Crop, country, year	Application				PHI, days	Residues[1]		Reference and Remarks
	Form.	No.	kg ai/ha	kg ai/hl		Acephate	Methamidophos	
					21 28	0.3 0.1		
Orange Greece 1995	SP	2	1.2	0.031	20	0.23	0.05	67

Satsuma mandarin Japan 1992	WP	3	2.5	0.05	30 45 60 	0.628(F) 0.50(P) <u>0.60(W)</u> 0.584(F) 0.41(P) 0.55(W) 0.564(F) 0.22(P) 0.50(W)	0.068(F) 0.09(P) 0.07(W) 0.052(F) 0.05(P) 0.05(W) 0.037(F) 0.02(P) 0.03(W)	49[3]
Satsuma mandarin Japan 1992	WP	3	2.5	0.05	30 45 60 	1.22(F) 0.68(P) <u>1.12(W)</u> 0.992(F) 0.44(P) 0.88(W) 0.623(F) 0.17(P) 0.52(W)	0.102(F) 0.14(P) 0.11(W) 0.062(F) 0.06(P) 0.06(W) 0.034(F) 0.02(P) 0.03(W)	48[3]
Sour oranges Japan 1993	WP	3	2.5	0.05	30 45 60	<u>0.134</u> 0.016 0.012	0.031 <0.005 <0.005	45
Yuzu Japan 1993	WP	3	2.5	0.05	30 45 60	<u>0.546</u> 0.261 0.104	0.044 0.019 0.010	55
Natsudaidai Japan 1992	WP	3	1.65	0.033	30 45 60 	0.121, 0.140(F) 0.166, 0.35(P) <u>0.132, 0.20(W)</u> 0.108, 0.114(F) 0.082, 0.15(P) 0.101, 0.12(W) 0.044, 0.048(F) 0.036, 0.04(P) 0.042, 0.05(W)	0.010, 0.013(F) 0.024, 0.06(P) 0.013, 0.03(W) 0.007, 0.009(F) 0.01, 0.02(P) 0.008, 0.01(W) <0.005,<0.005(F) <0.005, <0.01(P) <0.005, <0.01(W)	50[3,4]
		3	2.5	0.05	30 45 60 	0.222, 0.403(F) 0.59, 0.59(P) <u>0.301, 0.45(W)</u> 0.144, 0.217(F) 0.142, 0.22(P) 0.144, 0.22(W) 0.119, 0.170(F) 0.16, 0.19(P) 0.175, 0.13(W)	0.017, 0.021(F) 0.09, 0.15(P) 0.033, 0.06(W) 0.010, 0.015(F) 0.026, 0.04(P) 0.013, 0.02(W) 0.010, 0.011(F) 0.03, 0.03(P) 0.015, 0.02(W)	
Natsudaidai Japan 1993	WP	3	1.7	0.033	30 45 60 	0.295, 0.475(F) 6.00, 8.42(P) <u>2.02, 2.95(W)</u> 0.173, 0.274(F) 2.53, 5.22(P) 0.90, 1.83(W) 0.238, 0.342(F) 3.55, 4.51(P) 1.25, 1.62(W)	0.036, 0.047(F) 0.82, 0.972(P) 0.27, 0.334(W) 0.024, 0.027(F) 0.42, 0.532(P) 0.15, 0.186(W) 0.028, 0.030(F) 0.531, 0.56(P) 0.182, 0.19(W)	51[3,4]

		3	2.5	0.05	30	0.264, 0.270(F)	0.020, 0.028(F)	
						4.88, 5.36(P)	0.682, 0.81(P)	
						1.67, 1.86(W)	0.230, 0.26(W)	
					45	0.433, 0.610(F)	0.054, 0.055(F)	
						5.88, 6.47(P)	0.78, 0.86(P)	
						2.06, 2.31(W)	0.265, 0.29(W)	
					60	0.334, 0.552(F)	0.042, 0.050(F)	
						3.55, 7.22(P)	0.56, 0.915(P)	
						1.35, 2.60(W)	0.21, 0.316(W)	
Mandarin New Zealand 1996	SP	7	2.7	0.075	14	2.59, 3.34	0.23, 0.29	68

[1] (F) Flesh (P) Peel (W) Whole
[2] No data on analytical recoveries
[3] The data were also submitted to the 1994 JMPR
[4] The 2 results were from duplicate analyses carried out in different laboratories. The higher values of each pair were used to estimate maximum residues and the means to estimate STMRs

Broccoli. Fourteen supervised trials data were carried out in Australia, Brazil, France, Italy, Japan and Spain. The results are shown in Table 4.

Table 4. Residues of acephate in broccoli.

Country, year	Application				PHI, days	Residues		Reference and Remarks
	Form.	No.	kg ai/ha	kg ai/hl		Acephate	Methamidophos	
	SP	6	0.98	0.21	7	<0.02, <0.02	<0.02, <0.02	62[1]
Australia					14	<0.02, 0.02	<0.02, 0.02	
1995					21	0.12, 0.32	0.04, 0.08	
					28	<0.02, <0.02	<0.02, <0.02	
		6	2	0.41	7	3.0, 3.1	0.41, 0.52	
					14	1.6, 3.4	0.34, 0.52	
					21	0.29, 0.58	0.06, 0.17	
					28	0.02, 0.02	<0.02, <0.02	
	SP	1	0.75	0.075	0	7.3		72
Brazil 1995		3	0.75	0.075	7	2.3		
					14	0.2		
					21	<0.1		
		1	1.5	0.15	0	11.8		
		3	1.5	0.15	7	6.5		
					14	0.3		
					21	<0.1		
France 1992	SP	3	0.75	0.25	7	0.32		46[2]
	SP	3	0.75	0.25	7	0.03		47[2]
	SP	3	0.73	0.22	0	2.2	0.13	78
France 1995					3	0.25	0.08	

Country, year	Application				PHI, days	Residues		Reference and Remarks
	Form.	No.	kg ai/ha	kg ai/hl		Acephate	Methamidophos	
					7	0.15	0.05	
					14	0.03	0.02	
France 1995	SP	3	0.75	0.25	0	0.37	<0.01	77
					3	0.06	0.01	
					7	<0.01	<0.01	
					14	<0.01	<0.01	
Italy 1991	WP	1	0.47	0.047	28	0.45	0.2	45[3]
		1	0.94	0.094	28	1.21	0.46	
Japan 1993	WP	3	1.25	0.05	7	0.488, 0.742	0.138, 0.166	53[3,4]
					14	0.070, 0.158	0.017, 0.040	
					21	0.016, 0.028	0.008, 0.008	
Japan 1993	WP	3	1.25	0.05	7	4.22, 6.29	0.962, 1.49	52[3,4]
					14	1.28, 1.66	0.415, 0.566	
					21	1.15, 1.24	0.470, 0.529	
Spain 1995	SP	3	1.1	0.11	14	0.05	0.03	63
Spain 1996	WP	3	1	0.091	0	5.6	0.2	79
					3	4.8	0.28	
					7	2.5	0.31	
					14	1.2	0.32	
					21	0.33	0.15	

[1] The 2 results were from duplicate plots. The higher values of each pair were used to estimate both maximum residues and STMRs
[2] Analytical recovery was too low
[3] The data were also submitted to the 1994 JMPR
[4] The 2 results were from duplicate analyses carried out in different laboratories. The higher values of each pair were used to estimate maximum residues and the means to estimate STMRs

Brussels sprouts. The results of ten supervised trials in Australia, Belgium, South Africa and the USA are shown in Table 5.

Table 5. Residues of acephate in Brussels sprouts.

Country, year	Application				PHI, days	Residues		Reference and Remarks
	Form.	No.	kg ai/ha	kg ai/hl		Acephate	Methamidophos	
Australia 1995	SP	6	0.98	0.21	1	2.8, 3.9	0.3, 0.3	58[1]
					3	2.6, 7.1	0.2, 0.4	
					5	1.1, 2.2	0.1, 0.2	
					7	4.8, 11.5	0.8, 1.0	
		6	2	0.41	1	20.3, 20.5	1.0, 1.3	
					3	13.0, 15.1	0.9, 0.9	
					5	9.5, 18.5	0.6, 1.4	
					7	10.1, 15.8	0.9, 1.2	
	SP	6	0.98	0.24	1	0.58, 0.78	0.05, 0.09	64[1,2]

Country, year	Application				PHI, days	Residues		Reference and Remarks
	Form.	No.	kg ai/ha	kg ai/hl		Acephate	Methamidophos	
Australia 1995					3	0.43, 1.33	0.05, 0.12	
					5	0.82, 1.04	0.07, 0.09	
					7	1.32, 1.51	0.10, 0.11	
		6	2	0.49	1	1.39, 1.58	0.13, 0.13	
					3	2.1, 2.6	0.15, 0.17	
					5	1.74, 3.54	0.13, 0.22	
					7	1.67, 2.34	0.12, 0.15	
Belgium 1972	SP	1		0.025	3	0.06, 0.09	0.83, 0.95	40[1,3]
South Africa 1972	SP	3	0.56	0.056	1	2.59, 3.05	0.07, 0.10	4[3]
					4	2.58, 2.92	0.12, 0.14	
					8	0.42, 1.43	0.03, 0.09	
					14	1.15, 1.28	0.08, 0.10	
					21	0.37, 0.74	0.04, 0.06	
USA, Trimmed heads	SP	5	1.1	0.24	0	4.7, 5.5	0.22, 0.23	1[1]
					3	0.62, 0.73	0.05, 0.06	
					7	0.16, 0.43	0.04, 0.04	
					14	0.10, 0.23	0.01, 0.0	
		5	2.2	0.48	0	2.9, 4.4	0.09, 0.11	
					3	0.33, 0.40	0.03, 0.03	
					7	0.45, 0.54	0.05, 0.02	
					14	0.03, 0.10	<0.01, <0.01	
Trimmings	SP	5	1.1	0.24	0	5.6, 7.0	0.26, 0.17	
					3	0.20, 0.28	0.04, 0.03	
					7	0.21, 0.34	0.04, 0.03	
					14	0.10, 0.15	0.01, 0.02	
		5	2.2	0.48	0	1.9, 4.6	0.08, 0.11	
					3	0.35, 0.55	0.04, 0.04	
					7	0.29, 0.43	0.03, 0.02	
					14	0.04, 0.05	<0.01, 0.01	
Trash	SP	5	1.1	0.24	0	22.2, 22.6	0.60, 0.59	
					3	1.98, 3.03	0.24, 0.32	
					7	0.50, 0.52	0.07, 0.07	
					14	0.20, 0.70	0.04, 0.10	
		5	2.2	0.48	0	6.28, 7.52	0.18, 0.24	
					3	0.93, 1.55	0.15, 0.21	
					7	0.38, 0.55	0.09, 0.14	
					14	0.21, 0.46	0.10, 0.05	
USA 1971	SP	8	1.1	0.27	0	2.28, 2.41	0.06, 0.07	3[1]
					3	2.19, 2.71	0.08, 0.10	
					7	0.58, 1.15	0.05, 0.08	
USA 1971	SP	9	1.1	0.4	0	1.41, 2.75	0.09, 0.20	4[1,4]
					3	1.03, 1.31	0.05, 0.07	
					7	0.16, 0.21	0.02, 0.03	

[1] Duplicate results were from duplicate plots. Only summary data were submitted

[2] No information on control samples and no sample chromatograms

[3] Abnormally high control values and no sample chromatograms

Head cabbages. The results of 11 supervised trials in Brazil, France, Germany, Japan and The Netherlands are shown in Table 6.

Table 6. Residues of acephate in head cabbages.

Country, year	Application				PHI, days	Residues		Reference and Remarks
	Form.	No.	kg ai/ha	kg ai/hl		Acephate	Methamidophos	
Brazil 1994	SP	3	0.26	0.075	7 14 21	<0.05 <0.05 <0.05		71
		3	0.52	0.15	7 14 21	<0.05 <0.05 <0.05		
France 1976	SP	3	0.75	0.075	0 7 10 14	0.83, 1.13 0.63, 1.08 0.36, 1.25 0.80, 0.93	0.03, 0.05 0.04, 0.07 0.04, 0.09 0.08, 0.09	24[1]
France 1973	SP	1	0.53	0.05	0 7 14 21	1.26 0.16 0.54 0.04	0.1 0.03 0.09 0.02	12
France 1974	SP	3	0.45	0.075	7	0.02, 0.03 0.05	0.01, 0.01 0.01	20[1]
France 1974	SP	1	0.45	0.075	7	0.02, 0.03 0.08	0.01, 0.01 0.01	19[1]
Germany 1976	SP	3	0.25 + 0.5 + 0.25	0.025 + 0.05 + 0.025	0 7 10 14 21	0.04 0.03 0.06 0.03 0.03	<0.01 <0.01 0.01 <0.01 <0.01	13
Germany 1976	SP	3	0.25 + 0.5 + 0.25	0.025 + 0.05 + 0.025	0 7 10 14 21	0.13 0.02 0.03 0.03 <0.02	<0.01 <0.01 <0.01 <0.01 <0.01	25
Japan 1988	WP	3	0.9	0.05	6 13 19	0.057, 0.083 0.028, 0.032 0.022, 0.101	0.010, 0.010 0.006, 0.008 <0.005, 0.016	44[2,3]
Japan 1987	WP	3	0.75	0.05	7 14 21	0.492, 0.664 0.276, 0.460 0.131, 0.139	0.096, 0.138 0.069, 0.140 0.044, 0.057	43[2,3]
Netherlands 1972	SP	1	0.75	0.075	14	0.313, 0.331	0.038, 0.050	5[1,3]

[1] The 2 results were from duplicate plots. The higher values of each pair were used to estimate both maximum residues and STMRs

[2] The 2 results were from duplicate analyses carried out in different laboratories. The higher values of each pair were used to estimate maximum residues and the means to estimate STMRs
[3] The data were also submitted to the 1994 JMPR

Cauliflower. The results of seventeen supervised trials carried out in Australia, Brazil, France, Germany, Japan and The Netherlands are shown in Table 7.

Table 7. Residues of acephate in cauliflower.

Country, year	Application				PHI, days	Residues		Reference and Remarks
	Form.	No.	kg ai/ha	kg ai/hl		Acephate	Methamidophos	
Australia 1995	SP	6	0.98	0.24	1	1.15, 1.50	0.11, 0.11	65[1]
					3	0.47, 0.81	0.05, 0.09	
					5	0.64, 0.80	0.09, 0.10	
					7	0.72, 1.37	0.12, 0.20	
		6	2	0.49	1	3.19, 4.93	0.31, 0.39	
					3	2.82, 3.37	0.26, 0.32	
					5	1.14, 3.33	0.17, 0.36	
					7	2.34, 2.39	0.27, 0.28	
Brazil 1995	SP	1	0.75	0.075	0	5.3		73
		3	0.75	0.075	7	1.2		
					14	0.1		
					21	<0.1		
		1	1.5	0.15	0	7.1		
		3	1.5	0.15	7	2.3		
					14	0.3		
					21	<0.1		
France 1975	SP	1	0.5	0.03	14	0.03	<0.01	22[2]
France 1988	SP	1	0.94	0.075	14	1.33, 1.64	0.19, 0.22	32[1,3,4]
					21	1.04, 1.06	0.20, 0.17	
		2	0.94	0.075	21	0.35, 0.41	0.14, 0.20	
France 1995	SP	3	0.73	0.23	7	0.15	0.03	81
France 1995	WP	3	0.75	0.42	0	0.2	0.01	80
					2	0.21	0.02	
					4	0.14	0.02	
					7	0.1	0.01	
France 1995	WP	3	0.75	0.32	0	0.42	0.04	86
					2	0.09	0.02	
					4	0.07	0.02	
					7	0.03	0.01	
Germany 1976	SP	3	0.25 + 0.5 + 0.25	0.025 + 0.05 + 0.025	0	0.45	0.03	26[2]
					7	0.12	0.02	
					10	0.31	0.06	
					14	0.39	0.06	
					21	0.04	0.01	
Japan 1995	WP	3	1	0.05	14	0.006, 0.008	<0.005, 0.006	60[5]
					21	<0.005,< 0.005	<0.005, <0.005	
					28	<0.005, <0.005	<0.005, <0.005	

Country, year	Application				PHI, days	Residues		Reference and Remarks
	Form.	No.	kg ai/ha	kg ai/hl		Acephate	Methamidophos	
Japan 1995	WP	3	1	0.05	14 21 28	0.586, 0.724 0.240, 0.290 0.162, 0.206	0.228, 0.214 0.088, 0.082 0.059, 0.071	61[5]
Netherlands 1972	SP	1	0.75	0.075	14	0.041, 0.117	0.008, 0.018	6[1,2]

Netherlands	SP	4	0.76	0.094	13	0.03	<0.01	82
1995		3	0.77	0.094	28	<0.01	<0.01	
Netherlands	SP	4	0.76	0.094	13	0.02	<0.01	83
1995		3	0.76	0.094	28	<0.01	<0.01	
Netherlands	SP	4	0.76	0.094	14	≤0.01	<0.01	84
1995		3	0.75	0.094	28	<0.01	<0.01	
Netherlands	SP	4	0.76	0.094	11	0.11	0.03	85
1995		3	0.76	0.094	19	0.03	<0.01, 0.01	

[1] The 2 results were from duplicate plots. The higher values of each pair were used to estimate both maximum residue levels and STMRs
[2] The data were also submitted to the 1994 JMPR
[3] Abnormally high control values and no sample chromatograms
[4] The data were also submitted to the 1990 JMPR
[5] The 2 results were from duplicate analyses carried out in different laboratories. The higher values of each pair were used to estimate maximum residues and the means to estimate STMRs

Tomatoes. Forty supervised trials were carried out in Australia, Brazil, Canada, France, Japan, South Africa, Spain and the USA. The results are given in Table 8.

Table 8. Residues of acephate in tomatoes.

Country, year	Application				PHI, days	Residues		Reference and Remarks
	Form.	No.	kg ai/ha	kg ai/hl		Acephate	Methamidophos	
Australia 1995	SP	6	0.98	0.35	1	1.1, 1.6	0.23, 0.30	69[1]
					3	1.5, 1.8	0.40, 0.50	
					5	0.85, 0.88	0.23, 0.26	
					7	1.2, 1.6	0.33, 0.43	
		6	2	0.7	1	1.9, 2.8	0.37, 0.58	
					3	0.76, 0.77	0.22, 0.23	
					5	1.6, 2.6	0.47, 0.55	
					7	2.1, 2.2	0.53, 0.54	
Brazil 1994	SP	3	0.3	0.075	3	<0.05		74
					7	≤0.05		
					14	<0.05		
		3	0.6	0.15	3	<0.05		
					7	<0.05		
					14	<0.05		
Canada 1980	WP	3	0.55	0.069	3	0.79	0.06	56[2,3]
					7	0.75	0.02	
					14	0.22	0.04	
		3	1.1	0.14	3	0.94	0.05	
					7	1.1	0.03	
					14	0.68	0.02	

Canada 1980	WP	3	0.55	0.069	3 7 14	0.47 0.46 0.21	0.11 0.14 0.01	57[2,3]
		3	1.1	0.14	3	0.71	0.15	
					7 14	0.72 0.06	0.21 0.03	
France 1973	SP	1	0.3	0.03	0 7	2.08 0.13	<0.02 <0.02	7
					13 20	0.07 0.06	<0.02 <0.02	
France 1973	SP	1	0.5	0.05	0 7 13 20	0.93 0.06 0.05 0.07	0.03 0.02 0.02 0.03	8
France 1973	SP	1	0.53	0.053	0 7 13 20	0.49 0.15 0.1 <0.05	0.02 0.02 0.04 0.03	9
France 1974	SP	2	0.83	0.05	1 3 7 10 15 21	0.38, 0.63, 0.72 0.19, 0.41, 0.43 0.14, 0.29, 0.36 0.12, 0.18, 0.36 0.11, 0.21 0.29 0.10, 0.14 0.19	0.02, 0.02 0.02 0.02, 0.02 0.02 0.02, 0.03 0.03 0.03, 0.03 0.04 0.03, 0.05 0.06 0.03, 0.04 0.06	14[1]
France 1974	SP	2	0.83	0.05	1 7	0.42, 0.56, 0.65, 0.53 0.08, 0.17 0.17, 0.17	0.01, 0.01 0.03, 0.04 0.01, 0.02 0.03, 0.03	10[1]
France 1974	SP	1	0.9	0.075	7	0.46, 0.61	0.04, 0.05	15[1,4]
France 1974	SP	3	0.9	0.075	7	1.10, 1.48	0.22, 0.25	16[1,4]
France 1974	SP	1	0.45	0.075	7	0.34, 0.38	0.06, 0.08	17
France 1974	SP	3	0.45	0.075	7	0.24, 0.32	0.08, 0.10	18
France 1974	SP	1	0.5	0.05	14	0.09	0.02	21
France 1974	SP	3	0.76	0.075	0 7 10 14	0.68, 0.77 0.35, 0.45 0.26, 0.48 0.27, 0.38	0.09, 0.11 0.11, 0.14 0.12, 0.17 0.13, 0.16	24[1]
France 1986	SP	1	0.75	0.075	14 21	0.39, 0.45 0.19	0.12, 0.16 0.04	27[1]
France 1986	SP	1	0.62	0.075	15 21	0.09, 0.26 0.06, 0.08	0.02, 0.04 0.03, 0.03	28[1]

France 1988	SP	1	0.55	0.075	13 20	<0.05, <0.05 <0.05, 0.12	<0.02, <0.02 <0.02, <0.02	30[1]
		2	0.55	0.075	20	0.09, 0.22	<0.02, 0.05	
France 1988	SP	1	1.64	0.075	14 21	0.40, 0.44 0.05, 0.08	0.06, 0.07 0.06, 0.07	31[1]
		2	1.64	0.075	21	0.54, 0.95	0.32, 0.44	
France 1992	SP	3	0.75	0.075	2	0.4		75
France 1992	SP	3	0.75	0.075	0 1 2 5 7	0.8 0.91 0.94 0.59 0.74		76
Japan 1985	WP	3	0.9	0.05	1 3 7	0.597, 1.03 0.703, 0.878 0.720, 0.893	0.063, 0.082 0.060, 0.072 0.072, 0.106	42[3,5]
Japan 1984	WP	3	0.75	0.05	1 3 7	0.225, 0.687 0.566, 0.867 0.352, 0.648	0.027, 0.059 0.058, 0.084 0.085, 0.123	42[3,5]
South Africa 1973	SP	5	0.38	0.013	0 1 2 3 7	0.16 0.14 0.16 0.12 0.14	0.04 0.03 0.03 0.03 0.03	11[3]
		5	0.75	0.026	3	0.23	0.07	
Spain 1995	SP	3	1.1	0.11	14	0.05	0.03	59
USA 1987	SP	8	1.12	0.4	3	1.4	0.08	29
USA 1990	SP	6	1.12	0.21-0.27	3 5 	0.50, 0.63 0.65, 0.69 0.36, 0.40 0.70, 0.73	0.12, 0.17 0.17, 0.18 0.12, 0.12 0.19, 0.19	90[1]
USA 1990	SP	6	1.12	0.21-0.27	3 5 	0.60, 0.64 0.75, 0.98 0.36, 0.49 0.63, 0.64	0.17, 0.18 0.23, 0.27 0.12, 0.16 0.19, 0.22	87[1]
USA 1989	SP	6	1.12	0.14-0.22	3 5 	0.69, 1.0 1.1, 1.3 0.76, 0.87 0.89, 1.00	0.12, 0.17 0.19, 0.25 0.16, 0.19 0.18, 0.20	88[1]
USA 1989	SP	6	1.12	0.096-0.19	3 5 	0.33, 0.43 0.47, 0.53 0.25, 0.28 0.29, 0.32	0.14, 0.18 0.23, 0.26 0.13, 0.15 0.18, 0.21	89[1]

USA 1990	SP	6	1.12	0.19-0.2	3 5	0.46, 0.66 0.57, 0.62	0.09, 0.14 0.16, 0.19	91[1]
USA 1990	SP	6	1.1	0.1-0.25	3	0.19, 0.20 0.24, 0.27	0.06, 0.06 0.08, 0.09	92[1]
USA 1993	SP	6	1.12	0.38-0.41	3	0.22, 0.28	0.06, 0.09	93[1]
USA 1993	SP	6	1.12	0.39-0.42	3	0.45, 0.47	0.09, 0.09	94[1]
USA 1993	SP	6	1.12	0.4	3	0.33, 0.51	0.07, 0.13	95[1]

[1] Multiple results were from replicate plots. The highest values of each set were used to estimate both maximum residue levels and STMRs
[2] Only summary data were submitted
[3] The data were also submitted to the 1994 JMPR
[4] Abnormally high control values and no sample chromatograms
[5] The 2 results were from duplicate analyses carried out in different laboratories. The higher values of each pair were used to estimate maximum residues and the means to estimate STMRs

FATE OF RESIDUES IN STORAGE AND PROCESSING

In processing

Tomatoes. In three studies in the USA harvested tomatoes were processed in the laboratory by typical commercial practices, but details were not provided. The results are given in Table 9.

Table 9. Residues of acephate and methamidophos in processed fractions of tomatoes, USA.

Application, year	Sample	Residues, mg/kg		Reference
		Acephate	Methamidophos	
1.12 kg ai/ha 0.4 kg ai/hl 8 applications PHI: 3 days 1987	Whole fruit	1.4	0.08	29
	Washed fruit	1.8	0.9	
	Canned whole fruit	0.54	0.04	
	Canned juice	1.3	0.08	
	Bulk paste	5.6	0.43	
	Canned purée	2.5	0.17	
	Wet pomace	0.84	0.04	
	Dry pomace	1.4	0.09	
1.12 kg ai/ha 6 applications PHI: 3 days	Whole fruit	0.36, 0.36	0.15, 0.17	91
	Washed fruit	0.34, 0.37	0.15, 0.18	
	Peeled fruit	0.25, 0.48	0.12, 0.24	
	Canned whole fruit	0.18, 0.27	0.11, 0.17	
	Canning waste	0.70, 0.75	0.06, 0.06	
1.12 kg ai/ha 0.096-0.19 kg ai/hl 6 applications PHI: 3 days	Whole fruit	0.49, 0.56	0.23, 0.28	89
	Washed fruit	0.54, 0.65	0.26, 0.33	
	Peeled fruit	0.24, 0.48	0.13, 0.25	
	Canned whole fruit	0.28, 0.25	0.18, 0.19	
	Canning waste	0.81, 1.1	0.26, 0.43	

Cooking studies were carried out on three vegetables containing acephate and methamidophos (Crossley, 1971). Field-treated tomatoes, cabbage and broccoli were analyzed for acephate and methamidophos before and after boiling for 30 minutes. The results are given in Table 10.

Table 10. Residues of acephate and methamidophos in crops before and after 30 minutes boiling.

Crop	Acephate, mg/kg		Methamidophos, mg/kg	
	Before cooking	After cooking	Before cooking	After cooking
Tomatoes	0.93, 1.13	0.93, 1.09	0.12, 0.14	0.13, 0.15
Cabbage	2.08, 2.20	2.06, 2.08	0.22, 0.22	0.24, 0.25
Broccoli	8.38, 9.92	8.02, 7.12	0.98, 1.17	1.00, 1.10

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

National monitoring data

The government of The Netherlands reported monitoring data on acephate in several crops (Table 11).

Table 11. Monitoring data on acephate in several crops in The Netherlands, 1991-1994.

Commodity	Samples analyzed	Number in which residues found[1]	Detection frequency, %	Mean residues[2], mg/kg
Peaches	379	16	4.2	0.04, <0.02[3]
Nectarines	401	19	4.7	<0.02, 0.02[3]
Plums	536	2	0.4	<0.02
Grapes	1335	18	1.3	<0.02
Tomatoes	330	4	1.2	<0.02
Lettuce	865	10	1.2	0.03, 0.05[3]
Endive	511	2	0.4	<0.02

[1] LOD = 0.02 mg/kg.
[2] For samples with residues below the LOD, half the LOD is taken for the calculation of the mean residues
[3] Means for 1991-1993 and 1994 respectively

Market basket surveys

Market basket surveys for acephate and methamidophos were carried out in the USA in 1984 and 1985. From 26 to 62 commodities including fresh vegetables, fresh fruit, canned food, meat and dairy products were collected from 24 locations. Acephate and methamidophos were found at or above the limit of determination (0.01 mg/kg) in 6 and 7 commodities respectively (Table 12).

Table 12. Residues of acephate and methamidophos found at or above the limit of determination in market basket surveys in the USA, 1984 and 1985.

Commodity	Residues, mg/kg		Reference
	Acephate	Methamidophos	
Cantaloupe	0.03	0.02, 0.02, 0.03, 0.10	38
Celery	0.01, 0.03, 0.04	0.04	
Cucumbers	-	0.04, 0.06	
Crisphead lettuce	0.01, 0.09	0.02	
Tomatoes	0.01, 0.02	0.02, 0.02, 0.03, 0.04, 0.10, 0.17	
Green sweet pepper	0.06, 0.72	0.02, 0.03, 0.26	
Canned snap beans	0.01, 0.02	0.01	

Farm gate to consumer studies

Farm gate to consumer studies were carried out on five crops in the USA in 1985 and 1986 (Lai, 1989a). Lettuce, snap beans, cauliflowers, Brussels sprouts and bell peppers were treated with acephate at the highest label use rate and monitored for residues from the time of harvest through typical commercial processes to the consumer. The results are given in Table 13.

Table 13. Residues of acephate and methamidophos in crisphead lettuce, snap beans, bell peppers cauliflowers and Brussels sprouts from farm gate to consumer, USA.

Application Year	Description (Location)	Average residues, mg/kg			
		Acephate	% of field	Methamidophos	% of field
0.63 kg ai/ha	Whole head lettuce (field)	0.30	100	0.02	100
+ 1.12 kg ai/ha	Head + cap leaf (cooler)	0.05	17	0.00	0
2 applications	Head + cap leaf (distributor)	0.06	20	0.00	0
PHI 21 days	Head + cap (market)	0.04	13	0.00	0
1985	Head + cap (supermarket shelf)	0.03	10	0.00	0
0.84 kg ai/ha	Fresh snap beans (field)	0.29	100	0.06	100
2 applications	Fresh snap beans (market)	0.10	35	0.02	36
PHI 24 days	Fresh snap beans (processing plant)	0.13	46	0.03	55
1985	Canned snap beans	0.05	18	0.02	36
	Frozen snap beans in butter sauce	0.03	11	0.00	0
1.5 kg ai/ha	Bell peppers (field)	3.8	100	0.52	100
7 applications	Bell peppers (packing shed)	2.8	74	0.43	83
PHI 9 days	Bell peppers (distributor)	2.7	71	0.45	87
1986	Bell peppers (supermarket)	3.1	82	0.51	97
1.12 kg ai/ha	Cauliflower head (field)	0.80	100	0.10	100
6 applications	Trimmed head (cooler)	0.34	43	0.04	40
PHI 14 days	Curd after coring (processor)	0.33	41	0.04	40
1986	Curd after processing and freezing	0.25	31	0.04	40
	Processing waste	0.73	91	0.10	95

1.12 kg ai/ha	Fresh Brussels sprouts (field)	1.85	100	0.03	100
6 applications	Fresh sprouts after sorting	0.79	43	0.02	67
PHI 14 days	Sorting waste	1.6	86	0.02	67
1986	Sprouts after blanching & freezing	0.13	7	0.01	33
	Processing waste	9.4	508	0.15	500

NATIONAL MAXIMUM RESIDUE LIMITS

The following national MRLs were reported to the Meeting.

Country	Commodity	MRL, mg/kg	Commodity	MRL, mg/kg
Australia	Apples/Pears	0.02	Pepper	5
	Broccoli	5	Potato	0.5
	Cabbage	5	Soya beans	1
	Citrus	5	Sugar beet	0.1
	Cotton seed	2	Tomatoes	5
	Lettuce	10		
Brazil	Beans	0.5	Peanut	0.2
	Broccoli	0.5	Pepper	1
	Cabbage	0.5	Potato	0.2
	Cauliflower	0.5	Soya beans	1
	Cotton seed	0.2	Tomatoes	0.5
Canada	Beans	1	Cranberries	0.5
	Brussels sprouts	1.5	Lettuce	1
	Cabbage	0.3	Pepper	2
	Cauliflower	2	Potato	0.5
Chile	Beans	3	Cauliflower	2
	Brussels sprouts	3	Lettuce	10
	Cabbage	3	Pepper	4
EU[1]	Apples/Pears	0.02	Lettuce	1
	Beans	0.02	Peanut	0.02
	Broccoli	0.02	Peas	0.1
	Brussels sprouts	2	Pepper	0.02
	Cabbage	2	Potato	0.02
	Cauliflower	0.02	Soya beans	0.02
	Celery	0.02	Spinach	0.02
	Citrus	1	Stone fruit	0.02
	Cotton seed	0.02	Sugar beet	0.02
	Grapes	0.02	Tomatoes	0.5
	Hops	0.1		
France	Apples/Pears	1	Lettuce	1
	Artichoke	1	Potato	0.02
	Cabbage	2	Tea	0.1
	Citrus	1	Tomatoes	0.5
	Cotton seed	0.02		
Germany	Apples/Pears	1	Hops	15
	Grapes	1.5	Stone fruit	1
Hungary	Cabbage	0.5	Sugar beet	0.1
	Peas	0.1		
Israel	Cabbage	5	Onion	0.5

Country	Commodity	MRL, mg/kg	Commodity	MRL, mg/kg
	Corn	2	Pepper	5
	Garlic	0.5	Tomatoes	5
	Mango	2	Watermelon	5
Italy	Apples/Pears	1.5	Lettuce	1.5
	Broccoli	1.5	Potato	1.5
	Cabbage	1.5	Stone fruit	1.5
	Cauliflower	1.5	Sugar beet	1.5
	Citrus	1.5	Tobacco	1.5
	Grapes	1.5	Tomatoes	1.5
Japan	Beans	3	Onion	0.5
	Broccoli	5	Parsley	0.5
	Brussels sprouts	5	Peanut	0.2
	Cabbage	5	Peas	0.1
	Cauliflower	5	Pepper	5
	Celery	10	Persimmon	2
	Chinese cabbage	5	Potato	1
	Citrus	10	Radish (leaf)	10
	Corn	0.1	Radish (root)	1
	Cotton seed	2	Soya beans	0.5
	Cranberries	0.5	Spinach	5
	Cucumber	5	Sugar beet	0.1
	Egg plant	5	Tea	10
	Garlic	2	Tomatoes	5
	Grapes	5	Turnip (leaf)	10
	Horseradish	5	Turnip (root)	1
	Kale	5	Watermelon	0.5
	Kidney bean	3	Welsh onion	0.1
	Lettuce	5	Yam	0.5
	Mustard	5		
New Zealand	Avocado	1	Lettuce	6
	Cabbage	2	Potato	1
	Cauliflower	2	Tamarillo	0.5
	Citrus	5	Tomatoes	1
USA	Beans	3	Lettuce	10
	Brussels sprouts	3	Mint hay	15
	Cauliflower	2	Peanut	0.2
	Celery	10	Pepper	4
	Cotton seed	2	Soya beans	1
	Cranberries	0.5		
Venezuela	Broccoli	5	Lettuce	10
	Cabbage	10	Potato	5
	Cauliflower	5	Rice	5
	Citrus	5	Sesame	2
	Corn	5	Tobacco	1
	Cotton seed	2	Tomatoes	5

[1] Proposed MRLs

APPRAISAL

Acephate was first evaluated in 1976, with further reviews of residue aspects in 1979, 1981, 1984, 1990 and 1994. The 1994 JMPR withdrew the previous recommendations for the MRLs for broccoli, Brussels sprouts, head cabbages, cauliflowers, citrus fruits and tomatoes which had been held at Step 7B by the 1989 CCPR (ALINORM 89/24A, para 126). The manufacturer has indicated that information on GAP and residue data would be available to support new MRLs for these commodities. This information was provided to the Meeting, together with information on analytical methods and residues in food in commerce or at consumption.

Analytical methods

Samples from the supervised trials were analysed by GLC and acephate and its metabolite methamidophos were determined individually. Recoveries of both acephate and methamidophos were generally >70%, with limits of determination of 0.01-0.02 mg/kg.

These methods were considered suitable for use in supervised trials and for enforcement.

Storage stability of analytical samples

Extensive storage stability studies were carried out with vegetables, pulses, oilseed, animal products, cereals and grasses. Acephate was shown to be stable in a wide range of macerated or ground commodities at -20°C.

Data validity

In view of the difficulty of determining methamidophos caused by its high polarity, the Meeting did not evaluate trials which lacked data on analytical recoveries or in which recoveries were below 70%, trials without analyses of control samples, or trials with abnormally high control values and for which sample chromatograms were not supplied.

Field trials data

Only residues of acephate are discussed. Residues of methamidophos are discussed in the monograph on that compound.

Citrus fruits. Eight supervised trials, two on Satsuma mandarins, four on Natsudaidai, one on Kabosu (sour orange) and one on Yuzu (lemons or limes) were carried out in Japan. The trials on mandarins and Natsudaidai had already been submitted to the 1994 JMPR. The residues from trials according to GAP (0.025-0.05 kg ai/hl, 30 days PHI) were 0.60 and 1.12 mg/kg in mandarins, 0.13-2.95 mg/kg in Natsudaidai, 0.13 mg/kg in Kabosu and 0.55 mg/kg in Yuzu. A single New Zealand trial on mandarins was according to GAP (0.075 kg ai/hl, 14 days PHI) with a residue of 2.59-3.34 mg/kg (duplicate determinations). In another trial in New Zealand reported in the 1984 JMPR monograph the conditions (0.075 kg ai/hl, 26 days PHI) complied with GAP. The residue was 1.6 mg/kg.

One trial in Greece was reported without information on GAP, but the trial conditions (0.031 kg ai/hl, 20 days PHI) were comparable to Italian GAP (0.024-0.036 kg ai/hl, 21 days PHI). The residue was 0.23 mg/kg. Two Brazilian trials were reported but there was no comparable GAP.

One supervised trial was carried out in Argentina, but there was no comparable GAP and

critical information on recoveries was lacking.

Since data on only four adequate additional trials (one Greek, two Japanese and one in New Zealand) were submitted, the Meeting could not recommend an MRL.

Broccoli and cauliflower. The Meeting agreed that the supervised trials on broccoli and cauliflower could be evaluated together because of the similarities in the use patterns and residue behaviour.

One Australian, one Brazilian and two Japanese trials on broccoli complied with national GAP (Australia 0.98 kg ai/ha, 0.098 kg ai/hl, 14 days PHI; Brazil 0.38-0.75 kg ai/ha, 0.075 kg ai/hl, 14 days PHI; Japan 0.05 kg ai/hl, 14 days PHI). In the Australian trial the spray concentration (0.21 kg ai/hl) was higher than the GAP concentration of 0.098 kg ai/hl but the rate in terms of kg ai/ha complied with GAP. Data on the Japanese trials had already been submitted to the 1994 JMPR. The residues were <0.02-0.32 mg/kg, 0.2 mg/kg and 0.07-1.66 mg/kg in the Australian, Brazilian and Japanese trials respectively.

Two French trials (0.73-0.75 kg ai/ha, 0.22-0.25 kg ai/hl, 0-14 days PHI) and two Spanish trials (1.0-1.1 kg ai/ha, 0.091-0.11 kg ai/hl, 0-21 days PHI) on broccoli were reported but information on GAP was not provided. Two Italian trials on broccoli which were submitted to the 1994 JMPR and resubmitted to the present Meeting did not comply with Italian GAP (0.034-0.064 kg ai/hl, 21 days PHI).

One Australian, one Brazilian and two Japanese trials on cauliflower were according to the relevant GAP (Australia 0.98 kg ai/ha, 0.098 kg ai/hl, 3 days PHI; Brazil 0.38-0.75 kg ai/ha, 0.075 kg ai/hl, 14 days PHI; Japan 0.05 kg ai/hl, 14 days PHI). In the Australian trial the spray concentration (0.24 kg ai/hl) again exceeded the GAP concentration but the kg ai/ha rate accorded with GAP. The residues were 0.47-1.37 mg/kg, 0.1 mg/kg and 0.006-0.72 mg/kg in Australia, Brazil and Japan respectively. Five trials in The Netherlands on cauliflower were submitted to the Meeting. One trial carried out in 1972 had been reported to the 1994 JMPR and was resubmitted this year. The trial conditions complied with GAP (0.75 kg ai/ha, 14 days PHI, 6 applications) except that there were only 1-4 applications. However the number of applications seems to have little influence on the residue of acephate. The residues were <0.01-0.12 mg/kg.

Four trials in France on cauliflower (0.50-0.75 kg ai/ha, 0.03-0.42 kg ai/hl and 0-14 days PHI) and one in Germany (0.25 kg ai/ha, 0.025 kg ai/hl and 0-21 days PHI) which had been submitted to the 1994 JMPR were resubmitted, but the trial conditions were not comparable with any relevant GAP.

The residues in one Australian, one Brazilian and 2 Japanese trials on broccoli at maximum GAP were 0.32, 0.2, 0.114 and 1.47 mg/kg respectively. Those in one Australian, one Brazilian, 2 Japanese and 5 Netherlands trials on cauliflower at maximum GAP were 1.37, 0.1, 0.007, 0.655, <0.01, 0.02, 0.03, 0.11 and 0.117 mg/kg respectively.

The residues in broccoli and cauliflower from the 13 trials in rank order were 0.007, <0.01, 0.02, 0.03, 0.1, 0.11, 0.114, 0.117, 0.2, 0.32, 0.655, 1.37 and 1.47 mg/kg. The Meeting estimated a maximum residue level of 2 mg/kg and an STMR level of 0.11 mg/kg for broccoli and cauliflower.

Brussels Sprouts. Two Australian trials complied with the Australian GAP application rate of 0.98 kg ai/ha although the spray concentration of 0.21 kg ai/hl was higher than the GAP concentration (0.098 kg ai/hl). The residues were 0.43-11.5 mg/kg at PHIs of 3 (GAP) to 7 days. One of two

American trials complied with American GAP (0.56-1.1 kg ai/ha, 14 days PHI) but the analyzed commodity (trimmed heads) was inappropriate for residue evaluation.

One trial in The Netherlands and three in South Africa according to GAP (0.75 kg ai/ha, 28 days PHI and 0.26-0.38 kg ai/ha, 3 days PHI respectively) were reported to the 1994 JMPR. The residues in The Netherlands were 0.29-0.94 mg/kg at 28 days and in South Africa 0.26-1.3 mg/kg at 7-28 days, and 0.95 and 1.4 mg/kg at 3 days.

The additional data were insufficient to estimate a maximum residue level.

Cabbages. One of two Brazilian trials, three of four French trials and two Japanese trials reflected appropriate GAP (Brazil 0.38-0.75 kg ai/ha, 0.075 kg ai/hl, 14 days PHI; France 0.075 kg ai/hl, 7 days PHI; Japan 0.025-0.05 kg ai/hl, 7 days PHI). In the Brazilian trial the dose rate of 0.26 kg ai/ha was lower than the GAP rate (0.75 kg ai/ha), but the spray concentration (0.075 kg ai/hl) complied with GAP. The data on the Japanese trials had already been submitted to the 1994 JMPR. The residues were <0.05 mg/kg, 0.02-1.25 mg/kg and 0.057-0.66 mg/kg in Brazil, France and Japan respectively.

Data from two German trials were submitted to the Meeting, but GAP was not available from Germany and the trial conditions were not comparable with other European GAP.

Data from one supervised trial in The Netherlands which had been submitted to the 1994 JMPR were resubmitted. The conditions (0.75 kg ai/ha, 14 days PHI, 1 application) accorded with GAP (0.75 ai/ha, 14 days PHI, 6 applications) except in the number of applications. The residues were 0.313-0.331 mg/kg.

Four supervised trials were carried out in New Zealand and the conditions in three of them (0.84 kg ai/ha, 0-7 days PHI; 1.1 kg ai/ha, 10 days PHI; 0.84 kg ai/ha, 16-23 days PHI) complied with GAP (0.75-1.1 kg ai/ha, 7 days PHI). The data were reported in the 1984 JMPR monograph. The residues were 0.8 mg/kg at 7 days, 1.2 mg/kg at 10 days and <0.4 mg/kg at 16 and 23 days respectively.

The residues found in one Brazilian, 3 French and 2 Japanese trials carried out at the maximum conditions complying with GAP were <0.05, 0.05, 0.08, 1.25, 0.07 and 0.578 respectively. Although the trial in The Netherlands was with a single application instead of the six allowed, the residue data could be used for the estimation of an STMR since the number of applications was not considered to be significant. The residue was 0.331 mg/kg. Two of the New Zealand trials also could be used for the estimation of an STMR. The residues were 0.8 and 1.2 mg/kg.

The residues from the 9 trials in rank order were <0.05, 0.05, 0.07, 0.08, 0.331, 0.578, 0.8, 1.2 and 1.25 mg/kg. The Meeting estimated a maximum residue level of 2 mg/kg and an STMR level of 0.33 mg/kg for acephate in cabbages

Tomatoes. GAP in several countries had changed since 1994. Current GAP and additional data on supervised trials were submitted to the Meeting. One Australian, one Brazilian, one Spanish and two Japanese trials reflected GAP (Australia 0.98 kg ai/ha, 0.098 kg ai/hl, 3 days PHI; Brazil 0.38-0.75 kg ai/ha, 0.075 kg ai/hl, 7 days PHI; Japan 0.025-0.05 kg ai/hl, 1 day PHI; Spain 0.038-0.11 kg ai/hl, 14 days PHI). The Australian spray concentration (0.35 kg ai/hl) was higher than the GAP concentration but the dose rate (kg ai/ha) complied with GAP. The data from the Japanese trials had been submitted to the 1994 JMPR and were resubmitted. The residues were 1.5-1.8 mg/kg, <0.05

mg/kg, 0.225-1.03 mg/kg and 0.05 mg/kg in Australia, Brazil, Japan and Spain respectively.

Seventeen trials in France were reported to the Meeting with no information on GAP, but four of them (0.62-0.83 kg ai/ha, 13-15 days PHI, 1-3 applications) complied with Polish GAP (0.75 kg ai/ha, 14 days PHI, 1 application) except in the number of applications which did not appear to influence the residues significantly. The residues were 0.09-0.45 mg/kg. A further ten of the French trials, at 0.03-0.075 kg ai/hl, 13-15 days PHI, were according to Spanish GAP (0.038-0.11 kg ai/hl, 14 days PHI) and showed residues of <0.05-0.95 mg/kg. Data on two South African trials reported in 1994 were resubmitted. The conditions in one trial (0.75 kg ai/ha, 3 days PHI) were according to GAP (0.56-1.5 kg ai/ha, 3 days PHI). The residue was 0.23 mg/kg. Data on ten American and two Canadian trials were submitted, but there was no relevant GAP. The Canadian trials had already been reported to the 1994 Meeting.

One supervised trial in New Zealand reported in the 1994 monograph was at 1.0 kg ai/ha, 0.067 kg ai/hl with 1-16 days PHI, close to the conditions of GAP (0.75 kg ai/ha, 0.075 kg ai/hl, 3 days PHI): the residues were 0.19 and 0.93 mg/kg at 3 and 7 days PHI.

The Meeting decided not to use the data from the Australian trials, only one of which complied with GAP, for the estimation of a maximum residue level because their population was different from that of the others and there were insufficient data.

The residues (mg/kg) in the 9 trials with treatments at maximum levels complying with GAP were <0.05 (Brazil), 0.814 and 0.717 (Japan), 0.05 (Spain), 0.26, 0.29, 0.38 and 0.45 (France), and 0.93 (New Zealand).

The residues from the 9 trials in rank order were <0.05, 0.05, 0.26, 0.29, 0.38, 0.45, 0.717, 0.814 and 0.93 mg/kg. The Meeting estimated a maximum residue level of 1 mg/kg and an STMR level of 0.38 mg/kg for acephate in tomatoes.

Processing studies

Tomatoes. The concentration factors from 3 trials were 1.29, 0.99 and 1.13 for washed fruit and 0.39, 0.63 and 0.50 for canned whole fruit. The mean concentration factors were 1.14 and 0.51 respectively. Washing appears to have no significant effect on the residue. The Meeting estimated an STMR-P of 0.19 mg/kg for canned tomatoes by applying the mean concentration factor to the STMR for tomatoes (0.38 mg/kg). The concentration factor for peeled tomatoes was calculated to be 0.85 from two trials, but the Meeting did not estimate an STMR-P because peeled tomato is only an intermediate product.

Concentration factors for canned juice, bulk paste, canned purée, wet pomace and dry pomace were 0.93, 4.0, 1.79, 0.60 and 1.0 from a single trial and STMR-P levels were calculated as 0.35, 1.52, 0.68, 0.23 and 0.38 respectively.

Cooking studies were carried out on tomatoes, cabbage and broccoli. Boiling for thirty minutes had no measurable effect on the residue levels of acephate.

Monitoring data

A total of 4,357 samples of peaches, nectarines, plums, grapes, tomatoes, lettuce and endive were monitored for acephate in The Netherlands in 1991-1994. The detection frequency ranged from 0.4% for plums and endive to 4.7% for nectarines and the highest mean residue was 0.05 mg/kg in lettuce in 1994.

Market basket survey

A market basket survey for acephate and methamidophos was carried out at 24 locations in the USA in 1984 and 1985. Acephate was found in 6 of 62 collected commodities; the highest residue was 0.72 mg/kg in green sweet peppers.

Farm gate to consumer studies

Farm gate to consumer studies on 5 commodities were carried out in the USA in 1985. Residues of acephate were reduced by 90% from field to supermarket in lettuce, 82% from field to canned product in snap beans, 13% from field to supermarket in bell peppers, 69% from field to processing and freezing in cauliflower and 93% from field to blanching and freezing in Brussels sprouts.

RECOMMENDATIONS

The Meeting estimated the maximum residue and STMR levels shown below. The maximum residue levels are recommended for use as MRLs.

Definition of the residue for compliance with MRLs and for estimation of dietary intake: acephate

Commodity		Maximum residue level, mg/kg		PHI, days	STMR, mg/kg[1]
CCN	Name	New	Previous		
VB 0400	Broccoli	2	W[2]	14	0.11
VB 0041	Cabbages, Head	2	W	7-14	0.33
VB 0404	Cauliflower	2	W	3-14	0.11
VO 0448	Tomato	1	W	1-14	0.38

[1] Since separate maximum residue levels and STMRs have been estimated for methamidophos, arising from the use of either acephate or methamidophos, the risk arising from methamidophos residues should be assessed separately against the methamidophos ADI and acute RfD

[2] Earlier recommendation for MRL withdrawn by 1994 JMPR

The estimated STMR-P levels listed below for acephate in processed commodities are recommended for use in estimates of dietary intake.

Raw agricultural commodity	STMR (mg/kg)	Processed commodity	STMR-P (mg/kg)
Tomato	0.38	Canned tomato	0.19
		canned juice	0.35
		bulk paste	1.52
		canned puree	0.68
		wet pomace	0.23
		dry pomace	0.38

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ALDICARB (117)

EXPLANATION

Aldicarb was last evaluated for residues in 1994 within the CCPR periodic review programme. The Meeting estimated maximum residue levels for a wide range of commodities including a temporary maximum residue level of 0.5 mg/kg for potato pending the submission of data on supervised trials corresponding with current use patterns. The previous recommendation for an MRL for banana was withdrawn because the use pattern had changed.

The manufacturer submitted extensive residue data obtained from recent supervised trials on potatoes and bananas reflecting the currently recommended uses. The new results are summarized and evaluated in this monograph together with other information provided by member countries.

The Meeting was informed that additional trials are being conducted in the USA on potatoes, and that a residue programme has been initiated in the French West Indies and West Africa on bananas to generate additional data to support the new use pattern and to determine whether the PHI can be further refined. The residue data will be submitted to the JMPR when the studies are completed.

USE PATTERN

The use patterns on potatoes reported by the 1994 Meeting are still in effect, except in the USA where requirements such as the use of positive-displacement application (PDA) equipment, the exclusion of in-furrow irrigation and new PHIs of 100 and 150 days in Florida and the Pacific Northwest States, respectively, have been included on the labels. The approved rate for PDA application is 3.36 kg ai/ha.

The countries in which aldicarb is still used on bananas are Argentina, Cameroon, Egypt, France, Ivory Coast, South Africa and Zimbabwe. The recommended use has been changed to a maximum of two applications per year, a maximum rate of 2 g ai per plant, and a PHI of 180 days.

RESIDUES RESULTING FROM SUPERVISED TRIALS

Supervised field trials were carried out with granular formulations of aldicarb. The samples were mainly analysed by HPLC methods which determine aldicarb, its sulfoxide and sulfone individually. The limits of determination were typically around 0.01-0.02 mg/kg for each residue component. In some of the trials the residues were oxidized to the sulfone and determined by GLC with an FPD. The residues, expressed as aldicarb sulfone, are shown in the following Tables.

Bananas. Trials were conducted in Cameroon, Egypt, France (Martinique), and the Ivory Coast. Aldicarb was applied to the soil at the recommended maximum rate of 2 g ai/plant, corresponding to 3.6-4 kg ai/ha. Samples were taken from 3 to >360 days after the last application. The residues were determined in composite samples consisting of 7-12 fingers, or in individual fingers. In some experiments the peel and pulp were analysed separately. The results are summarized in Table 1.

Potatoes. The reported supervised trials represented a wide geographical distribution and covered a period of 15 years. The samples were analysed either by GLC or HPLC methods based on the principles described in the 1994 evaluation. The major residue component was aldicarb sulfoxide.

Supervised trials were carried out in Argentina, Belgium, Canada, Czechoslovakia, Ecuador, Germany, Greece, Hungary, Italy, Netherlands, Spain, and the UK between 1978 and 1992. The trial conditions reported and the residues detected in composite samples are summarized in Table 2. In addition, a number of "commercial trials" were reported from South Africa in a summarized form without details of the trial conditions, the actual dosage rates or the analytical methods. The data from the trials are also given in Table 2, but are distinguished from the fully reported trials by shading.

Table 1. Aldicarb residues[1] in bananas from supervised trials.

Country, Location, Year	Variety	Application		PHI, days	Portion of commodity analyzed	Residues mg/kg			Ref.
		No., Form.	Rate, ai, kg/ha &/or g/plant			Mean	Min.	Max.	
Cameroon Nyombe, 1992	Grand Naine		3.6 2 g/plant	33 63 92 124	Whole fruit[2]	0.11 0.23 0.11 0.04	0.05 0.09 0.04 0.02	0.20 0.41 0.19 0.08	JM27
Cameroon Penja, 1993	Grand Naine		3.6 2 g/plant	114	Whole fruit[2]	0.02	0.02	0.03	JM28
Egypt, Menoufia 1992	Balika		2 g/plant	87	Whole fruit[2]	0.02	0.02	0.03	JM29
			2.55 g/plant	154	Whole fruit[2]	0.02	0.02	0.02	
France (Martinique) Danoux, 1991	Poyo	3	2 g/plant	119	Pulp of mature yellow fruit	0.04			JM30
Barriere 1991	Grand Naine	3		175-205	Pulp of mature yellow fruit	<0.01			
Moulin a vent, 1991	Grand Naine	2		148-175	Pulp of mature yellow fruit	0.04			
Camille 1990	Grand Naine	2		>360	Pulp of mature yellow fruit	0.02			
Daroux 1991	Grand Naine	2		85-114	Pulp of mature yellow fruit	0.02			
Mangot 1991		3		175-205	Pulp of mature yellow fruit	0.025			
Cloture 1991	Grand Naine	2		175-205	Pulp of mature yellow fruit	0.02			
La Pointe 1991	Poyo	2		85-114	Pulp of mature yellow fruit	0.025			
Sapotille 1991	Grand Naine	3		146-173	Pulp of mature yellow fruit	0.01			
Ravine 1991	Poyo	1		85-114	Pulp of mature yellow fruit	0.06			
Terre Grasse 1991	Grand Naine	1		119	Pulp of mature yellow fruit[2]	0.13			
Etuve 1991	Grand Naine	1		85-114	Pulp of mature yellow fruit[2]	0.10			
Savane	Grand	1	2 g/plant	94	Pulp of mature	0.01			

Country, Location, Year	Variety	Application		PHI, days	Portion of commodity analyzed	Residues mg/kg			Ref.
		No., Form.	Rate, ai, kg/ha &/or g/plant			Mean	Min.	Max.	
1991	Naine				yellow fruit				
CAF9 1991	Grand Naine	1		55-85	Pulp of mature yellow fruit	0.05			
Papaye 1991	Poyo	1		113-143	Pulp of mature yellow fruit	0.04			
Fefe 1991	Grand Naine	1		122	Pulp of mature yellow fruit	0.01			
Dinde 1991	Grand Naine	1		151	Pulp of mature yellow fruit	0.03			
Beauvallon 1991	Grand Naine	3		88	Pulp of mature yellow fruit	0.29			
Neuf Chateau 1991	Grand Naine	4		86-89	Pulp of mature yellow fruit	0.12			
Lamberty 1991	Grand Naine	1		167	Pulp of mature yellow fruit	0.03			
La Rose 1991	Grand Naine	1		126	Pulp of mature yellow fruit[2]	0.09	0.05	0.18	
Mapoue 1991	Poyo	2		114-144	Pulp of mature yellow fruit[2]	0.07	0.02	0.18	
Carangaise 1991	Poyo/G. N	2		94	Pulp of mature yellow fruit[2]	0.15	0.03	0.25	
France/ Guadeloupe 1991	Grand Naine	1	2 g/plant	100	Pulp of mature green fruit[2]	0.172	0.04	0.75	JM31
Martinique 1993	Grand Naine	1	2 g/plant	89	Pulp of mature green fruit[2]	0.39	0.08	0.81	
Martinique 1987		1	2 g/plant	3	Whole fruit[3] Pulp	0.015 0.012	0.01	0.02	JM32
				7	Whole fruit[3] Pulp	0.067 0.056	0.02	0.09	
				14	Whole fruit[3] Pulp	0.21 0.20	0.09	0.41	
				30	Whole fruit[3] Pulp	0.20 0.16	0.04	0.29	
				45	Whole fruit[3] Pulp	0.35 0.28	0.02	0.53	
				60	Whole fruit[3] Pulp	0.50 0.45	0.23	0.70	
				75	Whole fruit[3] Pulp	0.23 0.18	0.05	0.38	
				90	Whole fruit[3] Pulp	0.31 0.26	0.12	0.38	
				120	Whole fruit[3] Pulp	0.077 0.071	0.01	0.11	
				150	Whole fruit[3] Pulp	0.037 0.035	0.01	0.11	
Grand Reduit 1988	Grand Naine	3	2 g/plant	19 19	Green fruit[4], whole Peel Pulp Yellow fruit, whole Peel	0.11 0.11 0.07 0.11 0.15			JM33

Country, Location, Year	Variety	Application		PHI, days	Portion of commodity analyzed	Residues mg/kg			Ref.
		No., Form.	Rate, ai, kg/ha &/or g/plant			Mean	Min.	Max.	
					Pulp	0.085			
Moulin l'Etang 1988	Grand Naine	3	2 g/plant	93 103	Green fruit[4], whole Peel Pulp Yellow fruit, whole Peel Pulp	0.04 0.06 0.04 0.13 0.20 0.08			
Union II 1989	Grand Naine	5G	2 g/plant	27 59 87	Pulp of mature fruit	< 0.11 < 0.06 < 0.04	0.02 < 0.02 < 0.02	0.23 0.09 0.08	JM34
		10G	2 g/plant	27 59 87	Pulp of mature fruit	< 0.09 0.05 < 0.03	< 0.03 < 0.03 < 0.02	0.13 0.06 <0.03	
		15G	2 g/plant	27 59 87	Pulp of mature fruit	0.06 < 0.05 < 0.03	0.04 0.02 < 0.02	0.09 0.07 0.05	
Gradis 1989	Poyo	1 5G	2 g/plant	27 60 88	Pulp of mature fruit	0.18 0.20 0.18	0.06 0.14 0.11	0.28 0.30 0.34	
		10G	2 g/plant	27 60 88	Pulp of mature fruit	0.17 0.39 0.19	0.12 0.14 0.08	0.26 0.65 0.32	
		15G	2 g/plant	27 60 88	Pulp of mature fruit	0.26 0.17 0.16	0.05 0.12 0.09	0.83 0.23 0.29	
Ressource 1989	Grand Naine	1 5G	2 g/plant	27 81 88	Pulp of mature fruit	0.11 0.13 0.23	0.04 0.07 0.16	0.20 0.27 0.3	
		10G	2 g/plant	27 81 88	Pulp of mature fruit	0.18 0.23 0.18	0.11 0.19 0.15	0.36 0.39 0.27	
		15G	2 g/plant	27 81 88	Pulp of mature fruit	0.43 ≤0.23 0.19	0.27 <0.02 0.07	0.98 0.55 0.29	
Ivory Coast Azaguie, 1992		1	4.0 ca. 2.0 g/plant	31 60 90 122	Whole fruit[2]	0.16 0.28 0.40 0.23	0.05 0.08 0.06 0.06	0.28 0.48 0.82 0.47	JM35
Thomasset 1993		1	4.0 ca. 2.0 g/mat	92 120	Whole fruit[2]	0.23 0.17	0.14 0.08	0.33 0.28	JM36
Bamacomoe 1993	Grand Naine		4.0 ca. 2.0 g/plant	94 123	Whole fruit[2]	0.20 0.07	0.05 0.05	0.31 0.10	
			3.6 ca. 2 g/plant	≥90	Whole fruit[2]	0.07	0.02	0.12	JM37
Damotte	Grand Naine		4.0 ca. 2 g/plant	≥90	Whole fruit	0.036			JM38
			4.0 ca. 2 g/plant	≥90	Whole fruit	0.032			

[1]Residues of aldicarb its sulfoxide and sulfone were either measured and reported individually or determined as sulfone after oxidation. In the Table the residues are expressed as aldicarb sulfone.

[2]Individual fingers were analyzed.

[3]Samples were taken from the upper and lower parts of the bunch. At each PHI the pulp and peel of 10 fruits were analyzed separately.

Whole fruit: residues in the peel and pulp of individual fingers were determined separately, but only the mean values of residues found in 10 fingers were reported.

Pulp: reported as the mean value and range of residues in the pulp of individual fingers.

[4]Results for mature green and yellow fruits are each based on 2 individual samples. Shipment of fruit lasted approximately 13 days; an additional 6 days was allowed for ripening to the yellow fruit stage.

Table 2. Aldicarb residues[1] in composite potato samples from supervised trials. All single applications except where otherwise shown.

Country Year	Application		Residues (mg/kg) at days after application							Ref.
	Rate, ai	Type[2]	38-56	57-63	64-84	85-99	100-110	111-140	141-206	
UK 1983	8.6 g/100m	F.							<0.05	JM16
	4.3 g/100m	S.					<0.05			
	8.6 g/100m	F.						<0.05		
	4.3 g/100m	S.					<0.09			
	8.6 g/100m	F.							<0.05	
	4.3 g/100m	S.				<0.05				
	8.6 g/100m	S.				<0.05				
UK 1982	12.8 g/100m	F.							0.09 0.10	JM17
	8.6 g/100m	F.							<0.05 0.07	
	8.6 g/100m	S.					<0.05			
	8.6 g/100m	F.							0.08 0.07	
	8.6 g/100m	F.						<0.05		
	8.6 g/100m	S.				<0.05				
	4.3 g/100m	F.						<0.05		
Argentina 1991	2 x 1.0 kg/ha	S.	0.0008							JM01
	2.0 kg/ha	F.						0.0006		
	2.0 kg/ha	S.		0.0006						
Belgium 1988	1.0 kg/ha	F.	0.35	0.18	0.19	0.09[3]	0.08	0.03		JM02
	3.0 kg/ha	F.	1.32	0.51	0.45	0.47	0.14	0.20		
	5.0 kg/ha	F.	1.95	1.35	0.69	0.47	0.53	0.29		
	10.0 kg/ha	F.	5.25	2.54	1.12	0.74	0.53	0.60		

Country Year	Application		Residues (mg/kg) at days after application							Ref.
	Rate, ai	Type[2]	38-56	57-63	64-84	85-99	100-110	111-140	141-206	
	25 kg/ha	F.	16.12	8.50	6.77	2.87	3.33	194		
Canada 1985	2.0 kg/ha		1.53	0.90	0.45 0.47 0.49	0.19 0.30	<0.02			JM03
	1.6 kg/ha		0.48		0.73 0.26 0.15	0.26 0.43	0.03			
	2.0 kg/ha		0.34	0.11	0.11	0.07				
Czechoslovakia 1987	2 x 5.0 kg/ha	B. B.						0.03 0.35		JM04
Germany 1984	6.0 g/100m	F.					0.114	0.067	0.038	JM06
	6.0 g/100m	F. i				0.072 0.044		0.03		
	6.0 g/100m	F.				0.022		0.016 0.012		
	6.0 g/100m	F.				0.104	0.101	0.046		
	6.0 g/100m	F.			0.343 0.322	0.217				
	6.0 g/100m	F.			0.056 0.038	0.009				
Greece 1986	3.5 kg/ha	B.						0.09		JM07
	7.0 kg/ha	B.						0.15		
	2.5 kg/ha	F.						<0.03		
	5.0 kg/ha	F.						0.12		
Hungary 1984	4.0 kg/ha	B.				<0.015				JM08
Italy 1992	1.425 kg/ha							0.035		JM09
	2.850 kg/ha							0.029		
Nethlnds 1982	1.0 kg/ha	F.							0.05	JM10
	1.0 kg/ha	F.							<0.03	
Nethlnds 1982	3.0 kg/ha	B.					0.09			JM11
	3.0 kg/ha	B.				0.12				

Country Year	Application		Residues (mg/kg) at days after application							Ref.
	Rate, ai	Type[2]	38-56	57-63	64-84	85-99	100-110	111-140	141-206	
	3.0 kg/ha	B.						<0.03		
	3.0 kg/ha	B.						<0.03		
	3.0 kg/ha	B.						<0.03		
	3.0 kg/ha	B.				0.06				
	3.0 kg/ha	B.							<0.03	
	3.0 kg/ha	B.							<0.03	
	3.0 kg/ha	B.						<0.03		
	3.0 kg/ha	B.							<0.03	
	3.0 kg/ha	B.						0.07		
	3.0 kg/ha	B.						<0.03		
	3.0 kg/ha	B.							0.05	
	3.0 kg/ha	B.					<0.03			
	3.0 kg/ha	B.							0.05	
	3.0 kg/ha	B.						0.25		
	3.0 kg/ha	B.						0.04		
	3.0 kg/ha	B.							<0.03	
	3.0 kg/ha	B.							0.03	
	3.0 kg/ha	B.							0.07	
	3.0 kg/ha	B.							<0.03	
	3.0 kg/ha	B.							<0.03	
	3.0 kg/ha	B.							0.18	
	3.0 kg/ha	B.							0.03	
	3.0 kg/ha	B.							<0.03	
	3.0 kg/ha	B.							0.09	
	3.0 kg/ha	B.						<0.03		
	3.0 kg/ha	B.						<0.03		
	3.0 kg/ha	B.							0.03	
	3.0 kg/ha	B.							0.05	
	3.0 kg/ha	B.							<0.03	
South	7.5 kg/ha at				0.04			<0.01		JM12

Country Year	Application		Residues (mg/kg) at days after application							Ref.
	Rate, ai	Type[2]	38-56	57-63	64-84	85-99	100-110	111-140	141-206	
Africa 1989	planting									
	2 x 3.75 kg/ha		0.14			0.04				
	3.75 kg/ha at emergence		0.10			0.04				
	7.5 kg/ha at planting				0.09			0.07		
	2 x 3.75 kg/ha		0.38		0.31					JM13
	3.75 kg/ha at emergence		0.36		0.22					
South Africa 1990	5.25 kg/ha					2.8		0.61		JM14
	5.25 kg/ha					2.1		1.14		
	5.25 kg/ha					0.3		0.12		
	4.2 kg/ha					0.28				
	7.5 kg/ha					0.64		0.55		
	7.5 kg/ha					0.94		0.75		
	7.5 kg/ha					1.02		0.40		
	7.5 kg/ha					0.40		0.46		
	7.5 kg/ha					0.49		0.6		
	7.5 kg/ha					0.77		0.54		
	3.3 kg/ha					0.43				
	5.25 kg/ha					1.06 0.74				
	5.25 kg/ha					1.86 0.66		0.64 0.43		
	5.25 kg/ha					0.11 0.64		0.01 0.12		
	5.25 kg/ha at planting					0.18				
	7.5 kg/ha at planting					0.66				
	3.75+3.75 kg/ha			0.02						
	3.75 kg/ha at emergence							0.24		

Country Year	Application		Residues (mg/kg) at days after application							Ref.
	Rate, ai	Type[2]	38-56	57-63	64-84	85-99	100-110	111-140	141-206	
1991	7.5 kg/ha at planting					0.009				
	5.25 kg/ha					0.007				
	3.75+3.75 kg/ha			0.027						
	3.75 kg/ha					0.39		0.58		
	7.5 kg/ha					0.56		0.78		
Spain 1980	1.5 kg/ha								0.02	JM15
	1.5 kg/ha								0.2	
	1.5 kg/ha							0.03		
	1.5 kg/ha								0.04	
	4.1 kg/ha							0.08		
	1.5 kg/ha							0.30		
	1.5 kg/ha							<0.02		
	1.5 kg/ha								0.07	
	1.5 kg/ha							0.7		
	1.5 kg/ha								0.2	
	1.5 kg/ha								0.02	
	1.5 kg/ha								0.03	
	3.0 kg/ha								0.07 0.06 0.20 0.08	
	2.0 kg/ha								0.06	
	1.5 kg/ha								0.04	
	1.5 kg/ha							0.20		
	1.5 kg/ha								<0.02	
	1.5 kg/ha								0.02	
	1.5 kg/ha								<0.02	
	1.5 kg/ha								0.04	
	1.5 kg/ha								0.03	
	1.5 kg/ha								0.08	
	1.5 kg/ha							0.02		

Country Year	Application		Residues (mg/kg) at days after application							Ref.
	Rate, ai	Type[2]	38-56	57-63	64-84	85-99	100-110	111-140	141-206	
	3.4 kg/ha							0.40		
1978	1.57 kg/ha								<0.005	
	1.71 kg/ha								<0.005	
	2.0 kg/ha								<0.005	
	2.28 kg/ha								<0.005	
	1.86 kg/ha								<0.05	
	1.28 kg/ha								<0.05	
	2.43 kg/ha								0.07	
	1.71 kg/ha								0.01	
	2.0 kg/ha								0.005	
	2.14 kg/ha								0.007	
	1.86 kg/ha								0.01	
	1.57 kg/ha								<0.005	
1979	1.57 kg/ha					0.23				
	1.57 kg/ha							0.05		
	1.86 kg/ha								0.03	
	1.57 kg/ha								0.03	
	1.86 kg/ha								0.03	
	1.57 kg/ha								<0.005	
	1.57 kg/ha							0.03		
	1.57 kg/ha							0.03		
	1.57 kg/ha							0.02		
	1.57 kg/ha								0.07	
	3.5 kg/ha							0.01		
1981	2.5 kg/ha 3.5 kg/ha							0.01	<0.005	
	3.5 kg/ha								0.03	
	3.5 kg/ha								0.04	
	3.5 kg/ha								0.20	
	3.5 kg/ha								0.03	

Country Year	Application		Residues (mg/kg) at days after application							Ref.
	Rate, ai	Type[2]	38-56	57-63	64-84	85-99	100-110	111-140	141-206	
	5.0 kg/ha								0.07	
	3.5 kg/ha								<0.005	
1982	2.0 kg/ha				0.08					
	2.0 kg/ha				0.25					
	2.0 kg/ha				0.10					
	2.0 kg/ha						<0.05			
	2.0 kg/ha					0.10				
	1.5 kg/ha							0.02		
	2.0 kg/ha							0.03		
	2.66 kg/ha							0.06		
	1.5 kg/ha							0.10		
	2.0 kg/ha							0.06		
	2.0 kg/ha							<0.02		
1983	1.7 kg/ha								<0.05	
	2.0 kg/ha								<0.05	
	2.1 kg/ha								<0.05	
	2.4 kg/ha								<0.05	

[1]Residues expressed as aldicarb sulfone
[2]B.: broadcast application. F.: in furrow application. S.: side band or side dress application.
[3]No GAP PHI, tubers were ready for marketing at new potato stage

Many studies have been conducted to determine the level of residues in potatoes in the USA. and the UK during 1990-1993. In these trials large number of individual potato tubers taken from single sites were analysed in order to obtain information on the within-field and the field-to-field variation of residues, depending on the mode of application, method of irrigation and climatic conditions. The trial conditions, numbers of tubers analysed and the sum of the carbamate residues expressed as aldicarb sulfone are given in Table 3. The residues deriving from trials according to the different use patterns established for the Northwest States and for Florida are underlined. In estimating maximum residue levels the growing and climatic conditions of the Northern States (Washington, Idaho, Oregon, Michigan, North Dakota, Pennsylvania and Maine) and the Southern States (Florida, Texas) of the USA were considered to be comparable.

The many residues measured in individual tubers provide an excellent basis for the precise estimation of acute dietary risk deriving from the use of the compound. However, since the range of residues in individual tubers is much wider than the range of average residues in composite samples consisting of 10-12 tubers, the results cannot be used directly for estimating maximum residue levels.

Therefore composite random samples consisting of 12 tubers, the recommended sample size according to FAO Guidelines (FAO, 1990), were drawn with replacement from some of the primary residue data sets obtained from individual tubers according to a computer programme described recently (Ambrus, 1996). The programme draws the composite samples and calculates their average residues from the residue content of the potatoes selected for each sample. Figures 1-6 show the relative frequency distribution of residues in individual potatoes and in composite samples. It can be seen that the distributions of the residues in the primary samples are far from normal, so distribution-free statistics have to be used for their analysis.In such cases a minimum of 90 samples are required to estimate the 95th percentile of the population with 99% confidence (FAO, 1993). Composite samples were therefore drawn from primary sample populations taken from uniformly treated areas (excluding the end sections of rows) and consisted of at least 100 samples with one exception where there were 79 samples.The means, medians, and minimum and maximum residues found in 100 replicate composite samples are also presented in Table 3 together with the corresponding primary residue data. Since the average residues in composite samples are used to determine whether the crop had been treated according to GAP, the maximum residues found in composite random samples drawn by the computer are underlined in the Table.

Six field trials were conducted in different locations of the UK at recommended and double rates. Fifty individual tubers were collected from each site and analysed for aldicarb residues (Maycey *et al.*, 1991; Brockelsby *et al.*, 1991). The average residues ranged from 0.14 to 0.46 mg/kg following applications according to GAP.

Table 3. Aldicarb residues[1] in individual potato tubers deriving from supervised trials. All single applications.

Location,[2] Trial no.	Application	PHI, days	No. of samples	Residues (mg/kg) at days	Ref.

	Rate, ai	Type[3]			Mean	Median	Min.	Max.	
UK trials, 1990									
Isle of Wright	3.36 kg/ha	B	90	49	0.46	0.455	0.07	0.9	JM18
Kilverstone	11.4 g/100m	F	84	50	0.14	0.099	0.02	0.44	JM18
Holbeach	3.36 kg/ha	B	91	50	0.21	0.175	0.05	0.84	JM18
Jersey	2.24 kg/ha	B	79	50	0.40	0.333	0.02	0.89	JM18
Scunthorpe	5.6 kg/ha	B	95	50	0.64	0.584	<0.02	1.98	JM18
Scunthorpe	5.6 kg/ha	B	95	50	0.66	0.613	<0.02	1.99	JM19
			121	10	0.32	0.274	0.06	0.62	
			145	10	0.57	0.327	0.06	1.23	
			171	10	0.23	0.204	0.05	0.54	
USA trials, 1990-1993									
WA. 90-131	3.27 kg/ha	B, f.i.	156	100	0.09		<0.02	0.48	JM21
WA. 90-132	3.35 kg/ha	B. f.i.	160	40	0.30	0.07	<0.02	2.82	JM21
WA. 90-133	3.36 kg/ha	B. f.i.	160	20	0.08	0.04	<0.02	0.57	JM21
WA. 90-134	3.27 kg/ha	B. f.i.	126	20	0.08	0.065	<0.02	0.37	JM21
WA. 90-135	3.35 kg/ha	B. f.i.	160	60	0.24	0.065	<0.02	5.30	JM21
WA. 90-136	3.36 kg/ha	B. f.i.	160	20	0.15	0.12	0.04	0.35	JM21
WA. 90-137	3.38 kg/ha	B. o.i.	159	100	0.07	0.05	<0.02	0.32	JM21
			Composite Samples		0.073	0.07	0.04	0.13	
WA. 90-138	3.38 kg/ha	B. o.i.	155	100	0.08	0.06	<0.02	0.49	JM21
			Composite samples		0.08	0.076	0.04	0.15	
WA. 90-139	3.35 kg/ha	B. o.i.	156	100	<0.02	<0.02	<0.02	0.02	JM21
WA 90-140	3.32 kg/ha	Be. o.i.	124	100	0.11	0.1	0.01	0.39	JM21
			Composite samples		0.11	0.108	0.072	0.174	
WA. 90-141	3.32 kg/ha	Be. o.i.	118	45	0.15	0.13	0.05	0.28	JM21
WA. 90-142	3.37 kg/ha	Be. o.i.	119	100	0.17	0.15	0.03	0.71	JM21
			Composite samples		0.163	0.160	0.09	0.26	
ID. 90-147	3.20 kg/ha	B. f.i.	139	20	0.02	0.015	<0.02	0.10	JM21
ID. 90-148	2.93 kg/ha	B. f.i.	108	20	<0.02	<0.02	<0.02	0.01	JM21
ID. 90-149	3.40 kg/ha	B. f.i.	135	20	0.21	0.115	0.03	0.91	JM21
ID. 90-150	3.82 kg/ha	B. f.i.	142	20	0.02	0.01	<0.02	0.17	JM21
ID. 90-151	3.42 kg/ha	B. o.i.	129	100	0.05	0.04	<0.02	0.21	JM21
			Composite samples		0.048	0.045	0.021	0.099	

Location,[2] Trial no.	Application		PHI, days	No. of samples	Residues (mg/kg) at days				Ref.
	Rate, ai	Type[3]			Mean	Median	Min.	Max.	
ID. 90-152	3.74 kg/ha	B. o.i.	146	100	0.03	0.01	<0.02	0.38	JM21
			Composite samples		0.031	0.027	0.011	0.063	
ID. 90-153	3.48 kg/ha	Be. o.i.	94	79	0.12	0.08	<0.02	0.74	JM21
			Composite samples		0.124	0.112	0.024	0.346	
CO. 92-112	3.34 kg/ha	F. o.i.	133	30[4]	0.05		<0.02	0.11	JM22
				24[5]	0.22		<0.02	1.11	JM22
CO. 92-113	3.34 kg/ha	F. o.i.	126	30[4]	0.06		0.04	0.15	JM22
				24[5]	0.10		<0.02	0.68	
NE. 92-114	3.18 kg/ha	F. o.i.	104	30[4]	0.03		<0.02	0.15	JM22
				24[5]	0.04		0.02	0.09	
FL. 92-115	3.21 kg/ha	F. o. i.	102	30[4]	0.21		0.04	0.57	JM22
				24[5]	0.25		0.02	1.04	
MN. 92-118	3.18 kg/ha	F. o.i.	109	30[4]	0.17		0.07	0.40	JM22
				24[5]	0.18		<0.02	0.98	
OR. 92-119	3.60 kg/ha	F. o.i.	167	30[4]	0.04		0.02	0.04	JM22
				24[5]	0.03		<0.02	0.06	
OR. 92-120	3.40 kg/ha	F. o.i.	167	30[4]	0.04		0.02	0.05	JM22
				24[5]	0.03		<0.02	0.06	
MT. 92-121	3.12 kg/ha	F. o.i.	107	30[4]	0.10		<0.02	0.26	JM22
				24[5]	0.23		<0.02	1.75	
MI. 92-122	6.63 kg/ha	F. o.i.	110	30[4]	0.16		0.04	0.56	JM22
				24[5]	0.24		<0.02	1.16	
MI. 92-123	6.63 kg/ha	F. o.i.	110	30[4]	0.40		0.05	0.91	JM22
				24[5]	0.53		<0.02	3.13	
WA. 92-124	3.20 kg/ha	F. o.i.	152	30[4]	<0.02		<0.02	0.02	JM22
				24[5]	<0.02		<0.02	0.02	
WA. 92-125	3.64 kg/ha	F. o.i.	140	30[4]	0.03		<0.02	0.06	JM22
				24[5]	0.03		<0.02	0.04	
ID. 92-126	3.74 kg/ha	F. o.i.	136	30[4]	0.05		0.02	0.13	JM22
				24[5]	0.07		<0.02	0.30	
ID. 92-127	3.74 kg/ha	F. o.i.	139	30[4]	0.02		<0.02	<0.02	JM22
				24[5]	<0.02		<0.02	0.02	
CA. 92-128	3.18 kg/ha	F. o.i.	145	30[4]	0.02		<0.02	0.04	JM22

Location,[2] Trial no.	Application		PHI, days	No. of samples	Residues (mg/kg) at days				Ref.
	Rate, ai	Type[3]			Mean	Median	Min.	Max.	
				24[5]	0.01		<0.02	0.06	
ND. 92-129	3.46 kg/ha	F. o.i.	118	30[4]	0.08		0.04	0.15	JM22
				24[5]	0.07		<0.02	0.20	
TX. 92-130[7]	3.42 kg/ha	F. o.i.	109	30[4]	0.12		0.04	0.35	JM22
				24[5]	0.12		0.02	0.57	
ME. 90-107	2.44 kg/ha	BR.	97	100	0.14	0.13	0.04	0.34	JM24
			Composite samples		0.14	0.135	0.09	0.19	
WA. 90-129	3.32 kg/ha	BR.	97	300	0.29	0.24	0.04	1.3	JM24
			Composite samples		0.296	0.277	0.183	0.458	
WA. 90-130	3.30 kg/ha	BR.	123	100	0.06	0.04	0.02	0.22	JM24
			Composite samples		0.055	0.053	0.04	0.081	
ID. 90-146	3.29 kg/ha	F.	146	100	0.019	0.02	<0.02	0.06	JM24
			Composite samples		0.022	0.022	<0.02	0.03	
PA. 90-191	2.93 kg/ha	F.	132	100	0.045	0.04	0.02	0.15	JM24
			Composite samples		0.045	0.043	0.037	0.059	
FL. 90-026	3.39 kg/ha	F.	106	100	0.085	0.07	0.02	0.54	JM24
			Composite samples		0.083	0.08	0.05	0.15	
MI. 90-095	3.36 kg/ha	BR.	100	100	0.09	0.075	0.02	0.37	JM24
			Composite samples		0.091	0.088	0.056	0.14	
MI. 90-096	3.52 kg/ha	F.	120	100	0.05	0.04	0.02	0.15	JM24
			Composite samples		0.056	0.051	0.034	0.099	
FL. 93-001	3.02 kg/ha	F. PDA	104	85	0.034[5]		<0.02	0.23	JM25
					0.075[4]		0.022	0.34	
	3.81 kg/ha	F. GFA	104	84	0.12[5]		0.04	0.59	
FL. 93-002	3.14 kg/ha	F. PDA	104	81	0.025[5]		<0.02	0.065	JM25
					0.061[4]		0.022	0.27	
	3.58 kg/ha	F. GFA	104	75	0.13[5]		0.04	1.3	
TX. 93-003	3.28 kg/ha	F. PDA	106	100	0.23[5]		0.02	1.2	JM25
	4.16 kg/ha	F. GFA	106	100	0.50[5]		0.05	7.7	
TX. 93-004	3.33 kg/ha	F. PDA	106	100	0.09[5]		<0.02	0.31	JM25

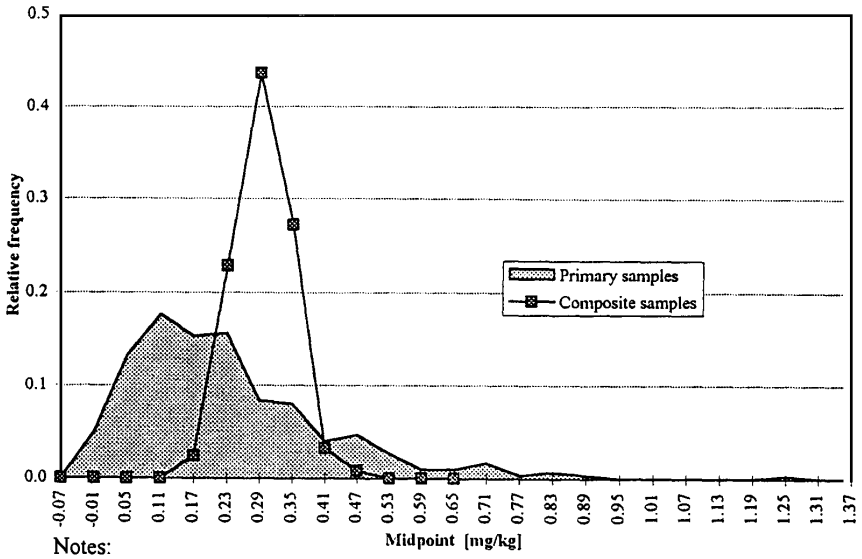
Location,[2] Trial no.	Application		PHI, days	No. of samples	Residues (mg/kg) at days				Ref.
	Rate, ai	Type[3]			Mean	Median	Min.	Max.	
	4.13 kg/ha	F. GFA	106	97	0.26[5]		<0.02	3.2	
OR. 93-005	3.33 kg/ha	F. PDA	152	94	0.01[5]		<0.02	0.21	JM25
			152	30	0.03[4]		<0.02	0.072	
	3.51 kg/ha	F. GFA	152	100	0.069[5]		<0.02	1.2	
OR. 93-006	3.30 kg/ha	F. PDA	152	87	<0.02[5]		<0.02	0.046	JM25
				30	≤0.02[4]		<0.02	0.023	
	3.62 kg/ha	F. GFA	152	90	0.03[5]		<0.02	0.165	
OR. 93-007	3.34 kg/ha	F. PDA	149	93	0.059[5]		<0.02	0.22	JM25
				30	0.053[4]		0.044	0.10	
	3.67 kg/ha	F. GFA	149	95	0.15[5]		0.022	0.98	
OR. 93-008	3.33 kg/ha	F. PDA	170	90	<0.02[5]		<0.02	0.063	JM25
				30	0.024[4]		<0.02	0.065	
	3.35 kg/ha	F. GFA	170	93	0.034[5]		<0.02	0.28	
WA. 93-009	3.33 kg/ha	F. PDA	152	100	0.06[5]		<0.02	0.29	JM25
				30	0.18[4]		0.049	0.61	
	3.6 kg/ha	F. GFA	152	96	0.46[5]		0.042	3.94	
WA. 93-010	3.33 kg/ha	F. PDA	142	98	0.06[5]		<0.02	0.20	JM25
				30	0.11[4]		0.042	0.32	
	3.64 kg/ha	F. GFA	152	100	0.79[5]		0.04	6.8	
WA. 93-011	3.08 kg/ha	F. PDA	152	100	0.030[5]		<0.02	0.112	JM25
				30	0.048[4]		0.042	0.094	
	4.55 kg/ha	F. GFA		100	0.072[5]		<0.02	0.342	
WA. 93-012	3.33 kg/ha	F. PDA	151	100	<0.02[5]		<0.02	0.048	JM25
				30	≤0.02[4]		<0.02	0.045	
	3.85 kg/ha	F. GFA	151	98	0.014[5]		<0.02	0.084	
ID. 93-013	3.36 kg/ha	F. PDA	131	101	<0.02[5]		<0.02	1.12	JM25
				30	0.14[4]		0.04	0.51	
	4.03 kg/ha	F. GFA	131	98	0.41[5]		0.042	7.2	
ID. 93-014	3.25 kg/ha	F. PDA	151	100	0.09[5]		<0.02	1.2	JM25
				30	0.09[4]		<0.02	0.26	
	3.81 kg/ha	F. GFA	151	100	0.69[5]		0.022	12.8	
ID. 93-015	3.36 kg/ha	F. PDA	146	100	<0.02[5]		<0.02	0.045	JM25
				30	≤0.02[4]		<0.02	0.17	
	3.58 kg/ha	F. GFA	146	100	<0.02[5]		<0.02	0.049	

Location,[2] Trial no.	Application		PHI, days	No. of samples	Residues (mg/kg) at days				Ref.
	Rate, ai	Type[3]			Mean	Median	Min.	Max.	
ID. 93-016	3.47 kg/ha	F. PDA	146	100	0.026[5]		<0.02	0.25	JM25
				30	0.034[4]		<0.02	0.17	
	4.03 kg/ha	F. GFA	146	100	0.043[5]		<0.02	0.39	

[1]Expressed as aldicarb sulfone (aldicarb residues mg/kg = aldicarb sulfone mg/kg x 0.856)
[2]States of the USA: WA Washington; ID Idaho; CO Colorado; NE Nebraska; FL Florida; MN Minesota; OR Oregon; MT Montana; MI Michigan; CA California; ND North Dakota; TX Texas; ME Maine; PA Pennsylvania.
[3]B broadcast application; Be broadcast at emergence; BR band over row application at emergence; F in furrow application; GFA gravity-flow application; PDA positive-displacement application; f.i. furrow irrigation; o.i. overhead irrigation
[4]Samples were taken from the centre of the row
[5]Samples were taken from the end of the row

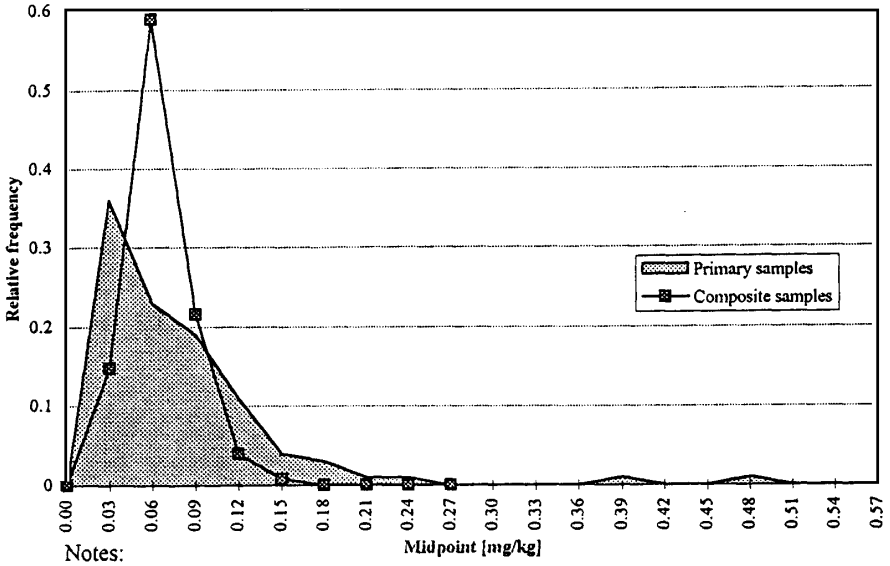
Nineteen field trials were conducted in the Pacific Northwest region of the USA to determine whether irrigation methods affected the magnitude of aldicarb residues in potatoes treated with a 15G formulation (Tew, 1992). Overhead and in-furrow irrigation methods were compared. All plots were treated with the nominal maximum label rate of 3.36 kg ai/ha with commercial ground equipment. Plots irrigated in-furrow were treated at planting, while overhead irrigation was either at planting or at emergence. A total of 340 tubers from plots irrigated in-furrow and 824 tubers from plots treated by overhead irrigation plots were analysed.

Figure 1. Relative frequency distribution of aldicarb sulfone residues in potato samples from trial 90-129.



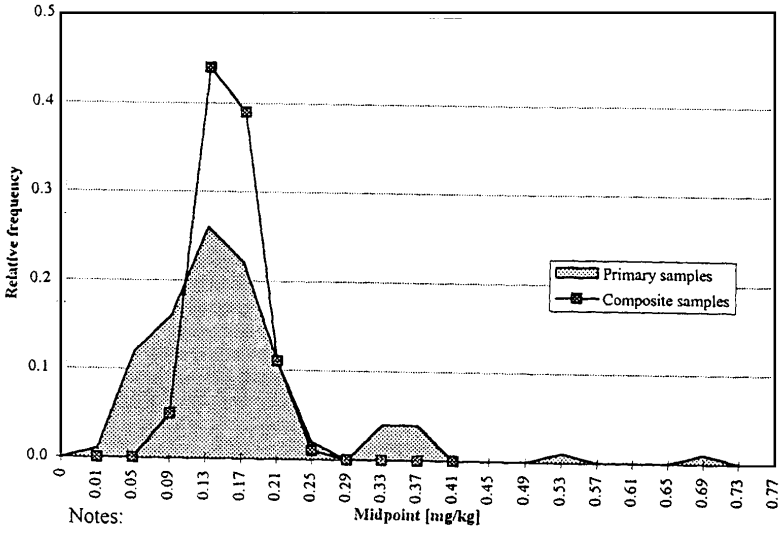
Notes:
Primary samples:
Mean = 0.295 Standard Deviation = 0.1820 Median = 0.255
Composite samples:
Mean = 0.294 Standard Deviation = 0.04989 Median = 0.2875

Figure 2. Relative frequency distribution of aldicarb sulfone residues in potato samples from trial 90-138.



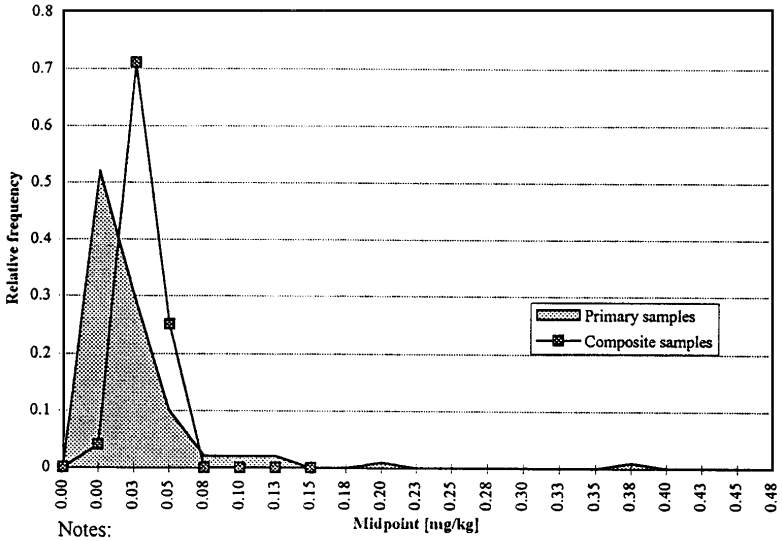
Notes:
Primary samples:
Mean = 0.0773 Standard Deviation = 0.0724723 Median = 0.06
Composite samples:
Mean = 0.077 Standard Deviation = 0.0200693 Median = 0.076

Figure 3. Relative frequency distribution of aldicarb sulfone residues in potato samples from trial 90-142.



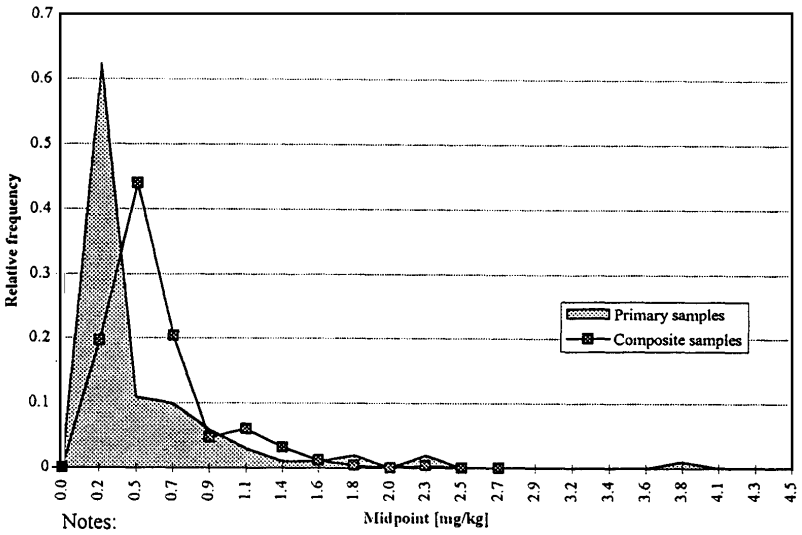
Notes:
Primary samples
Mean = 0.166 Standard Deviation = 0.10245 Median = 0.15
Composite samples
Mean = 0.163 Standard Deviation = 0.02897 Median = 0.160

Figure 4. Relative frequency distribution of aldicarb sulfone residues in potato samples from trial 90-152.



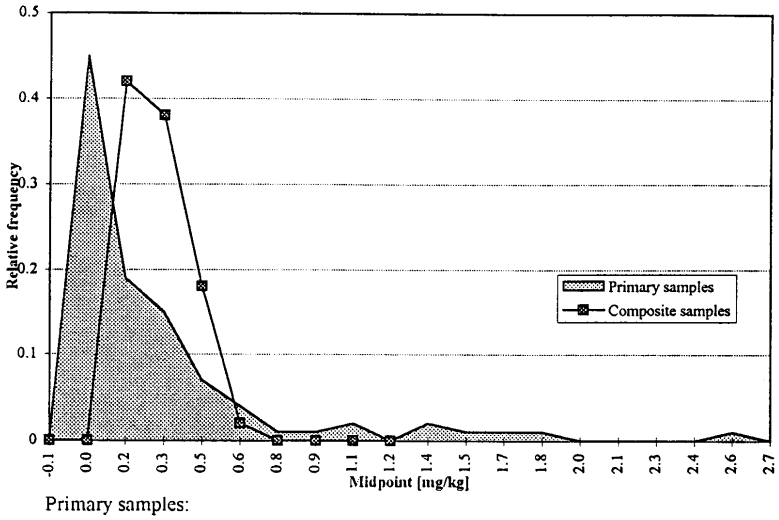
Notes:
Primary samples:
Mean = 0.0306 Standard Deviation = 0.04690 Median = 0.01
Composite samples:
Mean = 0.0311 Standard Deviation = 0.01242 Median = 0.0275

Figure 5. Relative frequency distribution of aldicarb sulfone residues in potato samples from several fields after harvest.



Notes:
Primary samples:
Mean = 0.454 Standard Deviation = 1.0590 Median = 0.12
Composite samples:
Mean = 0.4576 Standard Deviation = 0.31584 Median = 0.371
Residues between 9.1 and 10.2 mg/kg level at
1% relative frequency are not shown

Figure 6. Relative frequency distribution of aldicarb sulfone residues in potato samples after 6 months storage.



Primary samples:
Mean = 0.301 Standard Deviation = 0.4347 Median = 0.125
Composite samples:
Mean = 0.302 Standard Deviation = 0.1108 Median = 0.2788

In the in-furrow irrigation trials the maximum residue (aldicarb plus its metabolites) was 5.30 mg/kg, and eight tubers contained residues above 1 mg/kg.

Following treatments at planting and at plant emergence with overhead irrigation, the maximum residues found were 0.49 and 0.74 mg/kg respectively, and 95% of the tubers contained residues below 0.15 and 0.3 mg/kg respectively. The average residues ranged from 0.03 to 0.3 mg/kg in both primary and composite samples. At 155 and 159 days PHI the maximum residues found in composite samples deriving from two trials were 0.15 mg/kg and 0.13 mg/kg, respectively.

Seventeen field trials were conducted in twelve States of the USA in which aldicarb was applied in-furrow at planting at a target rate of 3.36 kg ai/ha (Tew, 1993a). Treated plots were subdivided into three sections at the time of sampling. The first and last 1.5 m sections of each row were marked and identified as row-end sections. The remaining centre parts of the plots were considered as the uniform application area (row centre). Samples were collected separately from the row centre and the row-end sections. In one trial (92-130) PDA equipment was used, while in the other trials the treatment was carried out by gravity-flow application (GFA). A total of 918 individual potato tubers were analysed. Of these, twelve contained total aldicarb residues above 1 mg/kg, and all of these samples were taken from row-end sections. The maximum residue found was 3.13 mg/kg. The maximum residue found in any row-centre sample was 0.91 mg/kg. The 95th percentile for all samples was 0.40 mg/kg, for row-end samples 0.52 mg/kg, and for row-centre samples 0.34 mg/kg. Since 24-30 potatoes were analysed from each sections, the residues in composite samples were not calculated.

Eight field trials in six States of the USA were designed to determine the variability of aldicarb residues within treated plots and within individual plants (Tew, 1993b). The pesticide was applied at the maximum nominal recommended rate in furrow at planting or with a granular spreader at emergence. One hundred individual potato tubers were analysed from each plot, except in trial 90-129 from which 300 tubers were analysed. Of the total of 1621 samples analysed, only two tubers contained residues above 1 mg/kg, the maximum residue found was 1.3 mg/kg. At PHIs corresponding to GAP the average residues ranged from 0.02 mg/kg to 0.09 mg/kg and the maximum residues in composite samples were between 0.03 and 0.15 mg/kg (Table 3).

Ten potato plants were randomly selected from each plot. All of the tubers found under the selected plants were analysed individually. The coefficient of variation of residues in the tubers from one plant ranged from 0% (three plants) to 350% (one plant), but some of these results are biased by the very low residue levels and few (4-6) tubers per plant . The average residues and their relative standard deviations (coefficients of variation) are shown in Table 4.

Sixteen trials were conducted in four States of the USA to compare the variability and magnitude of the residues in potato tubers grown in the 6 m end-sections of rows, where the tractor comes to a stop before the applicator is lifted, with the mid-row variability and residue levels (Tew, 1994a). Positive-displacement application (PDA) equipment and the conventional gravity-flow applicator (GFA) were used on the side-by-side test plots in each trial. The number of samples analysed from the end-row sections varied from 75 to 101. Thirty tubers were analysed from the mid-row section. A total of 3414 individual potato tubers from the treated plots were analysed. The results are summarized in Table 3.

From the PDA-treated plots a total of 1529 end-row tubers were analysed, of which three contained residues above 1 mg/kg (1.2, 1.2, and 1.12 mg/kg). The highest residues found in mid-row samples were 0.61, 0.51 and 0.32 mg/kg. The average residues in mid-row and end-row samples from treatments complying with GAP ranged from <0.02 to 0.18 mg/kg and from <0.02 to 0.23 mg/kg respectively.

A total of 1525 individual tubers were analysed from end-row sections following GFA treatment. Of these 67 tubers contained residues over 1 mg/kg with a maximum value of 12.8 mg/kg.

The average residues in end-row samples ranged from 0.03 to 0.79 mg/kg.

The Meeting examined the relationship between the average and maximum residues detected in individual potato tubers at sites treated according to the GAP rate and PHI, taking into consideration all middle and end-row ground applications, representing most of the world-wide conditions of use, where the number of potato tubers was above 80 (in most cases it was 100). The selected primary sample populations cover about 95% of residues with 98-99% confidence, which provides a valid data base for estimating the ratio of maximum to average residues. The data are shown graphically in Figure 7. The linear regression of the data resulted in a regression coefficient of 0.904 which indicates an acceptable correlation.

Figure 7. Relation between maximum and average residues from trials according to GAP, including end-row samples.

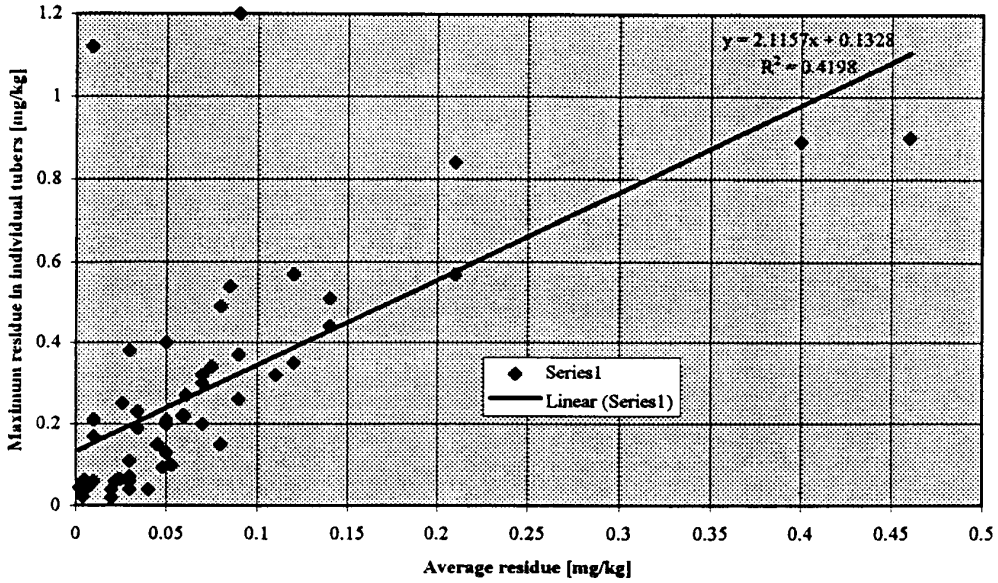


Table 4. Average numbers of tubers found under one plant, and the average residues and their coefficients of variation in single potato tubers under individual plants and within experimental plots.

Trial No.	Within-plant			Within-plot	
	Mean no. of tubers	Mean residue, mg/kg	CV%	Residue, mg/kg	CV%
90-026	6	0.12	24	0.085	88.8
90-095	8	0.1	46	0.089	67.2
90-096	8	0.06	27	0.05	81.9
90-107	7	0.09	37	0.14	51.7
90-129	11	0.24	55	0.295	65.7
90-130	14	0.06	51	0.057	57.4
90-146	8	0.06	109	0.019	31.7
90-191				0.046	40.9
Grand average		0.104	49.9	0.098	60.7
Between-plot CV					87.4

The within-field variation of composite potato samples taken between 118 and 155 days after treatment (Table 5) gave an average CV of 23% and an average variance (square of standard deviations) of 0.0002. The field-to-field variation of the average residues in different regions following GAP applications (Table 6) gave CVs ranging from 70% to 139% with an average of 102%. The average variance for centre-row samples from GAP treatments was 0.0105. The total variance of residues in the samples (V_s) is the sum of the within-field (V_{wf}) and between-field (V_{bf}) variances:

$$V_s = V_{bf} + V_{wf}$$

Since the average within-field variation is only 2-40% of the between-field variation, it effectively does not influence the total variance of the residues and the average residues obtained by the analyses of ≥12 individual potato samples can be used to estimate the maximum residue levels reflecting GAP.

Table 5. Variation of aldicarb residues in composite and primary samples arising soil treatment with aldicarb

Trial No.	Composite samples	Primary samples CV

	Residues expressed as sulfone, mg/kg				SD	Variance	CV	
	Mean	Median	Minimum	Maximum				
90-026	0.083	0.080	0.050	0.150	0.0184	0.00034	0.22	0.89
90-096	0.056	0.051	0.034	0.099	0.0137	0.00019	0.24	0.82
90-137	0.073	0.070	0.044	0.132	0.0166	0.00028	0.23	0.82
90-138	0.077	0.076	0.038	0.148	0.0202	0.00041	0.26	0.94
90-146	0.022	0.022	0.020	0.030	0.0023	0.00001	0.10	0.32
90-151	0.048	0.045	0.021	0.099	0.0151	0.00023	0.31	1.12
90-152	0.031	0.028	0.011	0.063	0.0125	0.00016	0.40	1.53
90-191	0.045	0.043	0.038	0.059	0.0050	0.00003	0.11	0.41
Average	0.054					0.00021	0.23	0.86
Between-field SD: 0.0221			Between-field variance: 0.000488					
Between-field CV: 0.405			Within-field average CV: 0.23					

Table 6. Distribution of average residues in potatoes expressed as aldicarb sulfone measured at experimental field sites.

Type of application	Residue as aldicarb sulfone, mg/kg				Variance	CV%
	Mean	Median	Minimum	Maximum		
Treatment according to US N.W. GAP	0.046	0.04	0.004	0.14	0.00114	73.1
Treatment according to US Florida GAP	0.11	0.075	0.061	0.21	0.003588	54.3
Treatment according to GAP other than USA & South Africa	0.071	0.03	0.005	0.7	0.0103	143.2
Treatment according to GAP other than USA	0.101	0.03	0.005	1.14	0.0299	170.9
All GAP samples	0.099	0.04			average: 0.0105	average: 99.4
Non-GAP applications						
US N.W., PHI 100-135 days	0.148	0.09	0.01	0.9	0.0369	130
US row-end samples, PHI around 150 days	0.10	0.038	0.005	0.69	0.0293	171
US row-end samples, PHI around 100 days	0.176	0.125	0.025	0.5	0.0247	89

FATE OF RESIDUES IN STORAGE AND PROCESSING

In storage

The rate of degradation of aldicarb residues in stored potatoes was determined by analysing 100 individual tubers after 0, 1 and 6 months of storage under simulated commercial storage conditions. No residues of parent aldicarb were detected (<0.02 mg/kg) in any of the samples. The residues were mainly the sulfoxide metabolite with smaller amounts of the sulfone. The mean total residues calculated as sulfone after 0, 1 and 6 months storage were 0.45, 0.44 and 0.30 mg/kg respectively (Hunt, 1991).

In processing

Potatoes were treated at 10 times the normal rate to obtain high residues for a study of the effects of processing. Three replicate potato samples (90 kg each) were processed to chips, flakes and French fries by the following procedures which closely resembled commercial practice.

Chips: washing in tub for 5-10 min; sorting; peeling with Hobart Abrasive Peeler; removing damaged potatoes; cutting to about 1.5 mm slices, frying at 163-177^{o}C for 90 seconds; draining and salting.

Flakes: washing in tub for 5-10 min; sorting; steam peeling for 45 seconds at about 0.55-1 MPa; scrubbing to remove peel;cutting into about 12.5 mm slabs; rinsing with tap water to remove starch; precooking 71-74^{o}C for 20 min.; cooling to 32^{o}C for 20 min; steam cooking at 95-100^{o}C for 45 min.; mashing and mixing with pre-weighed additives; drying into thin sheet; milling to obtain finished potato flakes.

Frozen French fries: washing in tub for 5-10 min; sorting; steam peeling for 45 seconds at about 0.55-1 MPa; scrubbing to remove peel; cutting into about 6 x 6 mm shoestrings with French Fry cutter; blanching at 71-74°C for 10 min.; further blanching at 88-92°C for 3 min.; dipping into 1% dextrose solution for 30 sec.; air drying at 71°C for 18 min.; frying dried strips at 175-190°C for 60 sec.; draining and freezing at about -18°C.

A portion of the fries were further fried at about 175°C for 1.5 min in the laboratory under conditions similar to those used in fast-food restaurants. Potato flakes, chips, fries and wet and dry peel samples were analyzed for aldicarb residues in triplicate. The results are summarized in Table 7.

Additional potatoes were harvested and shipped directly to the laboratory where they were used to determine the effects of baking and microwaving on aldicarb residues. The tubers were cut in half and half of each tuber was frozen. The other half of each potato was then either baked or microwaved and frozen after cooking. All raw tuber halves were individually analyzed. Altogether 200 raw halves, and the 30 pieces from the baked and cooked groups corresponding to the raw halves with the highest residues (60 processed halves altogether) were analyzed.

The mean residues, expressed as aldicarb sulfone, before and after cooking were 0.98 and 0.63 mg/kg for baking, and 1.05 and 0.88 mg/kg for microwaving (Tew 1993c).

Table 7. Effect of processing potatoes on the residue levels of aldicarb.

Sample	Aldicarb residues expressed as aldicarb sulfone, mg/kg			Average change, %
	1st. proc.	2nd proc.	3rd proc.	
Fresh potato	0.57	0.67	0.75	
Flakes	0.77	0.76	0.73	+15
Chips	0.40	0.48	0.55	- 27
Wet peel	0.42	0.33	0.40	- 41
Dry peel	1.36	0.51	1.36	+ 65.3
Frozen fries	0.25	0.29	0.34	- 55.5
Cooked fries	0.34	0.40	0.42	- 41.7

APPRAISAL

Residue aspects of aldicarb were last evaluated in 1994 within the CCPR periodic review programme. A temporary MRL of 0.5 mg/kg was recommended for potato pending the submission of data on supervised trials according to current use patterns. The previous estimate of a maximum residue level for banana was withdrawn owing to a change in the use pattern.

Extensive new information was provided on residues deriving from the currently recommended uses on bananas and potatoes, and on the revised GAP for potatoes in the USA. The Meeting was

informed about ongoing trial programmes for refining use patterns on bananas and expanding the use permit for potatoes within the USA, where the use of compound is authorised at present only in Florida and the Northwest States.

The new GAP for bananas allows a maximum of two applications at a rate of 2 g ai/plant each season with a 180-day PHI. The new US GAP for potatoes specifies positive-displacement application equipment, a single application at a maximum rate of 3.36 kg ai/ha, and the exclusion of in-furrow irrigation. PHIs are 100 days for Florida and 150 days for Northwest States.

The trials were with granular formulations of aldicarb. The samples were mainly analysed by HPLC methods which determined aldicarb, its sulfoxide and its sulfone individually. In some cases the residues were oxidized to, and determined as, the sulfone by GLC. The typical limit of determination was about 0.01-0.02 mg/kg for each residue component. The residues are reported in the monograph and appraisal as the total carbamate residue expressed as aldicarb sulfone, which can be converted to the parent aldicarb by multiplying by 0.856.

In trials with bananas, aldicarb was applied to the soil at the recommended maximum rate of 2 g ai/plant, corresponding to 3.6-4 kg ai/ha. Samples were taken from 3 to >360 days after the last application. Aldicarb residues were determined in composite samples consisting of 7-12 fingers or in individual fingers. The average residue ranges found in whole bananas were 0.23-0.5 mg/kg at 45 and 75 days, 0.02-0.4 mg/kg at 87 and 124 days, and 0.04 mg/kg at 150 days.

The residues were determined in the peel and pulp separately and calculated for the whole fruit in 14 trials. There was little difference between the residues in whole fruit and pulp, the average ratio of whole fruit to pulp residues being 1.19.

The residues were determined only in the pulp in most of the samples, at PHIs much shorter than the recommended 180 days. In 6 samples the residues in the pulp were between 0.02 and 0.04 mg/kg at PHIs of about 150 days and longer. Of these 6 trials, one or two applications (GAP) were made at five occasions resulting in maximum, median and mean residues of 0.04 mg/kg, 0.03 mg/kg and 0.029 mg/kg, respectively.

The main residue was aldicarb sulfoxide. Aldicarb was not detected in any of the samples and the sulfone in only a few. The sulfoxide/sulfone ratio ranged from 3 to 23 in those samples where both residues were present in detectable concentrations.

Taking into consideration the slow decline of residues, the factor for the conversion of aldicarb sulfone to the parent compound according to the residue definition, and the factor of 1.2 for the ratio of the residues in the whole fruit to those in the pulp, the Meeting concluded that a maximum residue level of 0.05 mg/kg expressed as aldicarb would be likely to cover residues from applications in accord with GAP. However, since residues were reported in the whole commodity in only a single trial which complied with GAP, the Meeting considered the data from GAP applications insufficient to recommend an MRL.

A number of trials on potatoes were reported from 14 countries. The residues were mainly aldicarb sulfoxide.

In supervised trials representing national GAP except in the USA and South Africa, the total residue expressed as aldicarb sulfone ranged from 0.005 mg/kg to 0.7 mg/kg with a median of 0.03 mg/kg. Of the total of 94 trials the highest residues measured in rank order were 0.21, 0.23, 0.25, 0.26, 0.3, 0.35, 0.4, 0.4, 0.46 and 0.7 mg/kg. The 95th and 98th percentiles were 0.25 and 0.4 mg/kg

respectively.

Twenty-nine trials were reported from South Africa in a summarized form without sufficient details of trial conditions or analytical methods. Aldicarb was applied once or twice at rates from 3.75 to 7.5 kg ai/ha, and PHIs were from 38 to 125 days. Eight of the 29 trials were in accordance with GAP for food and feed potatoes (1 application with 2.55-5.25 kg ai/ha and 120 days PHI). Residues in composite samples from these trials ranged from 0.01 to 1.12 mg/kg. Although the residue levels fit well into the distribution of residues in Spanish trials (0.02-0.7 mg/kg), taking into account the higher rate (5.25 kg ai/ha compared to 1.5 kg ai/ha in the Spanish trials), the Meeting was not able to evaluate the trials because essential details were not reported.

In most of the reported US trials the nominal application rate was the maximum recommended 3.36 kg ai/ha. Treatment was either with positive-displacement application (PDA) equipment according to the new GAP requirement or with the traditional gravity-flow applicator (GFA).

Residues were measured in a very large number of individual potato tubers to determine the effects of the mode of application, method of irrigation, and climatic conditions on the magnitude and distribution of the residues. The residues in individual potatoes extended over wide ranges and their relative frequency distributions were not normal, in accord with previous findings with other crop-pesticide combinations. In order to estimate maximum residue levels, 100 composite random samples of 12 tubers each were drawn from the selected primary sample populations with replacement, according to a computer programme.

The relative frequency distributions of composite samples taken from a single site were close to normal. The average residues found in composite samples and the average of the residues in the primary samples (field site residues) were very similar.

In the case of trials according to US GAP the between-field variance (square of the standard deviation) of the maximum residues in the composite samples (V_{bf} = 0.000488) was about 2.4 times the average within-field variance (V_{wf} = 0.00021). The between-field coefficient of variation of the residues was found to be 40%, while the combined between- and within-field CV was 48%. When all US trials complying with GAP were taken into consideration (either with field site residues or with maximum composite residues) the V_{bf}/V_{wf} ratio was >10. Consequently the within-field variation has little or no effect on the overall coefficient of variation of residues, and the calculated average residues obtained from the analysis of large numbers of primary samples (79-100 from an experimental site) or the average of smaller numbers of primary samples (>12 equal to one composite sample) can be used to estimate maximum residue levels.

Following single applications at planting or at emergence with commercial ground equipment and at PHIs above 139 days, in-furrow irrigation in 6 trials resulted in higher residues (average 0.18 mg/kg; range 0.025-0.316 mg/kg) than overhead irrigation (average 0.048 mg/kg; range 0.01-0.077 mg/kg) in four trials. The between-field coefficients of variation of the average residues were very similar: 66% and 69% respectively.

Eight field trials were conducted in 6 States of the USA to determine the variability of residues within fields and within plants. One hundred potato tubers and ten potato plants were selected from each field. The average within-plant and within-field coefficients of variation were 49.9% and 60.7% respectively. The between-field coefficient of variation was 87.4%. At trial sites where the PHIs complied with GAP, the field site residues ranged from 0.02 mg/kg to 0.09 mg/kg and the maximum residues in composite samples were from 0.03 to 0.15 mg/kg.

Seventeen field trials were conducted in twelve States of the USA to determine the distribution of residues in centre-row and end-row areas. The average residues in the centre and end parts of the rows were 0.094 and 0.12 mg/kg respectively. Twelve of a total of 918 tubers contained residues above 1 mg/kg with a maximum of 3.13 mg/kg. All of these tubers were taken from the end sections of the rows. The 95th percentiles for the centre and end parts of rows were 0.34 and 0.52 mg/kg respectively.

Positive-displacement application (PDA) equipment and the conventional gravity-flow applicator (GFA) were used in sixteen trials to compare the variability and magnitude of residues in potato tubers grown in the 6 m end-sections with rows to the residues in mid-row tubers. Following PDA treatment according to the field site residues in mid-row and end-row samples ranged from <0.02 to 0.18 and <0.02 to 0.23 mg/kg respectively. The averages of the field site residues found in mid-row and end-row sections of the experimental sites (0.065 and 0.052 mg/kg) did not differ significantly. The average residue in end-row samples from GFA treatments (0.234 mg/kg) was significantly higher than that found following PDA treatments, while the mid-row samples were not analyzed. The between-field variation (CV%) of the average residues from mid-row and end-row sections following PDA and GFA treatments did not differ significantly and were 78.2%, 115% and 109% respectively. The highest 10 residues found in individual potato tubers in trials according to US GAP including end-of-row sections were 1.2, 1.2, 1.12, 0.61, 0.51, 0.32, 0.31, 0.29, 0.26 and 0.25 mg/kg.

To estimate a maximum residue level the Meeting took into consideration all of the 87 residues measured in composite field samples and the averages of residues measured in individual potatoes from trials which complied with relevant national GAP, but excluded the summarized data from the South African trials and the results of the trials from The Netherlands (where GAP is limited to seed and starch potatoes).

To estimate the STMR level only those samples were considered which were taken within ±30% of the GAP PHI and which had been treated by applications at rates within the range GAP to GAP +30%. The residue values considered in reverse rank order were 0.7, 0.43, 0.4, 0.4, 0.35, 0.3, 0.3, 0.25, 0.25, 0.24, 0.23, 0.23, 0.1-0.18 (6), 0.09, 0.09, 0.053-0.08 (7), 0.03-0.048 (5) and <0.03 (5) mg/kg. The same residues were considered for the estimation of a maximum residue level: these gave a 98th percentile value between 0.4 and 0.43 mg/kg.

On the basis of the results the Meeting confirmed its previously estimated maximum residue level (no longer temporary), of 0.5 mg/kg for potato, and estimated an STMR level of 0.09 mg/kg expressed as aldicarb sulfone (0.077 mg/kg expressed as aldicarb).

The Meeting examined the relationship between the average and maximum residues found in individual potato tubers at sites treated according to the US GAP rate and PHI, including both PDA and GFA treatments to reflect the current world-wide uses. For acute risk assessment the residues found at the end-of- row sections were also included in the data base. Taking into consideration the populations consisting of >80 residues determined in individual potatoes which provided an estimate for 95% of the residues with 98-99% confidence, the linear regression of the data resulted in a regression equation of

$$R_{max} = 13.25R_{av} - 0.46 \qquad \text{(eq. 1)}$$

with a correlation coefficient of 0.904, which indicates an acceptable correlation. Consequently, the maximum residues in individual tubers in a treated field may be expected on average to be in the range of 13-14 times the average residue found at the site. It should be noted that higher values may occasionally occur.

When only the residues arising from PDA treatment according to current US GAP were taken into account the linear regression equation was

$$R_{max} = 5.5R_{av} + 0.06 \quad \text{(eq. 2)}$$

with a correlation coefficient of 0.79.

The Meeting noted that the data points available for estimating maximum residues arising from PDA treatments were limited and rather scattered, and the correlation between the average and maximum residues was poor. The results were of some interest however, and underline the importance of considering the effect of different use patterns individually first, and pooling only residues from similar populations. The results may otherwise be distorted and unrealistic. The Meeting therefore decided to present both estimates.

On the basis of equation 1 and the estimated maximum residue level (0.5 mg/kg) the estimated maximum residue in individual potatoes is 6.2 mg aldicarb/kg. When the 98th percentile residue value (between 0.4 and 0.43 mg/kg as aldicarb sulfone or 0.415 x 0.856 mg/kg as aldicarb) which was obtained from all field trials which were in accordance with GAP is used, the calculated maximum residue in individual potato tubers is 4.2 mg/kg.

However, if only the residues arising from PDA treatments according to new US GAP are taken into account, with a maximum field site residue of 0.2 mg/kg, the maximum residue in individual potatoes (see equation 2) would be 0.94 mg aldicarb/kg.

Under normal commercial storage conditions the mean total residues calculated as sulfone after 0, 1 and 6 months storage were 0.45, 0.44 and 0.30 mg/kg respectively.

Three replicate potato samples were processed by treatments closely resembling commercial procedures. A portion of deep-frozen French fries were further cooked in hot oil for 1.5 min under conditions similar to those used in fast-food restaurants. The average decrease of the total carbamate residue level was 3.2% in potato flakes, 27% in chips, 55.5% in processed fries, 41.7% in cooked fries and 41% in wet peel. After drying, the residue level in the peel increased to 65.3% of that in the fresh potato owing to the loss of moisture.

The effects of baking and microwave cooking were studied on additional potato samples. The residues decreased by 35.7% and 16.1% respectively.

RECOMMENDATIONS

On the basis of the data on residues resulting from supervised trials the Meeting estimated the maximum residue level shown below for potato, which is recommended for use as an MRL.

Definition of the residue for compliance with MRLs and for estimation of dietary intake:
sum of aldicarb, its sulfoxide and sulfone, expressed as aldicarb.

Commodity		Recommended limits, mg/kg			PHI on which based, days
		MRL		STMR or STMR-P	
CCN	Name	New	Previous		
VR 0589	Potato	0.5	0.5 T	0.077	56-150
	Potato chips			0.056	

	Potato fries			0.045	
	Potato (microwaved)			0.065	
	Potato (baked)			0.050	56-150

FURTHER WORK OR INFORMATION

Desirable

1. Results of supervised trials according to maximum Spanish and South African GAP on potatoes.

2. Residue data on whole bananas and banana pulp reflecting current GAP.

3. Data on the effect of boiling (cooking) on aldicarb residues in potatoes.

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BIFENTHRIN (178)

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 Meeting on the use of bifenthrin as a grain protectant on stored grain but no new recommendations were made for cereals because a number of points needed to be clarified. The following information was listed as desirable.

Efficiency of extraction by acetone of aged bifenthrin residues.
National MRLs for bifenthrin relating to uses on stored grains.
Fate during the commercial milling of wheat.
Fate during the baking of bread.
Fate during commercial malting of barley.

Information on milling and baking studies with bifenthrin-treated wheat was provided.

METHODS OF RESIDUE ANALYSIS

Roland (1993) described a residue analytical method for bifenthrin and malathion in cereal grains. The method relied on hexane/acetone extraction followed by solvent evaporation and capillary GLC analysis. Electron-capture detection was used for bifenthrin and flame-photometric or thermionic detection for malathion. No specific information was available on the efficiency of extraction of aged bifenthrin residues from grain by hexane/acetone, but the fact that the bifenthrin residue levels on wheat at day 1 were unchanged by week 12 in the storage trials suggests that the solvent adequately extracted aged residues.

Measured recoveries of bifenthrin were in the range 94-101% from wheat fortified at 0.01, 0.25 and 0.50 mg/kg (Roland *et al.*, 1995b) and 91-100% from wheat fortified at 0.1, 0.2, 0.3 and 0.4 mg/kg (Roland *et al.*, 1995a). The data suggest an LOD of 0.01 mg/kg for bifenthrin on wheat.

Roland (1995) described a residue analytical method for bifenthrin and malathion in flour. The method relied on acetone extraction followed by solvent evaporation and clean-up by Florisil column chromatography. Bifenthrin and malathion were determined by capillary GLC, using an ECD for bifenthrin and an FPD for malathion. Bifenthrin recoveries with this method were 102, 103% from white flour at 0.05 mg/kg; 101, 102% from bran at 1.0 mg/kg; and 86, 95% from wholemeal bread at 0.2 mg/kg. The LOD was stated to be 0.01 mg/kg, but no recovery data were available below 0.05 mg/kg. No data were available for recoveries from white bread.

FATE OF RESIDUES IN STORAGE AND PROCESSING

In processing

Residues and application rates have generally been rounded to 2 significant figures or, for residues near the LOD, to 1 significant figure. Residues were not detected in control samples except for an occasional value at the LOD.

Wheat grain was treated with two formulations (EC and UL) of a bifenthrin + malathion mixture at a nominal application rate of 0.30 g ai/t for bifenthrin and 6.0 g ai/t for malathion. The grain was treated in 25 kg batches (2 batches treated and combined for each formulation) by applying the pesticides with a hand pump sprayer to the grain tumbling in a small concrete mixer with clean shiny, bare steel interior surfaces.

The treated grain and controls were delivered to Weston Research Laboratories for storage and milling (Creighton and Charlton, 1995). The grain was kept in open-topped bins at a room temperature of 20°C. The grain moisture was 13.2% throughout the duration of the storage. Samples (1 or 4 kg) were withdrawn periodically for cleaning and milling. The 4 kg samples were milled when the flour was destined for bread production.

The grain was cleaned before milling with a Laboratory Carter Day Dockage Tester, which simulates commercial cleaning. Screenings removed from the grain amounted to 0.7-1.5%. The grain was conditioned to 15.7% moisture and milled on a Buhler Mill to produce flour and bran. The white flour extraction rate was 81-82%. Wholemeal was made by recombining bran and white flour. Bread was produced in 400 g loaves by the Chorleywood Breadmaking Process. Uncleaned grain, flour, bran and bread were sent to Gembloux for residue analysis (Roland *et al*., 1995b). The residues found in wheat are shown in Table 1, and those in flour, bran and bread in Tables 2-5.

In another storage trial wheat was treated with UL or EC formulations of bifenthrin + malathion and stored at 20°C and 25°C at a relative humidity of 65% for 6 months (Roland *et al.*, 1995a). Samples were withdrawn for analysis 0, 90 and 180 days after treatment. The residues are shown in Table 1.

Concentration factors for residues of bifenthrin in milling and baking fractions from wheat are given in Table 6. The factors are calculated from the bifenthrin residues in Tables 2 and 3. Approximately 16% of the residues were lost in producing wholemeal flour from uncleaned wheat. The bifenthrin level in white flour was about 30%, and the level in bran 3.5 times that in uncleaned wheat.

The results of these trials suggest that about 70% of the bifenthrin disappears on baking wholemeal or white bread.

Table 1. Residues of bifenthrin and malathion resulting from supervised trials on stored wheat after post-harvest applications (Roland *et al*., 1995a,b).

Country, year	Grain weight, temp	Form	Treatment g ai/t	Storage time	Residues, mg/kg		Ref.
					Bifenthrin	Malathion	
UK, 1995	50 kg, 20°C	UL	b 0.3 + m 6.0	1 day 4 weeks 8 weeks 12 weeks	0.24 0.24 0.23 0.22	4.1 2.6 2.4 1.4	CRP/95/1363
UK, 1995	50 kg, 20°C	EC	b 0.3 + m 6.0	1 day 4 weeks 8 weeks 12 weeks	0.25 0.24 0.25 0.24	4.0 2.8 2.3 2.0	CRP/95/1363
France, 1995	100 kg, 20°C	UL	b 0.3 + m 6.0	day 0 day 90 day 180	0.26 0.25 0.21	4.5 2.6 0.68	CRP/95/1362
France, 1995	100 kg, 25°C	UL	b 0.3 + m 6.0	day 0 day 90 day 180	0.28 0.24 0.22	4.5 2.4 0.98	CRP/95/1362
France, 1995	100 kg, 20°C	EC	b 0.3 + m 6.0	day 0 day 90 day 180	0.31 0.27 0.25	5.0 2.2 0.92	CRP/95/1362
France, 1995	100 kg, 25°C	EC	b 0.3 + m 6.0	day 0 day 90 day 180	0.32 0.28 0.24	5.3 2.4 0.90	CRP/95/1362

b: bifenthrin m: malathion

Table 2. Bifenthrin residues in wheat and processed commodities from wheat treated post-harvest with a UL formulation at bifenthrin 0.3 g ai/t and malathion 6 g ai/t and stored for 12 weeks at 20°C. Samples were withdrawn on day 1 and weeks 4, 8 and 12 for milling (Roland *et al*., 1995b).

Sample	Bifenthrin residues, mg/kg							
	Day 1		Week 4		Week 8		Week 12	
	fresh wt	dry wt	fresh wt	dry wt	fresh wt	dry wt	fresh wt	dry wt
Wheat	0.24	0.27	0.24	0.27	0.23	0.26	0.22	0.24
Wholemeal flour	0.20	0.23	0.19	0.22	0.21	0.24	0.17	0.19
Wholemeal bread	0.05	0.08			0.04	0.07		
White flour	0.07	0.08			0.06	0.07		
White bread	0.02	0.03			0.01	0.02		
Bran	0.75	0.85			0.88	1.0		

Table 3. Bifenthrin residues in wheat and processed commodities from wheat treated post-harvest with an EC formulation at bifenthrin 0.3 g ai/t and malathion 6 g ai/t and stored for 12 weeks at 20°C. Samples were withdrawn on day 1 and weeks 4, 8 and 12 for milling (Roland *et al*., 1995b).

Sample	Bifenthrin residues, mg/kg							
	Day 1		Week 4		Week 8		Week 12	
	fresh wt	dry wt	fresh wt	dry wt	fresh wt	dry wt	fresh wt	dry wt
Wheat	0.25	0.28	0.24	0.28	0.25	0.28	0.24	0.27
Wholemeal flour	0.20	0.23	0.18	0.20	0.22	0.25	0.22	0.25
Wholemeal bread	0.03	0.05			0.05	0.08		
White flour	0.09	0.10			0.07	0.08		
White bread	0.02	0.03			0.02	0.03		
Bran	0.83	0.95			0.92	1.1		

Table 4. Malathion residues in wheat and processed commodities from wheat treated post-harvest with a UL formulation at bifenthrin 0.3 g ai/t and malathion 6 g ai/t and stored for 12 weeks at 20°C. Samples were withdrawn on day 1 and weeks 4, 8 and 12 for milling (Roland *et al*., 1995b).

Sample	Malathion residues, mg/kg							
	Day 1		Week 4		Week 8		Week 12	
	fresh wt	dry wt	fresh wt	dry wt	fresh wt	dry wt	fresh wt	dry wt
Wheat	4.1	4.7	2.6	2.9	2.4	2.7	1.4	1.6
Wholemeal flour	2.0	2.3	1.4	1.7	1.5	1.8	1.5	1.7
Wholemeal bread	0.53	0.88			0.25	0.42		
White flour	0.74	0.85			0.55	0.64		
White bread	0.11	0.18			0.08	0.12		
Bran	3.5	4.0			6.6	7.5		

Table 5. Malathion residues in wheat and processed commodities from wheat treated post-harvest with an EC formulation at bifenthrin 0.3 g ai/t and malathion 6 g ai/t and stored for 12 weeks at 20°C. Samples were withdrawn on day 1 and weeks 4, 8 and 12 for milling (Roland *et al*., 1995b).

Sample	Malathion residues, mg/kg							
	Day 1		Week 4		Week 8		Week 12	
	fresh wt	dry wt	fresh wt	dry wt	fresh wt	dry wt	fresh wt	dry wt
Wheat	4.0	4.6	2.8	3.2	2.3	2.7	2.0	2.2
Wholemeal flour	2.6	3.0	1.6	1.8	1.5	1.8	1.8	2.0
Wholemeal bread	0.30	0.44			0.27	0.45		
White flour	0.91	1.0			0.48	0.56		
White bread	0.16	0.26			0.07	0.11		
Bran	4.4	5.1			4.8	5.5		

Table 6. Mean processing factors for bifenthrin residues on wheat, wholemeal, flour and bread calculated from data in Tables 2 and 3 (Roland *et al*., 1995b).

Process	Bifenthrin residues in second commodity ÷ bifenthrin residues in first commodity, mean (range)	
	Both commodities fresh weight basis	Both commodities dry weight basis
Uncleaned wheat → wholemeal flour	0.83 (0.75-0.92)	0.84 (0.71-0.93)
Uncleaned wheat → white flour	0.30 (0.26-0.36)	0.30 (0.27-0.36)
Uncleaned wheat → bran	3.5 (3.1-3.8)	3.5 (3.1-3.8)
Wholemeal flour → wholemeal bread	0.20 (0.15-0.25)	0.30 (0.22-0.35)
White flour → white bread	0.24 (0.17-0.29)	0.33 (0.29-0.38)

APPRAISAL

Bifenthrin was first evaluated at the 1992 Meeting and MRLs of 0.05* mg/kg were recommended for barley, maize and wheat to cover field applications. The 1995 JMPR reviewed information about the use of bifenthrin as a grain protectant but made no recommendations pending the receipt of information on the following points.

Efficiency of extraction by acetone of aged bifenthrin residues.
National MRLs for bifenthrin relating to uses on stored grains.
Fate during the commercial milling of wheat.
Fate during the baking of bread.
Fate during commercial malting of barley.

Information on milling and baking studies with bifenthrin-treated wheat was made available to the Meeting.

A method for the determination of residues in cereal grains relied on hexane/acetone extraction followed by solvent evaporation and capillary GLC analysis with EC detection. Recoveries were good and the LOD for bifenthrin on wheat was 0.01 mg/kg.

No specific information was available on the efficiency of extraction of aged bifenthrin residues from grain by hexane/acetone, but the fact that the bifenthrin residue levels on wheat at day 1 were unchanged by week 12 in the storage trials suggests that the solvent adequately extracted aged residues.

A residue method for bifenthrin in flour, bran and bread involved acetone extraction followed by solvent evaporation, clean-up by Florisil column chromatography and capillary GLC with EC detection. Recoveries of bifenthrin were good from white flour, bran and wholemeal bread at 0.05, 1.0 and 0.2 mg/kg respectively. No data were available for recoveries from white bread.

In two grain storage trials wheat was treated with EC or UL formulations of a bifenthrin + malathion mixture at rates of 0.3 and 6.0 g ai/t for bifenthrin and malathion respectively, and then stored for 12 weeks or 180 days. The grain was sampled at intervals for analysis.

The results were consistent with those of the trials of bifenthrin used as a grain protectant evaluated by the 1995 Meeting. Bifenthrin residues are stable on stored grain at 20°C and 25°C and the levels of bifenthrin on the grain at the beginning and end of the storage will be essentially the same.

On the basis of the approved use of bifenthrin as a grain protectant on stored grain in Belgium at 0.3 g ai/t and its stability on wheat during storage the Meeting estimated a maximum residue level of 0.5 mg/kg for bifenthrin in wheat.

Data from 8 trials of bifenthrin on stored wheat in Belgium, France and the UK were evaluated by the 1995 JMPR. The highest bifenthrin residues recorded in each trial were 0.22, 0.19, 0.24 and 0.25 mg/kg in Belgium, 0.26 and 0.24 mg/kg in France and 0.28 mg/kg in the UK. The highest bifenthrin residues in each of the 6 trials evaluated by the present Meeting were 0.24 and 0.25 mg/kg in the UK and 0.26, 0.28, 0.31 and 0.32 mg/kg in France.

In summary the highest bifenthrin residues in each of the 14 grain protectant trials according to GAP in rank order (median underlined) were 0.19, 0.22, 0.24, 0.24, 0.24, 0.25, <u>0.25</u>, <u>0.26</u>, 0.26, 0.27, 0.28, 0.28, 0.31 and 0.32 mg/kg. The Meeting estimated an STMR of 0.255 mg/kg for bifenthrin on wheat.

In the milling studies grain was cleaned before milling with a Laboratory Carter Day Dockage Tester, which simulates commercial cleaning. Bifenthrin-treated wheat was taken for milling and baking on the first day and 8 weeks after treatment.

Approximately 16% of the bifenthrin residues were lost in producing wholemeal flour from uncleaned wheat. The bifenthrin level in white flour was about 30% (26-36%) and the level in bran about 3.5 (3.1-3.8) times the level in the original wheat.

Residues of bifenthrin in <u>white flour</u> in the 4 milling trials (2 milling trials from each of 2 storage trials) were 0.06, 0.07, 0.07 and 0.09 mg/kg. Taking into account the possibility that bifenthrin residue levels on wheat could be higher than the levels on the wheat in these milling trials (0.23-0.25 mg/kg), the Meeting estimated a maximum residue level of 0.2 mg/kg for bifenthrin in flour.

The bifenthrin residues in the flour were 0.26, 0.28, 0.29 and 0.36 (mean 0.30) times those in the wheat. The Meeting therefore estimated an STMR-P of 0.076 mg/kg for bifenthrin in flour (0.30×0.255).

The bifenthrin residues in <u>bran</u> in the 4 milling trials were 0.75, 0.83, 0.88 and 0.92 mg/kg. Since the bifenthrin residue levels on wheat might be higher than those found in the trials (0.23-0.25 mg/kg) the Meeting estimated a maximum residue level of 2 mg/kg for bifenthrin in bran.

The concentration factors for bifenthrin residues in the processing of wheat to bran in the 4 milling trials were 3.1, 3.3, 3.7 and 3.8 (mean 3.5). The Meeting estimated an STMR-P of 0.89 mg/kg for bifenthrin in bran (3.5×0.255).

<u>Wholemeal flour</u> produced from bifenthrin-treated wheat on 4 occasions in each of the 2 storage trials contained bifenthrin residues of 0.17, 0.18, 0.19, 0.20, <u>0.20</u>, 0.21, 0.22 and 0.22 mg/kg. These residues were 0.75, 0.77, 0.79, 0.80, 0.83, 0.88, 0.91 and 0.92 (mean 0.83) times those in the uncleaned wheat. The Meeting estimated a maximum residue level of 0.5 mg/kg and an STMR-P of 0.21 mg/kg (0.83×0.255) for bifenthrin in wholemeal.

Wholemeal bread and white bread were produced from the wholemeal and white flours generated in the milling studies. Residues in the bread and flour were reported on both a fresh and a dry weight basis. The results suggest that about 70% of the bifenthrin disappears on baking wholemeal or white bread. This is not consistent with the behaviour of other pyrethroids, which are largely retained

through the baking process.

The Meeting was reluctant to draw a firm conclusion on the fate of bifenthrin during baking until some aspects of the analytical method had been clarified. Validation of analytical recoveries from bread at the bifenthrin residue levels which occur in practice and at the LOD is needed, as is investigation into the possibility that bifenthrin residues are bound in the bread and not extractable by the current method.

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 MRLs and for estimation of dietary intake: bifenthrin.

The residue is fat-soluble.

Commodity		Recommended MRL, mg/kg		STMR, mg/kg	STMR-P, mg/kg
CCN	Name	New	Previous		
GC 0654	Wheat	0.5 Po	0.05*	0.255	
CM 0654	Wheat bran, unprocessed	2 PoP			0.89
CF 1211	Wheat flour	0.2 PoP			0.076
CF 1212	Wheat wholemeal	0.5 PoP			0.21

Po: the recommended MRL accommodates post-harvest treatment
PoP: the recommended MRL accommodates post-harvest treatment of the primary food commodity

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. 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).

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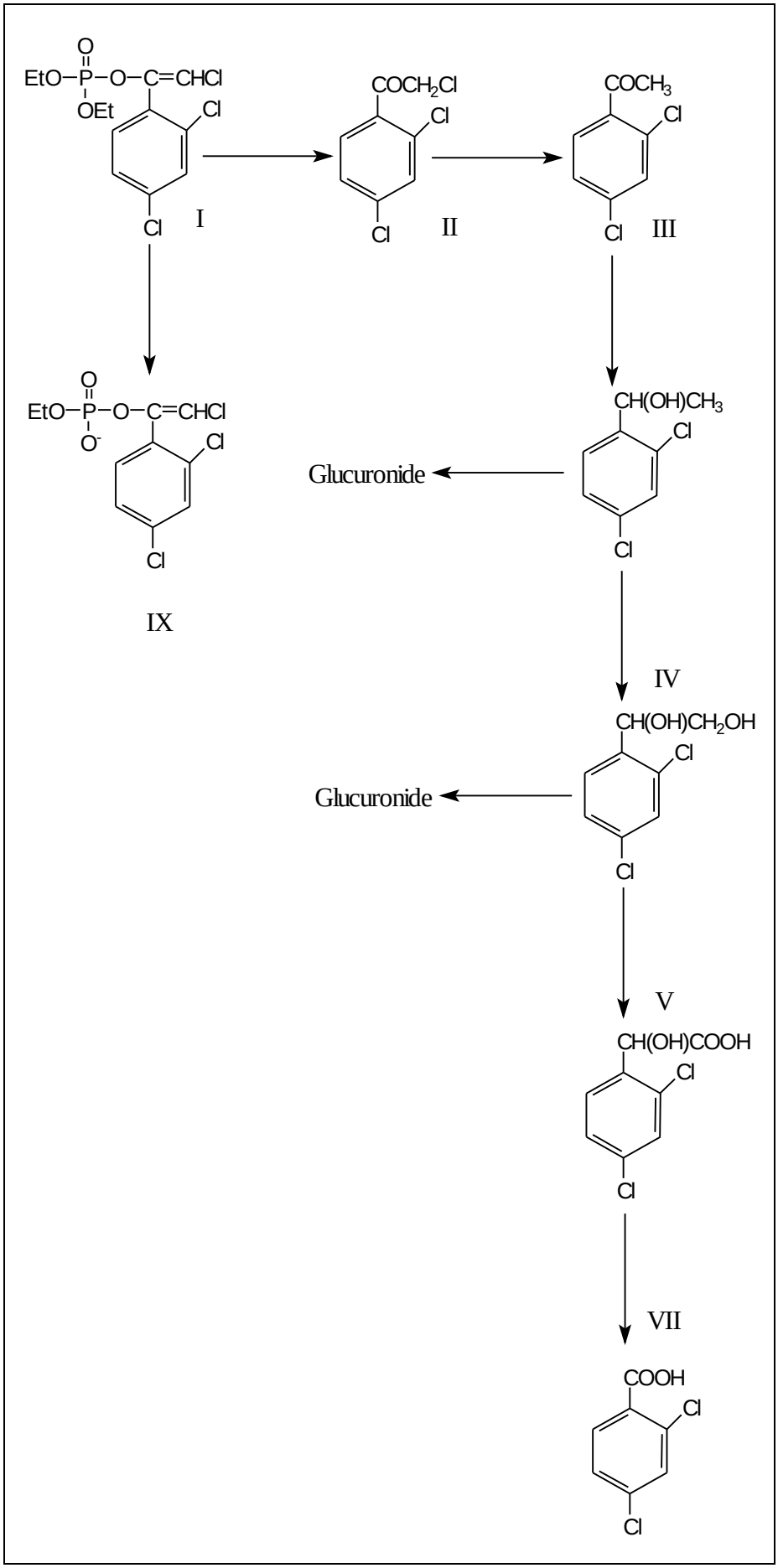
Roland, L., Marien, L. and Sprangers, O. 1995b. Residues of bifenthrin and malathion in grains of wheat - flour, bran and bread. (Wheat treated with Prostore grain protectant - 1995). Report CRP/95/1363. Study BEAGX 5/1995/1148. B.E.A.Gx, Centre de Recherches de Phytopharmacie, Belgium. Unpublished.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Cl
Cl
I
COCH2Cl
Cl
Cl
II
COCH3
Cl
Cl
III
EtO–P(=O)(O-)–O–C=CHCl
Cl
Cl
IX
CH(OH)CH3
Cl
Glucuronide
Cl
IV
CH(OH)CH2OH
Cl
Glucuronide
Cl
V
CH(OH)COOH
Cl
Cl
VII
COOH
Cl
Cl

Figure 2. Metabolism of chlorfenvinphos in plants following foliar treatment.

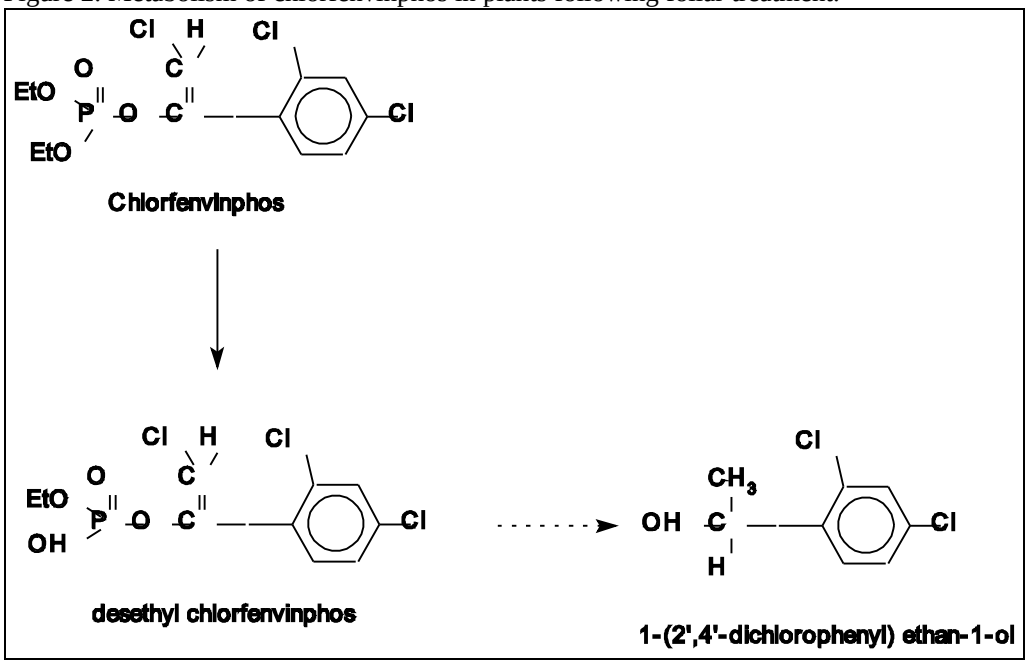
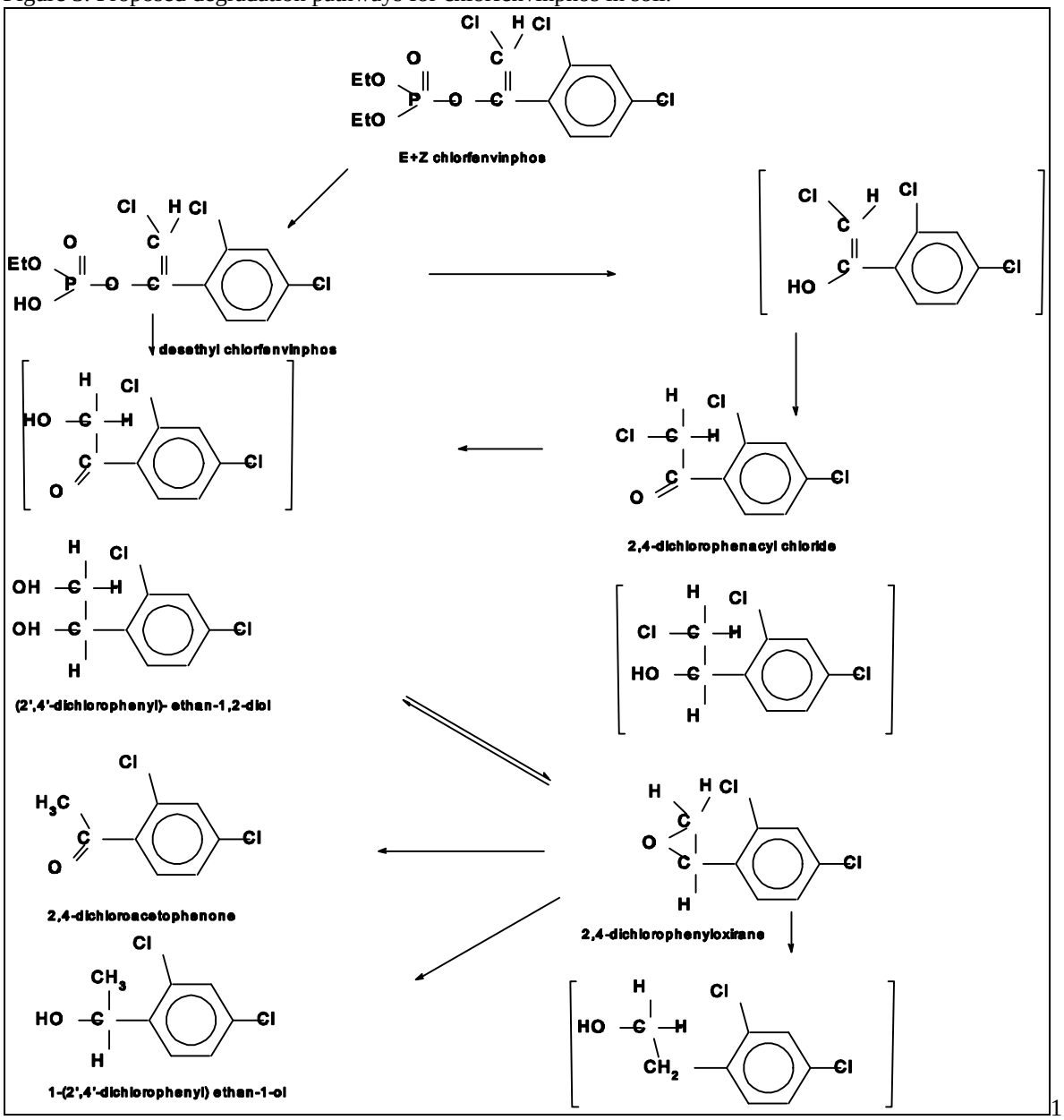


Figure 3. Proposed degradation pathways for chlorfenvinphos in soil.



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CHLORFENVINPHOS (014)

EXPLANATION

Chlorfenvinphos was evaluated for residues by the JMPR in 1971 and 1984 and maximum residue levels for a number of commodities were estimated.

Chlorfenvinphos was proposed for re-evaluation by the Working Group on Priorities at the 1989 CCPR (ALINORM 89/24A, para 298 and Appendix V). The review was scheduled for 1994 at the 1990 CCPR (ALINORM 91/24, Appendix V Part II) and confirmed by the 1991 CCPR on the understanding that new data would be available (ALINORM 91/24A, para 316 and Appendix VI, Annex I).

Information on current GAP and data on residues were requested from governments by CL 1991/15-PR.

The manufacturer informed FAO that data on residues would not be available in time for the 1994 JMPR and the review was therefore delayed until the 1996 Meeting.

The Meeting received data on residues and information on GAP from the manufacturer, and additional information was provided by Australia, Germany, The Netherlands, Poland and the UK.

IDENTITY

ISO common name: chlorfenvinphos

Chemical name

IUPAC: 2-chloro-1-(2,4-dichlorophenyl)vinyl diethyl phosphate

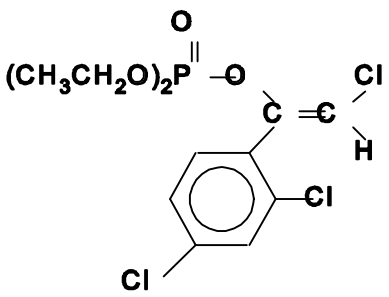
CA: 2-chloro-1-(2,4-dichlorophenyl)ethenyl diethyl phosphate

CAS registry no: 470-90-6 (formerly 2701-86-2) (*Z*)- + (*E*)- isomers;
18708-87-7 (*Z*)- isomer;
18708-86-6 (*E*)- isomer

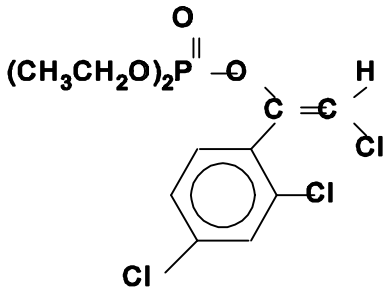
CIPAC No: 88

Synonyms: “Birlane”, “Supona”, CL 58,085, SD 7859, GC 4072

Structural formula:



Z-isomer



E-isomer

Ratio	8.4	1

Molecular formula: $C_{12}H_{14}Cl_3O_4P$

Molecular weight: 359.6

Physical and chemical properties

Pure active ingredient

No information was submitted.

Technical material

Purity:

Typical specification based on the analysis of 12 manufacturing batches in 1994 was 90-91.4% (total (*E*)- + (*Z*)-).

The purity of the technical material with which the physical and chemical properties listed below were determined was 93.1% (83.3% (*Z*)- isomer, 9.8% (*E*)- isomer) or 94.5% (84.2% (*Z*)- isomer, 10.3% (*E*)- isomer).

Colour:	amber
Physical state:	liquid at 25°C
Odour:	weak inherent smell
Melting point:	below -30°C
Boiling Point:	above 280°C
Relative Density	1.351
Surface tension of aqueous solutions	
	90% sat 51.8 mN/m
	80% sat 53.0 mN/m
Vapour Pressure	
at 25°C:	(*Z*)- isomer 0.37 x 10^{-3} Pa
	(*E*)- isomer 5.4 x 10^{-5} Pa

Flash Point: No flash point was observed up to a temperature of 285°C.

Auto-flammability: 542.6°C ± 1.1°C (mean of 5 assays)

Hydrolysis: Half-life in hours for the (*Z*)- isomer 6300 (pH 4), 6500 (pH 7) and 2100 (pH 9); (*E*)- isomer 6600 (pH 4), 4900 (pH 7) and 1700 (pH 9).

Photolysis: Half-life for phototransformation in water at 21°C and a nominal pH of 7 was 482 hours (Calmels, 1992; Robson 1992, 1993, 1994)

Data on the solubility of chlorfenvinphos in water, fat and organic solvents and the octanol-water partition coefficient were also supplied but were not supported by full study reports (Anon, 1996c)

Formulations

Chlorfenvinphos is formulated as GR, WP and EC products.

METABOLISM AND ENVIRONMENTAL FATE

Animal metabolism

Humans. In a volunteer study (Hutson, 1969) a male was given a single oral dose of 12.5 mg of [^{14}C]chlorfenvinphos in olive oil. The radiolabel was rapidly excreted in the urine with 72% of the applied dose excreted in the first 4.5 hours and 94.2% in 26.5 hours. Five metabolites were identified in the urine, two of which were quantified. These were 2-chloro-1-(2,4-dichlorophenyl)vinyl ethyl hydrogen phosphate and 2,4-dichloromandelic acid, which accounted for 23.8 and 23.9 % of the applied dose respectively. The other three metabolites were tentatively identified as [1-(2,4-dichlorophenyl)ethyl-â-D-glucopyranosidyl]uronic acid, 2,4-dichlorophenylethanediol glucuronide and 2,4-dichlorohippuric acid (*N*-2,4-dichlorobenzoylglycine).

Rats and dogs. In a study on rats and dogs (Hutson and Hathway, 1966) rats were given single oral doses of 2 mg/kg [^{14}C]chlorfenvinphos. Within 96 hours 87% of the applied dose was excreted in the urine, 1.4% in expired air and 11% in the faeces. Most of the radiolabel in the urine was excreted in the first 24 hours.

Dogs were given single oral doses of 0.3 mg/kg [^{14}C]chlorfenvinphos in gelatine capsules. In the first 24 hours 86% of the applied dose was excreted in the urine, and in 96 hours 89.4% was excreted in the urine and 4.5% in the faeces.

The urine was analysed for metabolites: five were identified from the rats and four from the dogs. Their relative proportions are shown in Table 1.

Table 1. Metabolites of chlorfenvinphos in rat and dog urine.

Metabolite	% of ^{14}C in urine	
	Rat	Dog
2,4-dichlorophenylethanediol glucuronide	3	3
[1-(2,4-dichlorophenyl)ethyl-â-D-glucopyranosidyl]uronic acid	47	4
2,4-dichlorohippuric acid	5	absent
2,4-dichloromandelic acid	8	5
2-chloro-l-(2,4-dichlorophenyl)vinyl ethyl hydrogen phosphate	37	78

Cattle. In a briefly reported study (Hutson and Hoadley, 1969; Hunter, 1969), one small (400 kg) Friesian cow was given a single intramuscular injection of 233 mg of [*vinyl*-1,2-^{14}C]chlorfenvinphos (unspecified radiochemical purity; specific radioactivity 2.8 µCi/mg) in 'Infonutrol'. The cow had free access to water and hay, was fed 3.6 kg of concentrate per day over the five day duration of the study and was milked twice daily (at 10 am and 4 pm).

Milk samples were analysed for total radioactive residues by LSC, and were found to contain a maximum initial radioactive residue of 0.076 mg/kg chlorfenvinphos equivalents. Overall, only 0.2% of the administered dose was recovered in the milk (Table 2).

Table 2. Radioactive residues in milk after intramuscular administration of [*vinyl*-^{14}C]chlorfenvinphos to a cow.

Day	Time	^{14}C	
		% of administered dose	mg/kg parent equivalents
1	4 pm	0.13	0.076
2	10 am	0.04	0.011
2	4 pm	0.01	0.006
3	10 am	0.01	0.004
3	4 pm	0.009	0.006
4	10 am	0.006	0.002
4	4 pm	0.0005	0.0003
5	10 am	0.001	0.0005

The nature of the residues was investigated in the first milk sample. The second sample was analysed for the parent compound only. The milk was separated into cream, residual whey, and precipitated protein by centrifugation. The cream was extracted with acetone and hexane. The radioactivity was distributed as follows: hexane-soluble fat 52%, acetone-soluble fat 28%, insoluble fat residue 3%, whey 13%, and insoluble protein 4%. A fat sample was prepared by mixing dried cream with sodium sulfate before dissolution in acetone/hexane and concentration by evaporation. The fat content of the milk was estimated as 5%. TLC of the fat solution with reference standards showed mainly chlorfenvinphos (0.049 mg/kg) with the metabolites (found in the range 0.0004 to 0.0023 mg/kg) shown in Table 3. The levels of unchanged chlorfenvinphos in the first and second milk samples represented 75% and 60% of the total radioactive residue (TRR) respectively. The major metabolite found in milk was 2,4-dichloroacetophenone (III), found only at a level of 0.0023 mg/kg (3.6% of the TRR). Of the radioactivity remaining in the whey, 29% was extracted with ether at neutral pH (postulated as parent) and 23% was extracted at pH 2 (considered to be indicative of metabolites VI and IX).

Table 3. Distribution and nature of the radioactive residue in milk fat.

Metabolites		Residue in milk fat expressed as mg/kg in whole milk
I	chlorfenvinphos	0.049
II	2,4-dichlorophenacyl chloride	0.0008
III	2,4-dichloroacetophenone	0.0023
IV	1-(2,4-dichlorophenyl)ethanol	0.0014
V	1-(2,4-dichlorophenyl)ethane-1,2-diol	not detected
VI	2,4-dichloromandelic acid	0.0011
VII	2,4-dichlorobenzoic acid	<0.0014
VIII	2-chloro-1-(2,4-dichlorophenyl)ethanol	0.0004
IX	desethyl-chlorfenvinphos	0.0007

Urine was sampled at an unspecified time and found to contain 29% of the administered dose, of which 90% was extracted with ether/ethanol. Paper chromatography in butanol/ammonia revealed the presence of metabolites IV, V, VI, and IX accounting for 34%, 23%, 12% and 57% of the extracted radioactive residue.

The proposed metabolic pathway for chlorfenvinphos in ruminants is given in Figure 1 below.

A number of investigations with [^{32}P]chlorfenvinphos were briefly reported in a paper published in 1966. In the first of these [^{32}P]chlorfenvinphos (unspecified radiochemical purity) was applied dermally to two calves in two litres of spray (one at 0.25% and the other at 0.05% concentration). Omental fat samples were taken at 3, 7 and 15 days after spraying and were found to contain radioactive residues of 0.675, 0.055 and “0” mg/kg from the 0.25% treatment and 0.06, 0.001 and “0” mg/kg from the 0.05% treatment.

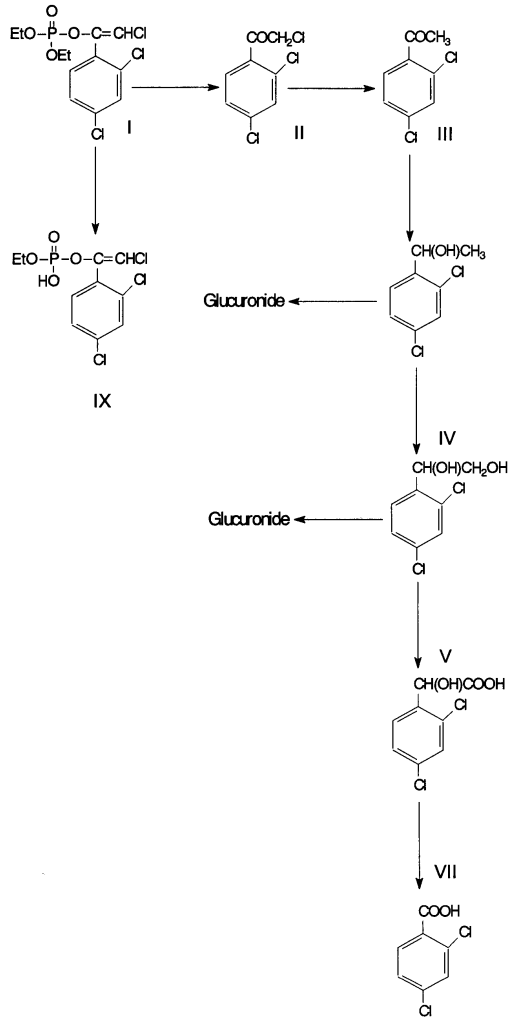
In a second investigation, two calves were similarly treated dermally with 2 litres of a spray emulsion, one at 0.25% and the other at 0.5% concentration. Both animals were killed 7 days after treatment and samples of renal and omental fat, heart, kidney, and muscle were taken for radiometric analysis. The results are shown in Table 4.

Table 4. Radioactive residues in fat and tissues of calves 7 days after treatment with a [^{32}P]chlorfenvinphos spray.

Sample	^{32}P as chlorfenvinphos, mg/kg	
	0.25% spray	0.5% spray
Renal fat	0.042	0.204
0mental fat	0.036 (0.36[1])	0.223
Heart	0.002	0.015
Kidney	0.001	0.008
Muscle	0.001	0.008

[1] Additional sample taken by omentectomy 24 hours after treatment

Figure 1. Proposed metabolic pathways of chlorfenvinphos in ruminants.



In a third investigation, three Hereford calves were treated “to saturation” with a 0.25% spray emulsion of chlorfenvinphos. The calves were killed 7 (calf A), 16 (calf B) and 28 (calf C) days after treatment. Samples of omental and renal fat, muscle, heart, kidney, liver, brain, and spleen were analysed for the parent compound by GLC (Table 5).

Table 5. Residues of chlorfenvinphos in fat and tissues of cattle sprayed ‘to saturation’ with a 0.25% spray of chlorfenvinphos.

Sample	Chlorfenvinphos, mg/kg, at intervals, days, after spraying		
	7	16	28
Omental fat	0.085	0.006	<0.005
Renal fat	0.021	<0.005	<0.005
Muscle	<0.004	<0.004	<0.004
Heart	<0.004	<0.004	<0.004
Kidney	<0.004	<0.004	<0.004

Sample	Chlorfenvinphos, mg/kg, at intervals, days, after spraying		
	7	16	28
Liver	<0.004	<0.004	<0.004
Brain	<0.004	<0.004	<0.004
Spleen	<0.004	<0.004	<0.004

In a fourth, more comprehensive, investigation (Ivey *et al.*, 1966) six Hereford cattle (group A) were sprayed 12 times at weekly intervals with a 1% emulsion of chlorfenvinphos. Another group (B) of six cattle was sprayed six times at two-week intervals with the same concentration of spray. Control animals were sprayed with "formulation blank". Fat samples were taken by omentectomy from three animals from group A, one week after the 1st, 2nd, 4th, 6th, 8th, 10th and 12th spray treatments, and from three animals from group B two weeks after each treatment. The samples were analysed for chlorfenvinphos and the metabolite 2,4-dichlorophenacyl chloride by GLC. 2,4-dichlorophenacyl chloride was not detected in any of the samples. The residues of chlorfenvinphos in the omental fat of the cattle in groups A and B are shown in Tables 6 and 7 respectively. All results were corrected for blanks and a recovery of 80%.

Table 6. Residues of chlorfenvinphos in omental fat from cattle sprayed weekly with a 0.1% emulsion.

Animal	Residues, mg/kg, in omental fat 7 days after indicated spray						
	1st	2nd	4th	6th	8th	10th	12th
A.1	0.012				0.161		0.010
A.2	0.009		0.065		0.121		0.010
A.3	0.056		0.142		0.245		0.020
A.4		0.047		0.051		0.020	
A.5		0.070		0.065		0.019	
A.6		0.020		0.035		0.009	

Table 7. Residues of chlorfenvinphos in omental fat from cattle sprayed biweekly with a 0.1% emulsion.

Animal	Residues, mg/kg, in omental fat 14 days after indicated spray					
	1st	2nd	3th	4th	5th	6th
B.1	<0.005		<0.005		0.247	
B.2	0.006		0.006		0.170	
B.3	<0.005		<0.005		0.080	
B.4		0.009		<0.005		0.180
B.5		0.008		0.007		0.110
B.6		<0.005		<0.005		

No residues of chlorfenvinphos were detected in omental or renal fat taken from animals of group A or B slaughtered 14 and 28 days after the last spray respectively.

In a very briefly reported study (Roberts *et al.*, 1961) two dairy cows were sprayed with ^{32}P-labelled chlorfenvinphos (unspecified radiochemical purity; specific activity 3.4 mCi/g). One cow (Holstein) was treated with 400 ml of a water-based spray formulated from a simple EC containing 5 g of the radiolabelled compound. This was done by spraying 200 ml on each side of the cow, avoiding the udder, and working into the hair with a comb. The second cow (Jersey) was similarly treated with 5 g of ^{32}P-labelled chlorfenvinphos (unspecified radiochemical purity; specific activity 1.7 mCi/g), using a

different EC formulation based on xylene and lanolin in a total spray volume of 60 ml; this was not worked into the hair, and resulted in a loss of about 5%. Duplicate milk samples (200 ml) were taken from the morning milk just before treatment and up to 12 days after treatment. The organosoluble radioactivity was extracted and determined with a Geiger tube. The maximum residues were found in the milk sampled 5 hours after treatment, 0.06 mg/kg in the Holstein and 0.03 mg/kg in the Jersey. One day after treatment the residues had decreased to 0.011 mg/kg and 0.005 mg/kg in the Holstein and Jersey milk, and residues were finally eliminated in 12 and 10 days after treatment respectively.

Chamberlain and Hopkins (1962) applied [^{32}P]chlorfenvinphos (radiochemical purity in the range 76 to 87%) at 55, 25 and 8 mg/kg body weight to the back and sides of three steers, A, B and C respectively, in a volume of 300 ml as an EC spray using a chromatography spray bottle held 1.2 cm from the surface of the skin, with subsequent combing into the skin. Blood samples and excreta were taken at regular intervals for 1 week after treatment and radioassayed with a gas-flow proportional counter. The results are shown in Table 8. It was stated that 18 to 42% of the chloroform-soluble radioactivity in the blood co-chromatographed with unchanged chlorfenvinphos. Twenty five to 35% of the applied radioactivity was excreted in the urine, but only 2% was recovered from the faeces.

It was reported, although full details were not given, that 9 or 10 radioactive compounds were excreted in the urine, one of which (representing 2 to 14% of the TRR) co-chromatographed with dimethyl hydrogen phosphate. Another metabolite (in the range 0.4 to 7%) was tentatively identified as diethyl 1-methyl-2-chlorovinyl hydrogen phosphate. The predominant component, which represented “49% of all the radioactive material in early hourly samples”, remained unidentified. It was stated to decrease in concentration with time. A further unidentified component was reported in the range 6 to 44% of the TRR.

Table 8. Total radioactive residues in blood, urine and faeces of dermally treated steers.

Time	^{32}P as chlorfenvinphos, mg/kg								
	Steer A			Steer B			Steer C		
	Blood[1]	Urine	Faeces	Blood[1]	Urine	Faeces	Blood[1]	Urine	Faeces
1 h	7.1 (1.4)	741					0.7	2.8	
2 h	7.8 (1.2)	2504		3.9 (0.3)			0.9	16	
3 h	6.7 (0.8)	2966	7.2	3.9 (0.7)	1148	1.1	0.8 (0.04)	27	0.5
6 h	3.8 (0.8)	2589	13	2.2 (0.3)	1117	2.9	0.6	84	2.2
9 h	3.2 (0.4)	1556	113				0.4	74	
12 h	3.3	1445					0.3	57	
18 h	2.9	918	428	0.8 (0.2)	408	56	0.2	56	
1 day	2.1	684	441	0.7	193	52	0.2	38	7.6
2 days	1.5	196	108		121	32	0.2	26	4.9
4 days	1.1	46	26	0.6	57	42	0.2	17	5.0
7 days	0.9	18	21	0.4	18	7	0.3	6.6	3.8

[1] Chloroform-soluble residues are shown in parentheses

A further study on the toxicology and metabolism of chlorfenvinphos (Herbst and Herbst, 1995) was submitted but was not evaluated because it was written in German.

Plant metabolism

In a 1965 study, later described in two papers and summarized in a further review (Beynon and Wright 1965, 1967; Beynon *et al.* 1973; Anon, undated) [^{14}C]vinyl-labelled (*E)*-chlorfenvinphos (radiochemical purity not specified) was applied to soil around cabbage plants at a rate of 4 mg per plant (growth stage not specified) to soil eight weeks after it had been sown with carrots at an application rate of 3.4 kg ai/ha, and to soil ten weeks after it had been sown with onions at a rate of 4.5 kg ai/ha. Cabbages were harvested 12-14 weeks, and carrots and onions 18 weeks, after treatment. All three crops were grown in the laboratory.

The samples were extracted with acetone and analysed by TLC (only brief details supplied). Quantification of the unextractable residues was by combustion analysis.

The results are summarized in Tables 9-11 below. In cabbages no radiolabel (<0.01 mg/kg as chlorfenvinphos) was detected in the heart but 0.11 mg/kg was found in the outer leaves, of which 0.05 mg/kg was extractable but not characterized. An acetone extract of the stump/root was found to contain a residue of 0.26 mg/kg, of which 95% was chlorfenvinphos and 5% 2,4-dichloroacetophenone. A total residue of 0.15 mg/kg was found in the roots of carrots, of which 0.12 mg/kg was chlorfenvinphos, and a total residue of 0.08 mg/kg in onion bulbs, of which 0.07 mg/kg was chlorfenvinphos.

Table 9. Residues of (*E*)-[*vinyl*-^{14}C]chlorfenvinphos and its breakdown products in cabbages grown indoors following application to the soil around the roots at transplanting.

Sample	^{14}C as chlorfenvinphos[1]	
	Acetone-extractable	Acetone-unextractable
Heart	0.005	0.005
Outer leaf	0.05	0.06
Dead leaf (on soil)	0.15	0.04
Stump and root	0.26[2]	0.26

[1] Controls <0.005 mg/kg
[2] 95% chlorfenvinphos, 5% 2,4-dichloroacetophenone

Table 10. Residues of (*E*)-[*vinyl*-^{14}C]chlorfenvinphos and its breakdown products in carrots grown indoors.

Sample	Acetone extractability	Component	^{14}C as chlorfenvinphos[1]
edible root	Extractable	Chlorfenvinphos	0.12
		2,4-dichloroacetophenone	0.01
	Unextractable	Unidentified	0.024
leaf	Extractable	Chlorfenvinphos	0.33
	Unextractable	Unidentified	0.02

[1] Recovery of [^{14}C]chlorfenvinphos at approximately 1 mg/kg was 82%

Table 11. Residues of (*E*)-[*vinyl*-^{14}C]chlorfenvinphos and its breakdown products in onions grown indoors.

Sample	Acetone extractability	Component	^{14}C as chlorfenvinphos[1]
Bulb	Extractable	Chlorfenvinphos	0.07
	Unextractable	Unidentified	0.01
Leaf	Extractable	Unidentified	0.05
	Unextractable	Unidentified	0.01

[1] Recovery of [^{14}C]chlorfenvinphos at approximately 0.7 mg/kg was 90-95%. Control plants showed ^{14}C corresponding to <0.01 mg/kg

In reviews of the metabolism and degradation of vinyl phosphate insecticides (Beynon *et al.*, 1973; Beynon and Wright, 1968) it was reported that [^{14}C]vinyl-labelled (*E)*-chlorfenvinphos of unspecified radiochemical purity was foliar-applied (precise method and rate not specified) to potatoes, cabbage and maize growing in a greenhouse. Analyses of crop samples taken 28-112 days after treatment gave the results shown in Table 12. The methods used to extract and analyse the samples were not described.

In potatoes, 39% of the applied ^{14}C was found in the foliage after 28 days and less than 0.5% in the tubers after 80 days. Evidence for identification was not given, but the authors indicated that 21% of the applied radiolabel represented chlorfenvinphos, 11% a conjugate of 1-(2,4-dichlorophenyl)ethanol and 7.2% could not be extracted with acetone. They suggested that plant metabolism studies with tetrachlorvinphos indicated that the unextracted residues were mainly further quantities of conjugates of 1-(2,4-dichlorophenyl)ethanol.

Twenty per cent of the radiolabel applied to cabbages was found in the foliage 24 days after treatment: 6.7% of the dose as chlorfenvinphos and 6.7% as the 1-(2,4-dichlorophenyl)ethanol conjugate; 6.7% could not be extracted with acetone and again appeared to consist mainly of conjugates of 1-(2,4-dichlorophenyl)ethanol.

In maize, 54% of the applied radiolabel was found in the foliage after 24 days and less than 0.5% in the grain after 112 days. In the foliage 26% of the dose was chlorfenvinphos, 12% the 1-(2,4-dichlorophenyl)ethanol conjugate and 16%, unextractable with acetone, apparently also conjugates of 1-(2,4-dichlorophenyl)ethanol.

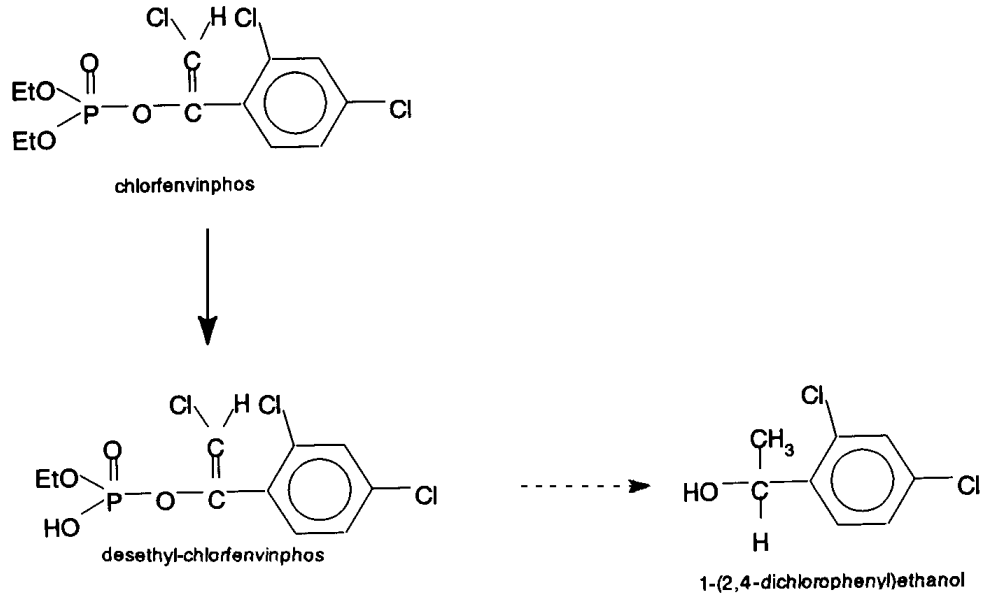
Table 12. Metabolites found after foliar treatment of glasshouse crops with [^{14}C]chlorfenvinphos.

Crop	Sample	Days from treatment to sampling	% of applied ^{14}C			
			Chlorfenvinphos	Conjugate of 1-(2,4-dichlorophenyl)ethanol[1]	Unextracted by acetone[2]	Total
Potato	Whole plant above ground Tubers	28 80	21 -	11 -	7.2 -	39 <0.5
Cabbage	Whole plant above ground	24	6.7	6.7	6.7	20
Maize	Whole plant above ground Grain	24 112	26 -	12 -	16 -	54 <0.5

[1] Approximately 1% of the activity ascribed to the conjugate could be from desethyl-chlorfenvinphos
[2] Probably also mainly conjugates of 1-(2,4-dichlorophenyl)ethanol

The metabolic pathway proposed on the basis of foliar application is shown in Figure 2.

Figure 2. Metabolism of chlorfenvinphos in plants following foliar treatment.



Environmental fate in soil and water/sediment systems

In the study of plant metabolism following soil application described above (Beynon and Wright,

1965), further work was carried out to identify degradation products in the soil. In addition, a second phase of the study involved the treatment of different soil types with higher rates of [*vinyl*-^{14}C]chlorfenvinphos (15 mg/kg) in closed containers. Acetone extracts of soil samples taken from below the onion crop were reported to contain chlorfenvinphos at 2.4 mg/kg, desethyl-chlorfenvinphos (near 0.02 mg/kg) and 2,4-dichlorophenacyl chloride. Further treatment of the soil with acid extracted 0.35 mg/kg chlorfenvinphos equivalents, which consisted of chlorfenvinphos (0.28 mg/kg), desethyl-chlorfenvinphos (0.07 mg/kg) and a trace of 2,4-dichlorophenacyl chloride. The authors stated that the desethyl-chlorfenvinphos in the acid extract may have been present as such in the soil but was more likely to have been in the form of a salt or conjugate which was hydrolysed to desethyl-chlorfenvinphos by the acid. Few further details were given, and no results of the second phase were presented.

In a summarized study of the degradation of chlorfenvinphos in soil under laboratory conditions (Anon., undated; Beynon *et al.*, 1973) [*vinyl*-^{14}C]chlorfenvinphos was applied to 4 different soils at an initial concentration of 15 mg/kg. The pH and water contents of the soils are given in Table 13. The soils were incubated in the dark at 22°C and samples were taken for analysis at intervals for 4 months.

Table 13. Characteristics of experimental soils.

Soil type	pH	Water content (% w/w)
Clay	8.0	21.1
Loam	8.0	15.1
Sand	7.9	13.9
Peat	6.4	88.6

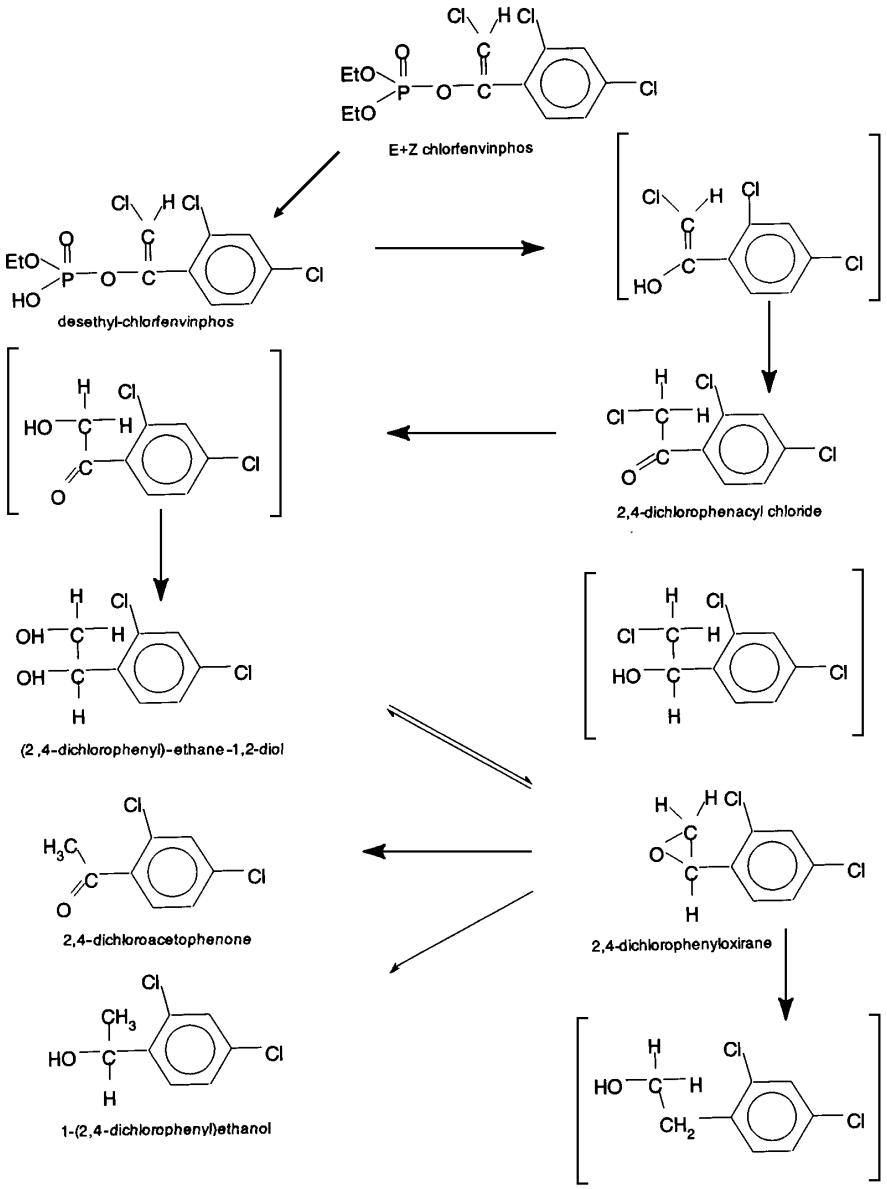
Extracts of the soils were examined for products of degradation by TLC with radio-analysis, with the results shown in Table 14. Radioactivity designated as unextractable was obtained by oxidation of the treated soil by “Van Slyke oxidation”.

Table 14. Residues of [^{14}C]chlorfenvinphos and its degradation products in soils four months after treatment.

Compound or fraction	Residue, mg/kg moist soil			
	Clay	Loam	Sand	Peat
desethyl-chlorfenvinphos	0.2	0.1	0.2	0.1
(2,4-dichlorophenyl)ethan-1,2-diol	≤0.02	≤0.02	≤0.03	≤0.02
unknown	0.07	0.06	0.04	0.1
1-(2,4-dichlorophenyl)ethanol	1.0	0.1	0.06	0.2
chlorfenvinphos	2.0	4.2	1.0	4.7
2,4-dichloroacetophenone	0.5	0.2	0.1	0.2
2,4-dichlorophenacyl chloride	≤0.005	≤0.005	≤0.005	≤0.005
2,4-dichlorophenyloxirane	≤0.005	≤0.005	≤0.005	≤0.005
salts or conjugates of desethyl-chlorfenvinphos	0.1	0.5	0.6	<0.05
unextractable radioactivity	2.0	1.8	-	-

The pathways for the degradation of chlorfenvinphos proposed by the authors are shown in Figure 3. Structures enclosed in brackets were described as “transient intermediates”, although no derivative of the phenethyl alcohol "intermediate" is suggested.

Figure 3. Proposed degradation pathways of chlorfenvinphos in soil.



The high application rate was employed to identify products which might not be identified at lower rates. Radioactivity which was not recovered from the soils represented 60-80% of the applied dose; it included $^{14}CO_2$ and residues which could not be extracted with common organic solvents. The predominant products were 1-(2,4-dichlorophenyl)ethanol, 2,4-dichloroacetophenone and the sodium salt of desethyl-chlorfenvinphos.

In additional summarized experiments (Beynon *et al.,* 1973) onions and carrots grown in boxes containing John Innes No 2 compost under glasshouse conditions were treated with [^{14}C]chlorfenvinphos at the commercial rate of 3.4-4.5 kg/ha. Eight weeks after application of the insecticide the ^{14}C in the compost, expressed as mg chlorfenvinphos equivalents/kg moist soil, was accounted for by 2.7 mg/kg of chlorfenvinphos, 0.09 mg/kg of desethyl-chlorfenvinphos and 0.03 mg/kg of 2,4-dichloroacetophenone or 2,4-dichlorophenacyl chloride.

A summarized study (Anon., undated), presented as a poor copy which was illegible in places, described three further experiments on degradation in field soils. In all of these it was unclear whether the application rate referred to product/ha or active ingredient/ha. In the first experiment, chlorfenvinphos was applied at 4.5 or 9 kg/ha to crops in the field at 4 sites in the UK in spring or summer. Soil samples were taken for analysis at intervals up to 6 months. The soils were a brick-earth, a sandy loam, a loam and a peat. Half-lives of chlorfenvinphos were in the range of about 14-84 days in the mineral soils and more than 150 days in the peat soil. 2,4-dichlorophenacyl chloride was found in peat samples taken 4 weeks or more after treatment at concentrations up to 0.1 mg/kg of soil (105 day sample) after application of chlorfenvinphos at 9 kg/ha. The properties of the soils were not given.

In the second experiment, chlorfenvinphos was applied to field soils at rates of 4.5, 6.7, 9 or 22 kg/ha. Samples of soil were taken for analysis at intervals up to 6 months after application in spring or summer and examined for the degradation products 1-(2,4-dichlorophenyl)ethanol, 2,4-dichloroacetophenone and 2,4-dichlorophenacyl chloride. There was no evidence of isomerisation of the (*Z*)- isomer in soil. 2,4-Dichlorophenacyl chloride was not detected in the soils within 6 months of application at 4.5 or 6.7 kg/ha but was found at a concentration of 0.1 mg/kg 105 days after application at 9 kg/ha. The highest residue of 2,4-dichloroacetophenone was 0.2 mg/kg, found 30 days after application at 9 kg/ha. 1-(2,4-dichlorophenyl)ethanol was not detected within 6 months of treatment at 4.5-9 kg/ha with a limit of detection of 0.2 mg/kg, but was found at 0.6 mg/kg 28 days after application of the unrealistically high rate of 22 kg/ha.

In the third experiment, carried out in 1966-7, labelled chlorfenvinphos was applied as a GR to a brick loam soil and as an EC to clay loam soil in the UK at 4 kg ai/ha. The residues remaining in soil samples taken at intervals are given in Table 15.

Table 15. Decay of chlorfenvinphos residues in soils.

Interval	Chlorfenvinphos equivalents, mg/kg	
	Faversham brick loam	Woodstock clay loam
0 days	-	3.2
2 days	4.6	-
1 week	4.4	-
2 weeks	2.6	-
4 weeks	4.4	3.3
10 weeks	1.1	1.9
20 weeks	-	1.1
52 weeks	0.11	0.4
82/86 weeks	0.05	0.3
99 weeks	Illegible	-
107 weeks		0.04

A further paper was submitted which provided an overview of the occurrence and fate of residues in soil, mainly of the work described above (Anon., 1985). Laboratory data on the degradation of chlorfenvinphos in water/sediment systems (Wable, 1993) and in fresh water aquatic systems (Edwards and Gibb, 1981) were also submitted but not reviewed.

METHODS OF RESIDUE ANALYSIS

Analytical methods

Fruit and vegetables. The Netherlands submitted a qualitative multi-residue TLC method which allows the determination of the (*E*)- and (*Z*)- isomers of chlorfenvinphos (Anon., 1988a). Samples are extracted with ethyl acetate in the presence of sodium sulfate. An aliquot of the extract is run on a TLC plate using an organic solvent mixture (chloroform/diethyl ether, benzene/acetone, benzene/acetone/hexane, or hexane/acetone). The plate is then sprayed with a homogenate of bee heads, incubated at 370°C and subsequently sprayed with a solution of 2-naphthyl acetate and Fast Blue B. The cholinesterase from the bee heads hydrolyses 2-naphthyl acetate to 2-naphthol, which reacts with the Fast Blue B to form a dye. Where inactivators of cholinesterase are present no dye is formed, so such places appear as white spots on a pink-violet background.

It was reported that 0.2 mg/kg of the (*E*)- isomer and 2 to <20 mg/kg of the (*Z*)- isomer could be detected. The method is not suitable for quantitative determination.

Fruit and vegetables, animal products, and grains. A quantitative multi-residue method, also submitted by The Netherlands, allowed determination of the (*E*)- and (*Z*)- isomers of chlorfenvinphos (Anon., 1988b,c). Samples are extracted with ethyl acetate in the presence of sodium sulfate, cleaned up where necessary by gel permeation chromatography using cyclohexane/ethyl acetate as eluant, and determined in the filtered extract by GLC with a phosphorus-specific detector. The LOD was stated to be in the range 0.01-0.05 mg/kg with a recovery of >80%, although no further information on validation of the method was given.

Carrots and onions. The Netherlands provided brief details of the methods of analysis used in the trials which they reported (Olthof, 1996). Extraction with petroleum ether or ethyl acetate is followed by analysis by GLC with FP detection. The limits of determination ranged from 0.005 to 0.02 mg/kg.

Crops and soil. In a method developed by Shell (Anon., 1966) samples were extracted by maceration with acetone in petroleum spirit in the presence of anhydrous sodium sulfate. After filtering, determination was by GLC with EC detection. Interfering co-extractives were removed with a Florisil column clean-up. An LOD of 0.01 mg/kg was reported although no chromatograms or details of the commodities with which this had been achieved were submitted. No recovery or other validation data were provided.

In a second reported method (Anon., 1990) soil was mixed with anhydrous sodium sulfate before extraction of soil and crop samples with acetone/hexane, and extracts of oily crops were partitioned between hexane and aqueous acetonitrile. The extracts were cleaned up on Florisil before analysis by GLC with an NPD. The method was validated with three soils (clay loam, sandy loam and silty clay), apples, soya beans, wheat grain and cabbage by fortifying with 0.05-0.5 mg/kg of each isomer. Recoveries were consistently between 75 and 115%. At each level the standard deviation was ≤12% of the mean. Sample chromatograms showed resolution of the isomers. The limit of determination was 0.01 mg/kg of each isomer in all samples.

A further method (Anon., 1969) was submitted for the determination of 2,4-dichloroacetophenone, 1-(2,4-dichlorophenyl)ethanol, and 2,4-dichlorophenacyl chloride. Crop and soil samples were extracted with a mixture of acetone and petroleum spirit. The extracts were washed with water, dried, and analysed by GLC with an ECD. Where required, an alumina column clean-up (elution with diethyl ether in petroleum spirit) was included. The method was stated to be suitable for determining metabolites down to a level of 0.01 mg/kg except 2,4-dichloroacetophenone, 2,4-dichlorophenacyl chloride and 1-(2,4-dichlorophenyl)ethanol. The LOD for the dichlorophenylethanol was 0.1 mg/kg.

Analysis of crops in supervised trials. Several other methods (Mathews, 1972; Bosio, 1981i) included in the reports of residue trials were modifications of the methods for crops reviewed above. Extraction was into either acetone/hexane or acetone/petroleum spirit and determination was by GLC with either FP or EC detection. LODs in the range 0.01-0.05 mg/kg were reported although generally no sample chromatograms were submitted. Some samples were analysed for 1-(2,4-dichlorophenyl)ethanol, 2,4-dichlorophenacyl chloride and 2,4-dichoroacetophenone, but with limited data on validation of the methods and few sample chromatograms. Confirmation of residues, when carried out, was by GC-MS.

Grass. Samples were extracted by tumbling with anhydrous sodium sulfate, acetone and petroleum spirit. The extracts were filtered and analysed without clean-up by GLC with an ECD (Elgar, 1966e).

Milk. In a briefly summarized method (Elgar, 1966e), samples of milk were diluted with ethanol and extracted with an ether/hexane mixture. After drying over anhydrous sodium sulfate the solvent was evaporated and the fatty residue washed with hexane and extracted into acetonitrile. The acetonitrile extract was cleaned up on Florisil columns, eluting with ether in petroleum spirit. Analysis was by GLC with EC detection.

Stability of pesticide residues in stored analytical samples

No data were submitted.

Residue definition

The studies of animal and plant metabolism indicate that chlorfenvinphos is the main residue in products of animal and plant origin. A definition of the residue as "chlorfenvinphos, sum of (*E*)- and (*Z*)- isomers" is therefore considered appropriate.

USE PATTERN

Chlorfenvinphos is registered in a number of countries for use on a wide range of vegetable crops, but no uses were reported on fruit crops. Topical veterinary uses on cattle and other animals were reported for Australia.

The information on GAP supplied by the manufacturer (Anon., 1996c) was incomplete. No copies of product labels were submitted, only summary sheets. In some cases the reported PHI appeared to be inappropriate for the type of treatment (e.g. a 21-day PHI for pre-planting or pre-emergence application).

Details of registered use patterns are given in Tables 16-18.

Table 16. Registered uses of chlorfenvinphos on vegetables.

Commodity	Country	Form.	F or G	Application				PHI, days	Ref.	Remarks
				Method	Rate kg ai/ha	Spray conc, kg ai/hl	No.			
Asparagus	Netherlands	WP, EC	F	Spraying without incorporation into soil	3.84-4.0[1]	0.5-0.768	1	within two days after casing	Olthof 1996	Soil treatment
Broccoli	Germany	GR	F	Spreading and mixing	100 g/m^2	--------	1	pre-planting	Anon 1996d	Soil treatment
	Germany	GR	F	Spreading	0.1 g/plant	--------	1	5-6 days after planting	Anon 1996d	Treatment of single plants
	Germany	GR	F	Spreading with rain	2 g/100 plants	--------	1		Anon 1996d	Nursery bed seedbed
	Germany	GR	F	Spreading	2 kg/ha	--------	1	5-6 days after planting	Anon 1996d	Row treatment
	Netherlands	WP, EC, GR	F	Spraying/granular application onto plant beds	3.84-4.0[1]	0.0768-0.08	1	60 before sowing	Olthof 1996	Soil application
	Netherlands	WP, EC, GR	F	Spraying/granular application onto “production fields”	1-3.75[2]	0.05 g (WP & EC) and 0.75 g (Gr) ai/plant	1	60	Olthof 1996	At planting or after cabbage fly eggs have set
	UK	EC	F	Seed bed spray	1.34[1]	0.268-0.446	1	pre-emergence	Anon 1996e	Applied immediately after drilling
	UK	EC	F	Overall soil incorporated spray	2.35[1]	0.47-0.78	1	21 pre-planting	Anon 1996e	
	UK	EC	F	Soil drench to base of plant	---------	0.0044	1	21 post-emergence	Anon 1996e	Applied April or within 4 days of transplanting if this is later
	UK	GR	F	Sub-surface band	4.5	----------	1	21 Pre- and post-emergence	Anon 1996e	Plants or seed placed into line of granules at drilling or transplanting
	UK	GR	F	Incorporated into peat blocks	--------	50 g ai/640 litre peat	1	21 pre-planting	Anon 1996e	To protect seedlings before planting out
Brussels sprouts	Netherlands	WP /EC/ GR	F	Spraying/granular application onto plant beds	3.84-4.0[1]	0.0768-0.08	1	60 before sowing	Olthof 1996	Soil application
	Netherlands	WP /EC/ GR	F	Spraying/granular application onto “production fields”	1-3.75	0.05 g (WP & EC) and 0.75 g (Gr) ai/plant	1	60	Olthof 1996	At planting or after cabbage fly eggs have set
	UK	EC	F	Seed bed spray	1.34[1]	0.268-0.446	1	pre-emergence	Anon 1996e	Applied immediately after drilling
	UK	EC	F	Overall soil incorporated spray	2.35[1]	0.47-0.78	1	21 pre-planting	Anon 1996e	
	UK	EC	F	Soil drench to base of plant	---------	0.0044	1	21 post-emergence	Anon 1996e	Applied April or within 4 days of transplanting if this is later
	UK	GR	F	Sub-surface band	4.5	----------	1	21 Pre- and post-emergence	Anon 1996e	Plants or seed placed into line of granules at

Commodity	Country	Form.	F or G	Application				PHI, days	Ref.	Remarks
				Method	Rate kg ai/ha	Spray conc, kg ai/hl	No.			
										drilling or transplanting
	UK	GR	F	Incorporated into peat blocks	--------	50 g ai/640 litre peat	1	21 pre-planting	Anon 1996e	To protect seedlings before planting out
Cabbage	Belgium	EC	-	----------	0.01 g/plant	--------	-	56	Anon 1996c	Post-emergence
	Belgium	GR	-	----------	3-5	--------	-	56	Anon 1996c	Post-emergence
	Denmark	EC	-	----------	0.96	--------	-	56	Anon 1996c	Post-emergence
	Denmark	EC	-	----------	3.8	--------	-	56	Anon 1996c	Pre-planting
	Denmark	EC	-	----------	4	--------	-	70	Anon 1996c	Pre-planting
	France	GR	-	soil treatment	6	--------	-	15	Anon 1996c	
	France	EC	-	soil treatment	0.6-6	--------	-	15	Anon 1996c	
	France	----	-	soil treatment	6	--------	-	15	Anon 1996c	
	Germany	EC	-	furrow treatment	1.4	--------	-	28	Anon 1996c	
	Germany	GR	-	seed bed treatment	0.02 g ai/ plant	--------	-		Anon 1996c	
	Germany	GR	-	single plant treatment	0.1 g ai/ plant	--------			Anon 1996c	
	Germany	GR	-	row treatment	2	--------	-		Anon 1996c	
	Germany	GR	-	incorporation before sowing	0.1 kg^2 soil	--------	-		Anon 1996c	
, Chinese	Germany	GR	F	Spreading	0.1 kg^2 soil	--------	1	pre-planting	Anon 1996d	Soil treatment spreading and mixing
, Chinese	Germany	GR	F	Spreading	0.1 g ai/ plant	--------	1	5-6 days after planting	Anon 1996d	Treatment of single plants
, Chinese	Germany	GR	F	Spreading	2 g ai/100 plants	--------	1		Anon 1996d	Nursery bed seedbed spreading with rain
, Chinese	Germany	GR	F	Spreading	2 kg/ha	--------	1	5-6 days after planting	Anon 1996d	Row treatment
, red	Germany	GR	F	Spreading	100 g/m^2	--------	1	pre-planting	Anon 1996d	Soil treatment spreading and mixing
, red	Germany	GR	F	Spreading	0.1 g/plant	--------	1	5-6 days after planting	Anon 1996d	Treatment of single plants
, red	Germany	GR	F	Spreading	24 g/100 plants	--------	1		Anon 1996d	Nursery bed seedbed spreading with rain
, red	Germany	GR	F	Spreading	2 kg/ha	--------	1	5-6 days after planting	Anon 1996d	Row treatment
, Savoy	Germany	GR	F	Spreading	100 g^2	-------	1	pre-planting	Anon 1996d	Soil treatment spreading and mixing
, Savoy	Germany	GR	F	Spreading	0.1 g/plant	--------	1	5-6 days after planting	Anon 1996d	Treatment of single plants
, Savoy	Germany	GR	F	Spreading	2 g/100 plants	--------	1		Anon 1996d	Nursery bed seedbed

Commodity	Country	Form.	F or G	Application				PHI, days	Ref.	Remarks
				Method	Rate kg ai/ha	Spray conc, kg ai/hl	No.			
										spreading with rain
, Savoy	Germany	GR	F	Spreading	2 kg/ha	--------	1	5-6 days after planting	Anon 1996d	Row treatment
, white	Germany	GR	F	Spreading	100 g/m^2	-------	1	pre-planting	Anon 1996d	Soil treatment spreading and mixing
, white	Germany	GR	F	Spreading	0.1 g/plant	----------	1	5-6 days after planting	Anon 1996d	Treatment of single plants
, white	Germany	GR	F	Spreading	2 g/100 plants	--------	1	--------	Anon 1996d	Nursery bed seedbed spreading with rain
, white	Germany	GR	F	Spreading	2 kg/ha	--------	1	5-6 days after planting	Anon 1996d	Row treatment
	Ireland	GR	-	----------	2.25	--------	---	21	Anon 1996c	at planting
	Ireland	EC	-	----------	2.4	--------	---	21	Anon 1996c	
	Italy	GR	-	----------	2-3	--------	---	30	Anon 1996c	at transplanting
	Italy	EC	-	foliar applied	----------	0.0438-0.0614	---	30	Anon 1996c	
	Italy	WP	-	foliar applied	----------	0.0625-0.075	---	30	Anon 1996c	
	Italy	GR	-	broadcast	0.018-0.023	----------	---	30	Anon 1996c	
	Italy	EC	-	foliar applied	---------	0.05-0.0583	---	30	Anon 1996c	
	Japan	DP	-	foliar applied	0.6-0.9	----------	4	14	Anon 1996c	
	Japan	EC	-	foliar applied	----------	0.024-0.048	---		Anon 1996c	
	Netherlands	GR	-	----------	0.075 g/plant	-------	---	60	Anon 1996c	at planting
, Chinese , Oxhead , Red , Savoy , White	Netherlands	WP /EC/ GR	F	Spraying/granular application onto plant beds	3.84-4.0[1]	0.0768-0.08	1	60 before sowing	Olthof 1996	Soil application
, Chinese , Oxhead , Red , Savoy , White	Netherlands	WP /EC/ GR	F	Spraying/granular application onto “production fields”	1-3.75	0.05 g (WP & EC) and 0.75 g (Gr) ai/plant	1	60	Olthof 1996	At planting or after cabbage fly eggs have set
	Sweden	GR	-	---	0.8-1.0 and 2	------	---	at planting	Anon 1996c	
	Sweden	GR	-	---------	1-1.5	------	---	at drilling	Anon 1996c	
	Sweden	GR	-	---	1.5-2	------	---	before drilling	Anon 1996c	
	Sweden	GR	-	---	1.5-2	------	---	at planting	Anon 1996c	
	Switzerland	EC	-	----	15 g/plant[3]	------	---	21	Anon 1996c	Treatment during vegetation period
	Switzerland	WG	-	---	0.025 g/plant	------	---	21	Anon 1996c	After planting
	UK	GR	-	---------	2.25	------	1	21	Anon 1996c	At planting

Commodity	Country	Form.	F or G	Application				PHI, days	Ref.	Remarks
				Method	Rate kg ai/ha	Spray conc, kg ai/hl	No.			
	UK	EC	-	---	4.7	------	2	21	Anon 1996c	Pre-emergence
	UK	EC	-	---	2.4	------	2	21	Anon 1996c	Post-emergence
	UK	EC	F	Seed bed spray	1.34[1]	0.268-0.446	1	pre-emergence	Anon 1996e	Applied immediately after drilling
	UK	EC	F	Overall soil incorporated spray	2.35[1]	0.47-0.78	1	21 pre-planting	Anon 1996e	
	UK	EC	F	Soil drench to base of plant	--------	0.0044	1	21 Post-emergence	Anon 1996e	Applied April or within 4 days of transplanting if this is later
	UK	GR	F	Sub-surface band	4.5	----------	1	21 Pre- and post-emergence	Anon 1996e	Plants or seed placed into line of granules at drilling or transplanting
	UK	GR	F	Incorporated into peat blocks	--------	50 g ai/640 litre peat	1	21 pre-planting	Anon 1996e	To protect seedlings before planting out
Carrots	Belgium	GR	-	---	3-5	-----	---	pre-planting	Anon 1996c	
	Belgium	EC	-	---	3-5	-----	---	pre-planting	Anon 1996c	
	Denmark	GR	-	---	4	-----	---	84	Anon 1996c	Pre-planting
	Denmark	EC	-	---	4	-----	---		Anon 1996c	Pre-planting
	France	----	-	soil treatment	5	-----	---	15	Anon 1996c	
	France	GR	-	soil treatment	5	-----	---	15	Anon 1996c	
	France	EC	-	soil treatment	0.6-5	-----	---	15	Anon 1996c	
	Germany	GR	-	incorporated by sowing	5	-----	---		Anon 1996c	
	Germany	GR	-	---	5	-----	---		Anon 1996c	Post-emergence
	Germany	EC	-	In furrow	1.44	-----	---		Anon 1996c	
	Germany	GR	-	Incorporation before sowing	5	-----	---		Anon 1996c	
	Germany	GR	F	Spreading	---	----	1		Anon 1996d	Post-emergence, at planting, after planting and Before sowing
	Ireland	GR	-	---	2.25 or 4.5	-----	---	21	Anon 1996c	Before drilling
	Ireland	EC	-	---	5	-----	---	21	Anon 1996c	Pre-emergence
	Ireland	EC	-	---	2.4	-----	---	21	Anon 1996c	Post-emergence
	Italy	GR	-	broadcast	0.0018-0.0023	-----	---	30	Anon 1996c	
	Italy	EC	-	foliar	---	0.05-0.0583	---	30	Anon 1996c	

Commodity	Country	Form.	F or G	Application				PHI, days	Ref.	Remarks
				Method	Rate kg ai/ha	Spray conc, kg ai/hl	No.			
	Italy	WP	-	foliar	---	0.04-0.05	---	30	Anon 1996c	
	Italy	EC	-	foliar	---	0.0351-0.0438	---	30	Anon 1996c	
	Italy	GR	-		2-3	----	---	30	Anon 1996c	Pre-sowing, pre-transplanting
	Luxembourg	EC	-		4	-----	---		Anon 1996c	Pre-planting
	Netherlands	GR	-	In furrow	2	-----	---	60	Anon 1996c	
	Netherlands	WP	-	seed treatment	25 g ai/kg seed	-----	---	60	Anon 1996c	
	Netherlands	EC	-	-------	3-4	-----	---	60	Anon 1996c	Pre-planting
	Netherlands	WP	-	-------	3-4	-----	---	60	Anon 1996c	Post-emergence
	Netherlands	WP	-	---	3-4	-----	---	60	Anon 1996c	Pre-planting
	Netherlands	EC	-	-------	3-4	-----	---	60	Anon 1996c	Post-emergence
	Netherlands	GR	-	-------	3-4	-----	---	60	Anon 1996c	Broadcast
	Netherlands	WP/ EC/ GR	F	Broadcast spraying or granular application followed by incorporation into 5-7 cm of soil	3.84-4.0[1]	0.5-1.92	1	60 (before sowing)	Olthof 1996	Lower dosages (2.88-4 kg ai/ha) for soils with low organic matter (<3%)
	Netherlands	WP/ EC	F	soil treatment by spraying	3.84-4.0	0.5-1.92	1	60 (post - emergence at 2-leaf stage)	Olthof 1996	Lower dosages (2.8-4 kg ai/ha) for soils with low organic matter (<3%)
	Netherlands	WP	F	seed treatment	25 g ai per kg seed	---	1	---	Olthof 1996	
, winter	Netherlands	GR	F	Granular application in furrow	2.0	-----	---	60	Olthof 1996	
	Switzerland	WG	-	-------	max 0.4	-----	1	56	Anon 1996c	Treatment at vegetation period every two years
	Switzerland	WG	-	-------	max 0.6	-----	1	56	Anon 1996c	Treatment at vegetation period every two years
	UK	EC	-	---	5	-----	3	21	Anon 1996c	Pre-emergence
	UK	EC	-	---	2.4	-----	3	21	Anon 1996c	Post-emergence
	UK	GR	-	---	2.25 or 4.5	-----	1	21	Anon 1996c	Before drilling
	UK	EC	F	Overall and soil incorporated spray	2.35 (mineral soils) 4.7 (organic soils)	0.235-0.94 or 0.47-1.88	1	---	Anon 1996e	Pre-planting
	UK	EC	F	Overall spray	2.35	0.235-0.39	1-2	21	Anon	Post-

Commodity	Country	Form.	F or G	Application				PHI, days	Ref.	Remarks
				Method	Rate kg ai/ha	Spray conc, kg ai/hl	No.			
									1996e	emergence[2]
	UK	GR	F	Broadcast incorporated	2.25 (mineral soils), 4.5 (organic soils)	----	1	21	Anon 1996e	Pre-planting
Cauliflower	Germany	GR	F	Soil treatment spreading and mixing	100 g/m^2	----	1		Anon 1996d	Pre-planting
	Germany	GR	F	Spreading	0.1 g/plant	-----	1	5-6 days after planting	Anon 1996d	Treatment of single plants
	Germany	GR	F	Spreading	2 g/100 plants	-----	1	---	Anon 1996d	Nursery bed seedbed spreading with rain
	Germany	GR	F	Spreading	2 kg/ha	-----	1	5-6 days after planting	Anon 1996d	Row treatment
	Ireland	GR	-	------	2.25	-----	---	21	Anon 1996c	
	Ireland	EC	-	-------	2.4	---	---	21	Anon 1996c	
	Netherlands	GR	-	---	0.075 g ai/plant	-----	---	60	Anon 1996c	At planting
	Netherlands	WP /EC/ GR	F	Spraying/granular application onto plant beds	3.84-4.0[1]	0.0768-0.08	1	60 before sowing	Olthof 1996	Soil application
	Netherlands	WP /EC/ GR	F	Spraying/granular application onto “production fields”	1-3.75	0.05 g (WP & EC) and 0.75 g (Gr) ai/plant	1	60	Olthof 1996	At planting or after cabbage fly eggs have set
	UK	EC	-	-------	5	-----	2	21	Anon 1996c	Pre-emergence
	UK	GR	-	-------	2.25	-----	1	21	Anon 1996c	At planting
	UK	EC	-	---	2.4	-----	2	21	Anon 1996c	Post-emergence
	UK	EC	F	Seed bed spray	1.34	0.268-0.446	1	pre-emergence	Anon 1996e	Applied immediately after drilling
	UK	EC	F	Overall soil incorporated spray	2.35	0.47-0.78	1	21	Anon 1996e	Pre-planting
	UK	EC	F	Soil drench to base of plant	---	0.0044	1	21	Anon 1996e	Applied post-emergence in April or within 4 days of transplanting if this is later
	UK	GR	F	Sub-surface band	4.5	----	1	21	Anon 1996e	Plants or seed placed into line of granules at drilling or transplanting
	UK	GR	F	Incorporated into peat blocks	------	50 g ai/640 litre peat	1	21	Anon 1996e	To protect seedlings before planting out
Celeriac	Netherlands	WP/ EC/ GR	F	Spraying or granular application	3.84-4.0	0.5-1.92	1	---	Olthof 1996	Broadcast; incorporation before sowing
	UK	EC	F	Overall spray	2.35	0.39-0.78	1	21	Anon	Pre-planting

Commodity	Country	Form.	F or G	Application				PHI, days	Ref.	Remarks
				Method	Rate kg ai/ha	Spray conc, kg ai/hl	No.			
									1996e	
	UK	GR	F	Broadcast incorporated	2.25 (mineral soils) 4.5 (organic soils)	---	1	---	Anon 1996e	Pre-planting
Celery, leaf and blanched	Netherlands	WP/ EC	F	Spraying directly to soil followed by incorporation before sowing	3.84-4.0	0.5-1.92	1	---	Olthof 1996	
Cucumber	Germany	GR	F	Spreading	---	3 kg ai/ha	1	---	Anon 1996d	At planting, after planting, before sowing
Fennel Bulb	Netherlands	WP/ EC	F	Spraying soil treatment	3.84-4.0	0.5-1.92	1	---	Olthof 1996	Incorp. at sowing
Horseradish	UK	EC	F	Overall and soil incorporated spray	2.35 (mineral soils) 4.7 (organic soils)	0.235-0.94 or 0.47-1.88	1	---	Anon 1996e	Pre-planting
	UK	EC	F	Overall spray	2.35	0.235-0.39	1-2	21	Anon 1996e	Post-emergence[2]
	UK	GR	F	Broadcast incorporated	2.25 (mineral soils), 4.5 (organic soils)	----	1	21	Anon 1996e	Pre-planting
Kale	Germany	GR	F	Soil treatment spreading and mixing	100 g/m^2	----	1	---	Anon 1996d	Pre-planting
	Germany	GR	F	Spreading	0.1 g/ plant	-----	1	5-6 days after planting	Anon 1996d	Treatment of single plants
	Germany	GR	F	Spreading	2 g/100 plants	-----	1	---	Anon 1996d	Nursery bed seedbed spreading with rain
	Germany	GR	F	Spreading	2 kg/ha	-----	1	5-6 days after planting	Anon 1996d	Row treatment
	Netherlands	WP/ EC/ GR	F	Spraying or granular application to soil. Incorporation before sowing	3.84-4.0	0.0768-0.08	1	60	Olthof 1996	Application on plant beds
	Netherlands	WP /EC/ GR	F	Spraying/granular application onto “production fields”	1-3.75	0.05 g (WP & EC) and 0.75 g (Gr) ai/plant	1	60	Olthof 1996	At planting or after cabbage fly eggs have set
	Portugal	24% EC	-	-------	100 ml/30-50l water	-----	---	42	Anon 1996c	Pre-emergence
	Spain	EC	-	-------	2	-----	---	30	Anon 1996c	Pre-planting
	Spain	EC	-	Spray	2	-----	---	30	Anon 1996c	
	Spain	GR	-	Broadcast	2-3	-----	---	30	Anon 1996c	
Kohlrabi	Germany	GR	F	Soil treatment spreading and mixing	100 g/m^2	----	1	---	Anon 1996d	Pre-planting
	Germany	GR	F	Spreading	0.1 g/plant	-----	1	5-6 days after planting	Anon 1996d	Treatment of single plants
	Germany	GR	F	Spreading	2 g/100	-----	1	---	Anon	Nursery bed

Commodity	Country	Form.	F or G	Application				PHI, days	Ref.	Remarks
				Method	Rate kg ai/ha	Spray conc, kg ai/hl	No.			
					plants				1996d	seedbed spreading with rain
	Germany	GR	F	Spreading	2 kg/ha	-----	1	5-6 days after planting	Anon 1996d	Row treatment
	Netherlands	WP /EC/ GR	F	Spraying/granular application onto plant beds	3.84-4.0[1]	0.0768-0.08	1	60 before sowing	Olthof 1996	Soil application
	Netherlands	WP /EC/ GR	F	Spraying/granular application onto "production fields"	1-3.75	0.05 g (WP & EC) and 0.75 g (Gr) ai/plant	1	60	Olthof 1996	At planting or after cabbage fly eggs have set
	UK	EC	F	Overall spray at pre-planting or root dip at transplanting	2.35	0.39-0.78	1	21	Anon 1996e	
Leek	Germany	EC	F	Spraying	0.144	0.024	1	28	Anon 1996d	At infestation
	Netherlands	WP/ EC/ GR	F	Spraying or granular soil treatment.	5.76-6.0	0.75-2.88	1	60	Olthof 1996	Incorp. before sowing
Mooli	UK	GR	F	Broadcast incorporated	2.0	----	1	---	Anon 1996e	pre-planting
Mushroom	UK	EC		Compost incorporated spray before spawning (inside)	------	72 4 g ai per tonne compost	1	---	Anon 1996e	Maximum of one treatment per spawning
	UK	EC		Casing incorporated spray before adding to bed (inside)	---	54 g ai per tonne casing	1	---	Anon 1996e	Maximum of one treatment per spawning
	UK	GR		Compost incorporated (inside)	------	110 g ai per tonne compost	1	21	Anon 1996e	At spawning
	UK	GR		Casing incorporated before adding to bed (inside)	---	50 g ai per tonne casing	1	21	Anon 1996e	At spawning
, edible fungi other than mushrooms	UK	EC	F	Compost incorporated spray before spawning	------	72 g ai per tonne compost	1	---	Anon 1996e	Maximum of one treatment per spawning
	UK	EC		Casing incorporated spray before adding to bed (inside)	---	54 g ai per tonne casing	1	---	Anon 1996e	Maximum of one treatment per spawning
	UK	GR		Compost incorporated (inside)	------	110 g ai per tonne compost	1	21	Anon 1996e	At spawning
	UK	GR		Casing incorporated before adding to bed (inside)	---	50 g ai per tonne casing	1	21	Anon 1996e	At spawning
Onion	Belgium	EC	-	------	3-5	-----	---	---	Anon 1996c	Pre-planting
	Belgium	GR	-	------	3-5	-----	---	---	Anon 1996c	Pre-planting
	Denmark	GR	-	-------	4	-----	---	35	Anon 1996c	Pre-planting
	Denmark	EC	-	-------	1	-----	---	56	Anon 1996c	Post-emergence

Commodity	Country	Form.	F or G	Application				PHI, days	Ref.	Remarks
				Method	Rate kg ai/ha	Spray conc, kg ai/hl	No.			
	France	GR	-	Soil treatment	5	-----	---	15	Anon 1996c	
	France	EC	-	Soil treatment	0.6-5	-----	---	15	Anon 1996c	
	France	---	-	Soil treatment	5	-----	---	15	Anon 1996c	
	Germany	GR	-	Incorporated by sowing	5	-----	---	---	Anon 1996c	
	Germany	EC	-	In furrow	1.4	-----	---	---	Anon 1996c	
	Germany	GR	-	Incorporation before sowing	5	-----	---	---	Anon 1996c	
	Germany	GR	--	---	5	-----	---	---	Anon 1996c	Post-emergence
	Germany	GR	F	Spreading	5 kg ai/ha	----	1	---	Anon 1996d	At planting, after planting, before sowing, post emergence
	Japan	DP	-	Broadcast	0.6-13.5	-----	---		Anon 1996c	
	Japan	EC	-	Foliar	---	0.024-0.032	---	7	Anon 1996c	
	Luxembourg	EC	-		4.8	-----	---		Anon 1996c	Pre-planting
	Netherlands	GR	-	In furrow	1.2	-----	---	60	Anon 1996c	
	Netherlands	WP	-	-------	6	-----	---	60	Anon 1996c	Pre-planting
	Netherlands	EC	-	---	6	-----	---	60	Anon 1996c	Pre-planting
	Netherlands	GR	-	Broadcast	6	-----	---	60	Anon 1996c	
, Bulb, Silverskin	Netherlands	WP/ EC/ GR	F	Spraying or granular broadcast soil application	5.76-6.0	0.75-2.88	1	---	Olthof 1996	Incorp. before sowing
, Bulb, Silverskin	Netherlands	GR	F	Granular application	1.2	----	1	---	Olthof 1996	Incorp. at sowing
	Sweden	GR	-	------	0.8-1	-----	---	---	Anon 1996c	Post-emergence
	Sweden	GR	-	---	1	-----	---	---	Anon 1996c	At planting
	Switzerland	WG	-	---	1-2 g ai/m soil	-----		21	Anon 1996c	Post-emergence. One treatment every two years
	Switzerland	EC	-	-------	37.5 ml ai/m	-----		21	Anon 1996c	Treatment at vegetation period
Parsley	Netherlands	WP	F	Spraying	3.84-4.0	0.4-1.92	1	---	Olthof 1996	Soil incorporation directly after treatment
	Netherlands	EC	F	Spraying	3.84-4.0	0.4-1.92	1	---	Olthof 1996	Soil incorporation directly after treatment
	Netherlands	WP	F	Spraying	3.84-4.0	0.5-1.92	1	---	Olthof 1996	Soil incorporation at sowing

Commodity	Country	Form.	F or G	Application				PHI, days	Ref.	Remarks
				Method	Rate kg ai/ha	Spray conc, kg ai/hl	No.			
	Netherlands	EC	F	Spraying	3.84-4.0	0.5-1.92	1	---	Olthof 1996	Soil incorporation at sowing
	UK	EC	F	Overall and soil incorporated spray	2.35 (mineral soils) 4.7 (organic soils)	0.235-0.94 or 0.47-1.88	1	---	Anon 1996e	Pre-planting
	UK	EC	F	Overall spray	2.35	0.235-0.39	1-2	21	Anon 1996e	Post-emergence[2]
Parsnip	Netherlands	WP	F	Spraying	3.84-4.0	0.4-1.92	1	---	Olthof 1996	Soil incorporation directly after treatment
	Netherlands	EC	F	Spraying	3.84-4.0	0.4-1.92	1	---	Olthof 1996	Soil incorporation directly after treatment
	UK	EC	F	Overall and soil incorporated spray	2.35 (mineral soils)4.7 (organic soils)	0.235-0.94 or 0.47-1.88	1	---	Anon 1996e	Pre-planting
	UK	EC	F	Overall spray	2.35	0.235-0.39	1-2	21	Anon 1996e	Post-emergence[2]
	UK	GR	F	Broadcast incorporated	2.25 (mineral soils), 4.5 (organic soils)	----	1	21	Anon 1996e	Pre-planting
Potato, seed, starch, ware	Netherlands	WP/ EC	F	Spraying of aerial parts	0.120-0.125	0.0208-0.06	1	14	Olthof 1996	At larvae infestation
	Poland	44% EC	F	High volume spray	220-330 ml/ha	---	1-2	14	Anon 1996a	
Radish, long	Germany	GR	F / G	Spreading	3 kg/ha (field) 4 kg/ha (glass)	-----	1	---	Anon 1996d	Before sowing and below/after planting
, small	Germany	GR	F / G	Spreading	3 kg/ha (field) 4 kg/ha (glass)	-----	1	---	Anon 1996d	
	Netherlands	WP/ EC/ GR	F	Soil incorporation before sowing	2.88-3.0	0.375-1.44	1	---	Olthof 1996	
, black	Netherlands	WP/ EC/ GR	F	Soil incorporation before sowing	2.88-3.0	0.375-1.44	1	---	Olthof 1996	
	UK	EC	F	Overall and incorporated spray	2.35	0.47-0.94	1	21	Anon 1996e	Pre-planting
Salsify	UK	EC	F	Overall and soil incorporated spray	2.35 (mineral soils) 4.7 (organic soils)	0.235-0.94 or 0.47-1.88	1	---	Anon 1996e	Pre-planting
	UK	EC	F	Overall spray	2.35	0.235-0.39	1-2	21	Anon 1996e	Post-emergence[2]
	UK	GR	F	Broadcast incorporated	2.25 (mineral soils), 4.5 (organic	----	1	21	Anon 1996e	Pre-planting

Commodity	Country	Form.	F or G	Application				PHI, days	Ref.	Remarks
				Method	Rate kg ai/ha	Spray conc, kg ai/hl	No.			
					soils)					
Shallots	Netherlands	WP/ EC/ GR	F	Spraying or granular broadcast application of soil	5.76-6.0	0.75-2.88	1	---	Olthof 1996	Before sowing
	Netherlands	GR	F	Granular application of soil in furrow	1.2	----	1	---	Olthof 1996	Incorp. before sowing
Swede	Netherlands	WP/ EC/ GR	F	Soil treatment followed by incorporation	2.88-3.0	0.375-1.44	1	---	Olthof 1996	Before sowing
	UK	EC	F	Overall soil incorporated spray	2.35	0.47-0.78	1	---	Anon 1996e	Applied immediately before drilling
	UK	EC	F	Band spray in furrow	2.35	----	---		Anon 1996e	Pre-emergence
	UK	EC	F	Overall post-emergence spray	0.72	0.12	2	21	Anon 1996e	1st application July/August, 2nd application 14 days later
	UK	GR	F	Band application incorporated	4.5	----	1	21	Anon 1996e	Post and pre-emergence
Turnip	Netherlands	WP/ EC/ GR	F	Soil treatment followed by incorporation	2.88-3.0	0.375-1.44	1	---	Olthof 1996	
	UK	EC	F	Overall soil incorporated spray	2.35	0.47-0.78	1		Anon 1996e	Applied immediately before drilling
	UK	EC	F	Band spray in furrow	2.35	----	---		Anon 1996e	Pre-emergence
	UK	EC	F	Overall spray in furrow	0.72	0.12	2	21 (pre-emergence)	Anon 1996e	1st application July/August, 2nd application 14 days later
	UK	GR	F	Band application in furrow incorporated	4.5	----	1	21 (pre-emergence)	Anon 1996e	

F = Field G = Glasshouse

[1] Application rate calculated from estimated l/ha
[2] Calculated from 0.05 g/plant
[3] For lifting October/November apply 1st week August, for lifting December or later apply 1st week August and repeat 4-6 weeks later (according to advice or pest level)
[4] Application rate appears high but is as stated by the manufacturer

Table 17. Registered uses of chlorfenvinphos on oilseeds and cereals.

Commodity	Country	Form	F or G	Application				PHI, days	Ref.	Remarks
				Method	Rate, kg ai/ha	Spray conc, kg ai/hl	No.			
Maize	Netherlands	WP or EC	F	Spraying of aerial parts at infestation	0.48-0.50	0.08-0.24	1	42 For cutting maize.	Olthof 1996	Application if and when the attack is expected in the 2-3 leaf stage of the crop.
, regrowth of potatoes in maize crop	Netherlands	WP or EC	F	Spraying of aerial parts at infestation	0.120-0.125	0.02-0.06	1	42 For cutting maize	Olthof 1996	Application if larvae of the Colorado beetle have the size of a wheat grain
Rape seed	Austria	EC	-	----------	0.15	----------	---	21	Anon 1996c	Treatment when pests occur
	Germany	EC	-	----------	0.14	----------	---	56	Anon 1996c	Treatment at infestation
	Germany	EC	F	Spraying	0.144	0.024	1	56	Anon 1996d	Treatment at infestation
	Netherlands	GR	-	Broadcast	3	----------	---	60	Anon 1996c	
, winter	Poland	44% EC	F	High volume spray	440 ml/ha	----------	1	35	Anon 1996a	Pest, ceutor-rhynchid beetle
	Poland	44% EC	F	High volume spray	330-400 ml/ha	----------	1	35	Anon 1996a	Pest, Pollen beetle
Rye and triticale	UK	EC	F	Overall soil incorporated spray	1.34	0.39-0.59	1	21[1]	Anon 1996e	Pre-planting
	UK	EC	F	Overall spray	1.01	0.29-0.44	1	21[1]	Anon 1996e	Autumn application after planting
	UK	EC	F	Overall spray	0.67 or 1.34 on organic soils	0.19-0.27 or 0.39-0.59	1	21[1]	Anon 1996e	Application at egg hatch of pest normally Jan/Feb
	UK	EC	F	Conventional seed treatment machine	---------	966 g ai /tonne seed	1		Anon 1996e	Pre-planting
Wheat ...., winter	UK	EC	F	Overall soil incorporated spray	1.34	0.39-0.59	1	21[1]	Anon 1996e	Pre-planting
...., winter	UK	EC	F	Overall spray	1.01	0.29-0.44	1	21[1]	Anon 1996e	Autumn application after planting
...., winter	UK	EC	F	Overall spray	0.67 or 1.34 on organic soils	0.19-0.27 or 0.39-0.59	1	21[1]	Anon 1996e	Application at egg hatch of pest normally Jan/Feb
...., winter	UK	LS	F	Conventional seed treatment machine	966 g ai/tonne seed		1	----------	Anon 1996e	
, durum	UK	EC	F	Overall soil incorporated spray	1.34	0.39-0.59	1	21[1]	Anon 1996e	Pre-planting
, durum	UK	EC	F	Overall spray	1.01	0.29-0.44	1	21[1]	Anon 1996e	Autumn application after

Commodity	Country	Form	F or G	Application				PHI, days	Ref.	Remarks
				Method	Rate, kg ai/ha	Spray conc, kg ai/hl	No.			
										planting
, durum	UK	EC	F	Overall spray	0.67 or 1.34 on organic soils	0.19-0.27 or 0.39-0.59	1	21[1]	Anon 1996e	Application at egg hatch of pest normally Jan/Feb
, durum	UK	EC	F	Conventional seed treatment machine	---------	966 g ai /tonne seed	1	----------	Anon 1996e	

[1] This 21-day interval which is currently stated on the UK notices of approval for use on winter wheat is shorter than that required in practice. The latest time of application in wheat would be March and the earliest time of harvest July

Table 18. Registered topical uses of chlorfenvinphos on livestock in Australia.

Animal	Application				Ref.	Remarks
	Form.	Method	Spray or dip conc, kg ai/hl	No.		
Cattle (cattle ticks, buffalo fly and lice), Horses, deer, goats, sheep and dogs may also be treated	138 g/l liquid	Plunge dip or spray	0.0552	Used at 19-21 day intervals	Anon 1996b	Treat in early Autumn when infestations first occur

The use of chlorfenvinphos on roses in The Netherlands was also reported (Olthof, 1996).

RESIDUES RESULTING FROM SUPERVISED TRIALS

The results of the residue trials are given in Tables 19-39. They were carried out under field conditions and reported in sufficient detail with acceptable analytical information unless otherwise indicated. Where analytical recoveries were outside the range 70-120% and/or where samples were stored for longer than 6 months or for an unspecified time this is indicated in a footnote. Analytical results have generally been rounded to one significant figure for residues below 0.1 mg/kg. Data in the JMPR format were submitted by the manufacturer only for carrots (some results), onions, kale, cabbage, cauliflower and rape seed.

Many of the trials were very old with reports which lacked details such as the method of analysis, duration of sample storage, recovery data and plot size.

The trials which were considered unsatisfactory have been identified by shading in the Tables. The acceptability of the results of some other trials in which the duration of sample storage was not reported will depend on the future availability of satisfactory data on the stability of residues in representative stored samples.

In most of the trials the samples were analysed for 1-(2,4-dichlorophenyl)ethanol, identified in the Tables as “met”. Several of the trials also included analyses for 2,4-dichlorophenacyl chloride and 2,4-dichoroacetophenone, but the residues were below the LODs of 0.02 mg/kg and 0.05 mg/kg respectively in all the analysed samples. Residues discussed in the text are parent chlorfenvinphos unless otherwise indicated.

Where residues of the (*E*)- and (*Z*)- isomers were originally reported separately their sum is given in the Tables. The limit of determination of the individual isomers reported in the studies was generally 0.01 mg/kg.

Leeks. GAP for leeks was reported for Germany and The Netherlands. The maximum application rates were 0.144 kg ai/ha at infestation and 6 kg ai/ha pre-sowing, with PHIs of 28 and 60 days respectively.

One trial was available from Germany. It was poorly reported and did not reflect the reported GAP.

Table 19. A supervised field trial on leeks in Germany (undated).[1]

Application				PHI, days	Portion analysed	Residues, mg/kg Parent Met	Ref.
Form.	No.	kg ai/ha	kg ai/hl				
GR	1	3	-	150	stem	ND ND	CH-601-001

[1] No detailed study report; only very brief details of the trial and analysis were available.
Met = 1-(2,4-dichlorophenyl)ethanol

Onions. GAP was reported for a number of countries. The maximum application rates were 1-13.5 kg ai/ha with PHIs between 7 and 60 days or as governed by pre-planting, pre-sowing or post-emergence treatments.

Residue trials on bulb onions were reported from Canada, France, Germany, Japan, Switzerland, Spain, the USA and the UK, as well as one trial on spring onions from The Netherlands. The application rates in four French trials with residues of <0.02 mg/kg were comparable with the granular application rates in France, but a PHI of 15 days was reported by the manufacturer as French GAP whereas the PHIs in the trials were 133-182 days. One German trial (CH-722-007), with a PHI of 175 days, was comparable with the Belgian and Netherlands GAP for pre-planting spray treatment. The residues were <0.02 mg/kg after 175 days. A further five German trials with granules were considered to accord with pre-planting GAP in Belgium, Denmark, Germany and The Netherlands. All showed residues below the LOD (<0.02 mg/kg). Two replicated Japanese trials reflected Japanese foliar GAP (which has a low application rate) with residues of <0.02 mg/kg 7-8 days after treatment. The only measurable residues of the parent reported were at the higher application rates of 4.8 kg ai/ha in a German spray trial (0.04 mg/kg, 60-day PHI) and 4.48 kg ai/ha in a UK trial (0.07 mg/kg, PHI 61 days) which was poorly reported with no detailed study report. These PHIs imply that the crops were immature and hence that the trials were not comparable with any reported GAP.

Table 20. Supervised field trials on bulb and spring onions. Bulbs analysed.

Location, Country, year	Application				PHI, days	Residues, mg/kg Parent Met	Reference
	Form	No.	kg ai/ha	kg ai/hl			
Bulb onions							
Guelph Canada 1969[1]	GR	1	1.1	-	154	<0.02 ----- <0.02 ----- <0.02 ----- <0.02 ----- <0.02 ----- <0.02 ----- <0.02 ----- <0.02 -----	CH-722 -002

Location, Country, year	Application				PHI, days	Residues, mg/kg Parent	Met	Reference
	Form	No.	kg ai/ha	kg ai/hl				
Althen les Paluds S. France 1969[1]	GR	1	5	-	182	<0.02	-----	CH-722 -003
	GR	1	5	-	182	<0.02	-----	
	GR	1	6	-	182	<0.02	-----	
Le Thor S. France 1969[1]	GR	1	5	-	168	<0.02	-----	CH-722 -003
	GR	1	5	-	168	<0.02	-----	
	GR	1	6	-	168	<0.02	-----	
	GR	1	5	-	154	<0.02	-----	
	GR	1	5	-	154	<0.02	-----	
	GR	1	5	-	154	<0.02	-----	
	GR	1	6	-	154	<0.02	-----	
Le Thor S. France 1971[1]	GR	1	4	-	133	<0.02	<0.02	CH-790 -029
	GR	1	8	-	133	<0.02	<0.02	
Le Thor S. France 1972[1]	GR	2	4	-	175	<0.02	<0.02	CH-790 -031
	GR	2	8	-	175	<0.02	<0.02	
Baden Germany 1973[1]	EC	1	4.8	-	60	0.04	<0.02	CH-722 -007
München Germany 1973[1]	EC	1	4.8	-	56	<0.02	-----	CH-722 -007
					74	<0.02	<0.02	
Frankfurt Germany 1973[1]	EC	1	4.8	-	49	0.39	-----	CH-722 -007
					70	0.08	-----	
					175	<0.02	<0.02a	
Baden Germany 1973[1]	WP	1	seed treatment 25 g ai/kg seed	-	42	<0.02	-----	CH-722 -008
					56	<0.02	<0.02	
Freising Germany 1973[1]	WP	1	seed treatment 25 g ai/kg seed	-	49	<0.02	-----	CH-722 -008
					77	<0.02	-----	
					126	<0.02	<0.02	
Fischenich Germany 1973[1]	WP	1	seed treatment 25 g ai/kg seed	-	91	<0.02	-----	CH-722 -008
					112	<0.02	-----	
					133	<0.02	-----	
					161	<0.02	<0.02	
Baden Germany 1973[1]	GR	1	5	-	42	0.70	-----	CH-722 -009
					60	<0.02b	<0.02	
Frankfurt Germany 1973[1]	GR	1	5	-	49	1.37	-----	CH-722 -009
					70	0.21	-----	
					175	<0.02	<0.02b	
Freising Germany 1973[1]	GR	1	5	-	49	0.72	-----	CH-722 -009
					77	<0.02	-----	
					147	<0.02	<0.02b	
Frankfurt Germany[1]	GR	1	5.0	-	86	<0.02	-----	CH-722 -013
					100	<0.02	-----	

Location, Country, year	Application				PHI, days	Residues, mg/kg Parent	Met	Reference
	Form	No.	kg ai/ha	kg ai/hl				
					114	≤0.02b	-----	
Bonn Bad Godesberg Germany[1]	GR	1	5.0	-	35	<0.02	-----	CH-722-013
					69	<0.02	-----	
					83	≤0.02b	-----	
Bad Segeberg Germany[1]	GR	1	5.0	-	55	<0.02	-----	CH-722-013
					69	<0.02	-----	
					83	≤0.02b	-----	
Germany 1965[1]	GR	1	3	-	120	<0.02	-----	CH-722-001
Chuo Japan 1972[1]	EC	5	0.32	0.032	8	≤0.02	<0.02c	CH-722-005
					8	≤0.02	<0.02c	
					14	<0.02	<0.02	
					14	≤0.02	<0.02c	
	EC	9	0.32	0.032	8	≤0.02	<0.02c	
					8	≤0.02	<0.02c	
					14	≤0.02	<0.02c	
					14	≤0.02	<0.02c	
Kimitami Japan 1972[1]	EC	6	0.32	0.032	7	≤0.02	<0.02c	CH-722-005
					7	≤0.02	<0.02c	
					14	≤0.02	<0.02c	
					14	≤0.02	<0.02c	
	EC	9	0.32	0.032	7	≤0.02	<0.02c	
					7	≤0.02	<0.02c	
					14	≤0.02	<0.02c	
					14	≤0.02	<0.02c	
Seville Spain 1971[1]	GR	1	2	-	133	<0.02	0.01	CH-722-004
	GR	1	3	-	133	<0.02	0.02	
	GR	1	4	-	133	<0.02	0.03	
Seville Spain 1972[1]	GR	1	4	-	140	<0.02	<0.02	CH-722-006
	GR	1	8	-	140	<0.02	<0.02	
Seville Spain 1973[1]	GR	1	4	-	140	<0.02	<0.02	CH-722-010
	GR	2	8	-	140	<0.02	<0.02	
Seville Spain 1974[1]	GR	1	4	-	175	<0.02	-----	CH-722-011
	GR	1	8	-	175	<0.02	-----	
	GR	1	4	-	175	<<0.02	-----	
	GR	1	8	-	175	<0.02	-----	
UK undated[2]	pure ai	1	4.48	- -	61	0.07	----	CH-601-001
USA undated[2]	GR	1	2.8	-	72	<0.05	----	CH-601-001
Switzerland undated[2]	EC	1	1	-	31	<0.02	----	CH-601-001

Spring onions							
Alkmaar Netherlands 1982[3]	GR	1	6	-	90	0.01 ----- 0.01 ----- 0.04 ----- 0.03 ----- 0.04 -----	J. W. Dornseiffen 1985

Results underlined once or twice are considered comparable with
a - Belgian and Netherlands GAP for spray treatments
b - GAP in Belgium, Denmark, Germany and The Netherlands for pre-planting granular treatments
c - Japanese GAP for foliar treatments
Double underlined residues are from maximum GAP treatments and have been used for estimating the STMR

[1] Duration of sample storage unspecified
[2] No detailed study report; only very brief details of the trial and analysis were available
[3] Information is taken from residue trial summary sheets submitted by The Netherlands. Full study reports were submitted but were in Dutch

Met = 1-(2,4-dichlorophenyl)ethanol

Head cabbage. GAP was reported for Belgium, Denmark, France, Germany, Ireland, Italy, Japan, The Netherlands, Sweden, Switzerland, and the UK. The maximum application rates were 0.96-6 kg ai/ha with PHIs of 14-70 days or as governed by pre-planting or post-emergence treatment .

Residue trials were available from the UK, Germany, the USA and India. In 7 German trials complying with German GAP at 100 g/m^2 all residues were <0.02 mg/kg. In 6 more German trials reflecting German GAP for granular seedbed treatment (2 g/100 plants) residues were again all <0.02 mg/kg. Residues of 0.07 mg/kg and 0.02 mg/kg were found in two Indian trials in samples taken 17 and 11 days after treatment, but no Indian GAP was reported. One UK trial was considered comparable with the UK pre-emergence spray GAP, but it was poorly reported with few details. No trials were considered to comply with GAP for foliar treatments, which have shorter PHIs.

Table 21. Supervised field trials on head cabbages. Heads analysed.

Location, Country, year	Application				PHI, days	Residues, mg/kg Parent Met	Ref.
	Form.	No.	kg ai/ha	kg ai/hl			
Wellesbourne UK 1965[1]	EC	1	0.84	-	0 4 10 20	4.2 ----- 2.89 ----- 0.29 ----- <0.02 -----	CH-640-002
Unknown UK undated[1]	GR GR	1 1	4.48 8.96	- -	112 112	<0.05 ---- <0.05 ----	CH-601-001
Unknown USA undated[1]	GR	1	0.52kg/1000 m row	-	77	<0.05 ----	CH-601-001
Geisenheim Germany 1980[2]	GR	1	0.1kg/m^2	-	63 74 94	0.2 ----- 0.05 ----- <u><u>≤0.02a</u></u> -----	CH-721 -014
Bamberg Germany 1980[2]	GR	1	0.1kg/m^2	-	70 84 98	0.3 ----- 0.10 ----- <u><u>≤0.02a</u></u> -----	CH-721 -014
Frankfurt Germany 1980[2]	GR	1	0.1kg/m^2	-	70 84 98	0.4 ----- 0.2 ----- <u><u>≤0.02a</u></u> -----	CH-721 -014
Frankfurt Germany 1989[2]	GR GR	1 1	100g/m^2 2g/100 plants	- -	144 180 190 144	<0.02 ----- <0.02 ----- <u><u>≤0.02a</u></u> ----- <0.02 -----	CH-721 -018

Location, Country, year	Application				PHI, days	Residues, mg/kg Parent Met	Ref.
	Form.	No.	kg ai/ha	kg ai/hl			
					180	<0.02 -----	
					190	<u>≤0.02b</u> -----	
			0.14				
	EC	1		-	0	0.1 -----	
					16	<0.02 -----	
					21	<0.02 -----	
					28	<0.02 -----	
					35	<0.02 -----	
Bonn	GR	1	100g/m^2	-	82	<0.02 -----	CH-721
Germany					103	<0.02 -----	-018
1989[2]					113	<u>≤0.02a</u> -----	
	GR	1	2g/100 plants	-	70	<0.02 -----	
					86	<0.02 -----	
					96	<u>≤0.02b</u> -----	
			2g/100 plants				
	GR	1		-	105	<0.02 -----	
					129	<0.02 -----	
			0.14		139	<u>≤0.02b</u> -----	
	EC	1		-	0	1.0 -----	
					14	0.01 -----	
					21	<0.02 -----	
					28	<0.02 -----	
					35	<0.02 -----	
München	GR	1	100g/m^2	-	108	<0.02 -----	CH-721
Germany					126	<0.02 -----	-018
1989[2]					136	<u>≤0.02a</u> -----	
	EC	1	0.14	-	0	0.3 -----	
					14	<0.02 -----	
					21	<0.02 -----	
					28	<0.02 -----	
					35	<0.02 -----	
Hannover	GR	1	100g/m^2	-	107	<0.02 -----	CH-721
Germany					121	<0.02 -----	-018
1989[2]					132	<u>≤0.02a</u> -----	
Poona	EC	1	0.25	-	17	<0.02 -----	CH-721
India							-002
1974[2]	EC	1	0.5	-	17	0.07 -----	
Holibazar	EC	3	0.25	-	11	<0.02 -----	CH-721
India							-002
1974[2]	EC	3	0.50	-	11	0.02 -----	
Geisenheim	GR	2	0.1kg/m^2	-	30	10.1 0.05	CH-721
Germany			and		50	1.6 <0.02	-008 & CH-
1978[2]			0.1g/plant	-	60	<u>0.9c</u> <0.02	721-010
München	EC	2	0.144	0.024	0	1.2 -----	CH-721
Germany					14	<0.02 -----	-032
1990[3]					21	<0.02 -----	
					28	<0.02 -----	
					35	<0.02 -----	
Bonn	EC	2	0.144	0.024	0	0.07 -----	CH-721
Germany					14	<0.02 -----	-033
1990[3]					21	<0.02 -----	

Location, Country, year	Application				PHI, days	Residues, mg/kg Parent Met	Ref.
	Form.	No.	kg ai/ha	kg ai/hl			
					28 35	<0.02 ----- <0.02 -----	
Buttelborn Germany 1990[3]	EC	2	0.144	0.024	0 14 21 28 35	0.6 ----- <0.02 ----- <0.02 ----- <0.02 ----- <0.02 -----	CH-721 -033
Frankfurt Germany 1990[3]	GR GR	1 1	2g/100 plants 2g/100 plants	-	65 98 108 60 84 98	<0.02 ----- 0.04 ----- <u>≤0.02b</u> ----- <0.02 ----- <0.02 ----- <u>≤0.02b</u> -----	CH-721 -034
Bonn Germany 1990[3]	GR	1	2g/100 plants	-	55 99 109	<0.02 ----- <0.02 ----- <u>≤0.02b</u> -----	CH-721 -034
Munich Germany 1990[3]	GR	1	2g/100 plants	-	42 56 66	<0.02 ----- <0.02 ----- <u>≤0.02b</u> -----	CH-721 -034
Hannover Germany 1990 [3]	GR	1	2g/100 plants	-	64 80 90	<0.02 ----- <0.02 ----- <u>≤0.02b</u> -----	CH-721 -034
Bonn Germany 1990[3]	GR	1	0.1	-	80 114 124	<0.02 ----- <0.02 ----- <0.02 -----	CH-721 -035
Frankfurt Germany 1990[3]	GR	1	0.1	-	100 144 154	<0.02 ----- <0.02 ----- <0.02 -----	CH-721 -035
Hannover Germany 1990[3]	GR	1	0.1	-	97 113 123	<0.02 ----- <0.02 ----- <0.02 -----	CH-721 -035
München Germany 1990[3]	GR	1	0.1	-	89 103 113	<0.02 ----- <0.02 ----- <0.02 -----	CH-721 -035

Results underlined once or twice are considered comparable with

a - German GAP for pre-planting soil treatments at 100 g/m^2

b - German GAP for granular treatments at 2 g/100 plants

c - German GAP for granular nursery bed treatment at 0.1 g/plant in combination with pre-planting soil treatment at 100 g/m^2

Double underlined residues are from maximum GAP treatments and have been used for estimating the STMR

[1] No detailed study report; only very brief details of the trial and analysis were available.

[2] Duration of sample storage unspecified

[3] Report not in English

Met = 1-(2,4-dichlorophenyl)ethanol

<u>Savoy cabbage</u>. GAP was reported for Germany and The Netherlands. A variety of treatment regimes are used although all applications are either before or soon after planting.

Only Germans trials were submitted. The German soil treatment at 0.1kg ai/m^2 was reflected by three trials, with all residues <0.02 mg/kg. The 0.1 g/plant granular treatment was used in 3 acceptable trials with residues of 0.02, 0.03 and 0.15 mg/kg. In one additional trial a combination of these two treatments gave a residue of 0.3 mg/kg. In three trials with the German 2 kg ai/ha GAP application all residues were <0.02 mg/kg.

Table 22. Supervised field trials on Savoy cabbage in Germany. Heads analysed.

Location, year	Application				PHI, days	Residues, mg/kg Parent Met	Ref.
	Form	No.	kg ai/ha	kg ai/hl			
München 1973[1]	EC	1	4.8	-	35 49 56	<0.02 ----- <0.02 ----- <0.02 <0.02	CH-721 -003
Baden 1973[1]	EC	1	4.8	-	49 59	<0.02 ----- <0.02 <0.02	CH-721 -003
Kiel 1973[1]	EC	1	4.8	-	0 10 28	33.3 ----- 1.0 ----- 0.3 0.04	CH-721 -003
Geisenheim 1977[1]	EC	1	4.8	-	40 60 80	0.2 <0.02 0.03 <0.02 <0.02 <0.02	CH-721 -004 & CH-721-005
Frankfurt 1977[1]	EC		4.8	-	30 50 63	0.04 <0.02 <0.02 <0.02 <0.02 <0.02	CH-721 -004 & CH-721-005
Bamberg 1977[1]	EC	1	4.8	-	40 60 80	0.02 <0.02 <0.02 <0.02 <0.02 <0.02	CH-721 -004 & CH-721-005
Geisenheim 1980[1]	EC	1+ 2	4.88 + 1.4	-	0 7 14 21 28	2.9 ----- 0.2 ----- 0.04 ----- <0.02 ----- <0.02 -----	CH-721 -012
Bamburg 1980[1]	EC	1+ 2	4.88 + 1.4	-	0 7 14 21 28	4.5 ----- 0.7 ----- 0.3 ----- 0.08 ----- <0.02 -----	CH-721 -012
Geisenheim 1980[1]	GR	1	2	-	49 56 77	0.03 ----- <0.02 ----- <u><0.02d</u> -----	CH-721 -015
	GR	1	0.1kg/m^2	-	49 56 77	0.08 ----- 0.03 ----- <u><0.02a</u> -----	
	GR	1	4 g/200 plants	-	49 56 77	0.2 ----- 0.03 ----- <0.02 -----	
Bamberg 1980[1]	GR	1	2	-	49 63 77	0.09 ----- <0.02 ----- <u><0.02d</u> -----	CH-721 -015
	GR	1	0.1 kg/m^2	-	49 63 77	0.2 ----- 0.05 ----- <u><0.02a</u> -----	

Location, year	Application				PHI, days	Residues, mg/kg Parent Met	Ref.
	Form	No.	kg ai/ha	kg ai/hl			
	GR	1	4 g/200 plants	-	49 63 77	0.3 ----- 0.05 ----- <0.02 -----	
Frankfurt 1980[1]	GR	1	2	-	49 63 77	0.3 ----- 0.04 ----- <u><0.02d</u> -----	CH-721 -015
	GR	1	0.1 kg/m^2	-	49 63 77	0.3 ----- 0.05 ----- <u><0.02a</u> -----	
	GR	1	4 g/200 plants	-	49 56 77	0.4 ----- 0.03 ----- <0.02 -----	
Bad Segeberg 1981[1]	EC	1+ 2	4.9 + 0.17	-	0 7 14 21	0.9 ----- 0.4 ----- 0.2 ----- 0.1 -----	CH-721 -017
Vorwohle 1981[1]	EC	1+ 2	4.9 + 0.17	-	0 7 14 21	0.5 ----- 0.07 ----- <0.02 ----- <0.02 -----	CH-721 -017
Hannover 1986[2]	GR	1	0.1 g/plant	-	40 60 81	0.2 ---- 0.1 ---- 0.08 ----	Anon 1995
Saarlouis 1986[2]	GR	1	0.1 g/plant	-	40 60 80	0.4 ---- 0.03 ---- 0.05 ----	Anon 1995
Frankfurt 1986[2]	GR	1	0.1 g/plant	-	40 60 81	0.07 ---- <0.02 ---- <0.02 ----	Anon 1995
Berlin 1986[2]	GR	1	0.1 g/plant	-	105 124 145	0.06 ---- 0.05 ---- <0.02 ----	Anon 1995
Bonn 1986[2]	GR	1	0.1 g/plant	-	40 60 80	0.1 ---- 0.06 ---- <0.02 ----	Anon 1995
Lübeck 1986[2]	GR	1	0.1 g/plant	-	38 63 83	0.3 ---- 0.2 ---- 0.07 ----	Anon 1995
München 1986[2]	GR	1	0.1 g/plant	-	40 60 80	0.74 ---- 0.06 ---- 0.01 ----	Anon 1995
Münster 1986[2]	GR	1	0.1 g/plant	-	42 63 84	0.2 ---- 0.02 ---- 0.01 ----	Anon 1995
Braunschweig 1986[2]	GR	1	0.1 g/plant	-	39 60 80	0.2 ---- 0.04 ---- 0.08 ----	Anon 1995
Stuttgart 1986[2]	GR	1	0.1 g/plant	-	40 60 80	0.34 ---- 0.03 ---- <0.02 ----	Anon 1995
Geisenheim 1977[1]	GR	1	0.1 g/plant	-	40 60 80	2.03 <0.02 0.14 <0.02 <u>0.03b <0.02</u>	CH-721 -006 & CH-721-007

Location, year	Application				PHI, days	Residues, mg/kg Parent Met	Ref.
	Form	No.	kg ai/ha	kg ai/hl			
Frankfurt 1977[1]	GR	2	0.1 kg/m^2 and 0.1 g/plant	-	30 50 63	3.4 <0.02 0.25 <0.02 0.15c <0.02	CH-721 -006 & CH-721-007
	GR	1	0.1 g/plant	-	30 50 63	0.9 <0.02 0.20 <0.02 0.15b <0.02	
Bamberg 1977[1]	GR	2	0.1 kg/m^2 and 0.1 g/plant	-	40 60 80	0.4 <0.02 0.1 <0.02 0.02c <0.02	CH-721 -006 & CH-721 -007
	GR	1	0.1 g/plant	-	40 60 80	0.4 <0.02 0.1 <0.02 0.02b <0.02	
Geisenheim 1978[1]	GR	2	0.1 kg/m^2 and 0.1 g/plant	-	30 50 60	3.1 0.02 0.4 <0.02 0.3c <0.02	CH-721 -009 & CH-721-011

Results underlined once or twice are considered comparable with
a - the German 0.1 kg ai/m^2 soil treatment
b - the German 0.1 g/plant granular nursery bed treatment
c - a combination of the German 0.1 kg ai/m^2 soil treatment and 0.1 g/plant granular treatment
d - the German 2 kg ai/ha treatment 5-6 days after planting
Double underlined residues are from maximum GAP treatments and have been used for estimating the STMR

[1] Duration of sample storage unspecified
[2] Only the JMPR residue trial summary sheets were supplied, no study report with further trial and analytical information

Met = 1-(2,4-dichlorophenyl)ethanol

Cauliflower. GAP was reported for Germany, Ireland, The Netherlands and the UK. Application is usually pre-emergence or at planting although post-emergence application is allowed in the UK and Ireland.

Residue trials were reported from Germany, India, the USA and the UK. There were three German trials according to each of three different German GAP treatments: 2 g/100 plants nursery granular, the 0.1 g/plant single bed treatment and the 2 kg ai/ha granular "spreading" application. The UK and Dutch spray treatment (ca. 4-5 kg ai/ha) at the time of drilling or transplanting was reflected by four German trials. All the residues in these trials were <0.02 mg/kg.

Table 23. Supervised field trials on cauliflower. Heads analysed.

Location, Country, year	Application				PHI, days	Chlorfenvinphos, mg/kg	Ref.
	Form	No.	kg ai/ha	kg ai/hl			
Frankfurt Germany 1980[1]	EC	1+2	4.8+ 0.14	- -	0 7 14 28	<0.02 <0.02 <0.02 <0.02	CH-721 -022
Geisenheim Germany 1980[1]	GR	1	4 g/200 plants	-	49 77 84 91	0.10 <0.02 <0.02 <u><0.02</u>a	CH-721 -023
	GR	1	0.1 g/ plant	-	49 77 84 91	0.5 <0.02 <0.02 <u><0.02</u>b	
	GR	1	2	-	49 77 84 91	0.1 <0.02 <0.02 <u><0.02</u>c	
Bamberg Germany 1980[1]	GR	1	4 g/200 plants	-	70 77 84	<0.02 <0.02 <u><0.02</u> a	CH-721 -023
	GR	1	0.1 g/ plant	-	70 77 84	<0.02 <0.02 <u><0.02</u>b	
	GR	1	2	-	70	<0.02	

Location, Country, year	Application				PHI, days	Chlorfenvinphos, mg/kg	Ref.
	Form	No.	kg ai/ha	kg ai/hl			
					77 84	<0.02 <u>≤0.02c</u>	
Frankfurt Germany 1980[1]	GR	1	4 g/200 plants	-	49 77 84 91	0.3 <0.02 <0.02 <u>≤0.02a</u>	CH-721 -023
	GR	1	0.1 g/ plant	-	49 77 84 91	1.9 0.02 <0.02 <u>≤0.02b</u>	
	GR	1	2	-	49 77 84 91	0.4 <0.02 <0.02 <u>≤0.02c</u>	
Bad Segeberg Germany 1981[1]	EC	1+ 2	4.9 + 0.17	-	0 7 14 21	1.0 0.1 0.05 0.07	CH-721 -024
Vorwohl Germany 1981[1]	EC	1+2	4.9 + 0.17	-	0 7 14 21	0.80 0.10 0.06 <0.02	CH-721 -024
Frankfurt Germany 1989[1]	EC	2	0.144	0.019	0 14 21 28	<0.02 <0.02 <0.02 <0.02	CH-721 -025
	EC	1	4.8	1.2	119 126 140	<0.02 <0.02 <u>≤0.02d</u>	
Bonn Germany 1989[1]	EC	1	4.8	1.2	91 98 112	<0.02 <0.02 <u>≤0.02</u>d	CH-721 -025
USA undated[2]	GR	1.1 2	1.12	-	20 48	<0.05 <0.05	CH-601-001
USA undated[2]	GR + EC	1+ 3	1.12+ 1.12	- -	 20 48	 1.3 <0.05	CH-601-001
Nasik India 1972[1]	EC EC	3 3	0.25 0.50	- -	7 7	0.1 0.2	CH-721 -019
Wellesbourne UK 1964[1,3]	WP WP EC EC	1 1 1 1	root dip root dip root dip root dip	0.05 0.05 0.1 0.1	88 88 88 88	<0.05 <0.05 <0.05 <0.05	CH-724 -065
Bonn Germany 1990[4]	EC	2	0.144	0.024	0 14 21 28	0.55 0.16 0.06 <0.02	CH-721 -030

Location, Country, year	Application				PHI, days	Chlorfenvinphos, mg/kg	Ref.
	Form	No.	kg ai/ha	kg ai/hl			
					35	<0.02	
Buttelborn Germany 1990[4]	EC	1	4.8	0.48	83 90 104	<0.02 <0.02 <u><0.02</u>d	CH-721 -031
Bonn Germany 1990[4]	EC	1	4.8	0.48	129 136 150	<0.02 <0.02 <u><0.02</u>d	CH-721 -031

Results underlined once or twice are considered comparable with

a - the German 2 g/100 plants nursery granular treatment

b - the German 0.1 g/plant single bed treatment

c - the Germans 2 kg ai/ha granular treatment

d - the UK and Dutch spray treatments (ca. 4-5 kg ai/ha) at time of drilling or transplanting.

Double underlined residues are from maximum GAP treatments and have been used for estimating the STMR

[1] Duration of sample storage unspecified
[2] No detailed study report; only very brief details of the trial and analysis were available.
[3] High analytical recovery (>120%)
[4] Report not in English

<u>Mushrooms</u>. GAP was reported only for the UK as either compost or casing incorporation. Only one trial was available which was poorly described with no detailed study report.

Table 24. Supervised residue trials on protected mushrooms, UK, undated. Fruit analysed.[1]

Application				PHI, days	Chlorfenvinphos, mg/kg	Ref.
Form.	No.	kg ai/ha	kg ai/hl			
GR	1	5 kg/tonne compost	-	30	<0.02	CH-601 -001
GR	1	17 kg/tonne compost	-	30	<0.02	

[1] No detailed study report; only very brief details of the trial and analysis were available

<u>Kale</u>. There are registered uses in Germany, The Netherlands, Portugal and Spain, but residue trials were available only from Germany. Five trials were according to the Dutch GAP for spray treatments at planting or before sowing. Residues were all <0.02 mg/kg. In one of these trials the residue of dichlorophenylethanol was 0.07 mg/kg. Three further trials complied with the German granular single plant treatment, and in two others this treatment was combined with soil treatment according to German GAP. Residues in these trials were <0.02 (2), 0.02, 0.07 and 0.09 mg/kg.

Table 25. Supervised field trials on kale in Germany.[1]

Location, year	Application			PHI, days	Residues, mg/kg Parent	Met	Ref.
	Form	No.	kg ai/ha				
Lübeck 1973	EC	1	4.8	56 63 140	<0.02 <0.02 <u><0.02</u>a	----- ----- <u><0.02</u>	CH-726 -001
Kiel	EC	1	4.8	0	1.58	-----	CH-726

Location, year	Application			PHI, days	Residues, mg/kg Parent	Met	Ref.
	Form	No.	kg ai/ha				
1973				7	0.52	-----	-001
				28	0.13	0.06	
Koldenbuttel 1973	EC	1	4.8	35	<0.02	-----	CH-726
				56	<0.02	-----	-001
				84	<0.02a	0.07	
Geisenheim 1977	EC	1	4.8	40	0.22	<0.02	CH-726
				60	<0.02	<0.02	-002 & CH-726-
				80	<0.02a	<0.02	003
Frankfurt 1977	EC	1	4.8	30	0.08	<0.02	CH-726
				50	<0.02	<0.02	-002 & CH-726-
				63	<0.02a	<0.02	003
Bamberg 1977	EC	1	4.8	40	0.47	<0.02	CH-726
				60	0.15	<0.02	-002 & CH-726-
				80	<0.02a	<0.02	003
Geisenheim 1977	GR	1	0.1 g/plant	40	1.44	<0.02	CH-726
				60	0.37	<0.02	-004 & CH-726-
				80	<0.02b	<0.02	005
Frankfurt 1977	GR	2	0.1 kg/m^2 and 0.1 g/plant	30	3.05	<0.02	CH-726
				50	0.10	<0.02	-004 & CH-726-
				63	0.07c	<0.02	005
	GR	1	0.1 g/plant	30	0.82	<0.02	
				50	0.15	<0.02	
				63	0.09b	<0.02	
Bamberg 1977	GR	2	0.1 kg/m^2 and 0.1 g/plant	40	0.40	<0.02	CH-726
				60	0.20	<0.02	-004 & CH-726
				80	0.02c	<0.02	-005
	GR	1	0.1 g/plant	40	0.71	<0.02	
				60	0.10	<0.02	
				80	<0.02b	<0.02	

Results underlined once or twice are considered comparable with
a - Dutch GAP where treatment is by spraying at or before planting
b - the German granular single plant treatment
c - the German granular single plant combined with soil treatment according to German GAP
Double underlined residues are from maximum GAP treatments and have been used for estimating the STMR

[1] Duration of sample storage was unspecified in all trials

Met = 1-(2,4-dichlorophenyl)ethanol

Carrots. GAP was reported for Belgium, Denmark, France, Germany, Ireland, Italy, Luxembourg, The Netherlands, Switzerland and the UK.

Residue trials were available from Canada, France, Germany, The Netherlands, South Africa, Spain, Sweden, Switzerland, Trinidad and the UK (Table 26). In addition the UK government provided data on residues in overwintered commercial carrots whose treatment history had been recorded (Table 27). The highest residues resulted from post-planting EC or WP sprays at *c.*4 kg ai/ha according to GAP in The Netherlands and France. Similar treatments at *c.*2.5 kg ai/ha are GAP in Ireland and the UK. The PHIs reported for these countries ranged between 21 and 60 days which reflects second-generation carrot fly control. French GAP was also reported to include an EC spray at 5 kg ai/ha with a PHI of 15 days, but the Meeting was informed that the use in practice was at the time of sowing. Several trials in France, Germany and The Netherlands complied with the higher rate GAP, with residues of <0.02, 0.05, 0.08, 0.12, 0.14, 0.2(3), 0.22, 0.3, 0.37, 0.45, 0.9, 1.2, 1.8, 2.0, and 3.8 mg/kg. In the overwintered commercial carrots treated in accordance with UK GAP the residues were <0.02-1.6 mg/kg. The Meeting estimated an STMR of 0.22 mg/kg and a maximum residue level of 5 mg/kg.

Table 26. Supervised field trials on carrots.

Location Country, year	Application				PHI, days	Sample	Residues, mg/kg Parent Met	Ref.
	Form	No.	kg ai/ha	kg ai/hl				
Canada 1970[1]	GR	1	2.2	-	14 147 126	Root Pulp Pulp	<0.02 ------ 0.7 0.04 0.5 0.07	CH-724 -014
Canada 1971[1]	GR	1	1.1	-	112	Root Pulp Boiled	0.1 <0.02 <0.02 <0.02 0.05 <0.02	CH-724 -0.15
	EC	4	3.5		49	Root Pulp Boiled	0.09 <0.02 <0.02 <0.02 0.04 <0.02	
	EC	5	4.6		49	Root Pulp Boiled	0.2 <0.02 <0.02 <0.02 0.08 <0.02	
Surtainville France 1969[1]	GR	1	5.0	-	210	Root	0.01 <0.05 0.02 <0.05	CH-724 -011
Avignon France 1969[1]	GR	1	5.0	-	175	Root	<0.02 <0.05 <0.02 <0.05	CH-724 -011
	GR	1	6.0		175	Root	<0.02 <0.05	
Entraigues	GR	1	5.0	-	294	Root	0.02 <0.05	CH-724

Location Country, year	Application				PHI, days	Sample	Residues, mg/kg Parent Met	Ref.
	Form	No.	kg ai/ha	kg ai/hl				
France 1970[1]							0.01 <0.05	-012
Surtainville France 1970[1]	GR	1	5.0	-	98	Root	0.3 <0.05 0.2 <0.05	CH-724 -012
	GR	1	6.0	-	98	Root	0.4 <0.05	
Le Thor France 1971[1]	GR	1	4.0	-	133	Root Canned Tops and peel	0.1 0.2 <0.02 0.03 0.09 0.05	CH-790 -029
	GR	1	8.0	-	133	Root Canned Tops and peel	0.2 0.25 <0.02 0.03 0.1 0.1	
	GR		4.0	-	133	Root Canned Tops and peel	0.1 0.2 <0.02 0.02 0.1 0.2	
	GR		8.0	-	133	Root Canned Tops and peel	0.2 0.3 <0.02 0.01 0.20 0.3	
Le Thor France 1972[1]	GR	1	4.0	-	504	Root	<0.02 <0.02	CH-790 -031
	GR	1	8.0	-	504	Root	<0.02 <0.02	
	GR	2	4.0	-	175	Root	0.03 0.03	
	GR	2	4.0	-	175	Juice Pulp	<0.02 <0.02 <0.02 <0.02	
	GR	2	8.0	-	175	Root	0.07 0.10	
	GR	2	8.0	-	175	Juice Pulp	<0.02 <0.02 <0.02 <0.02	
	GR	1	4.0	-	504	Root	<0.02 <0.02	
	GR	1	8.0	-	504	Root	<0.02 <0.02	
	GR	2	4.0	-	175	Root	0.06 0.1	
	GR	2	4.0	-	175	Juice Pulp	<0.02 <0.02 <0.02 <0.02	
	GR	2	8.0	-	175	Root	0.1 0.2	
	GR	2	8.0	-	175	Juice Pulp	<0.02 <0.02 <0.02 <0.02	
Le Thor France 1973[1]	GR	3	4.0	-	175	Root	0.02 <0.02	CH-790 -033
	GR	3	8.0	-	175	Root	0.03 0.04	
	GR	3	8.0	-	175	Root	0.02 0.07	

Location Country, year	Application				PHI, days	Sample	Residues, mg/kg Parent Met	Ref.
	Form	No.	kg ai/ha	kg ai/hl				
Frankfurt Germany 1973[1,2]	EC	1	5.0	-	49 77 168	Root Root Root	3.1 ----- 1.5 ----- 0.1 <0.02	CH-724 -017
	EC	1	5.0	-	42 63 112	Root Root Root	1.2 ----- 0.3 ----- <0.02 <0.02	
Frankfurt Germany 1973[1]	GR	1	5.0	-	42 56 112	Root Root Root	0.4 ----- 0.1 ----- <0.02 <0.02	CH-724 -018
	GR	1	5.0	-	49 77 168	Root Root Root	8.8 ----- 1.4 ----- <0.02 <0.02	
Lübeck Germany 1973[1]	GR	1	55.0	-	42 63 112	Root Root Root	2.4 ----- 0.7 ----- <0.02 <0.02	CH-724 -018
Geisenheim Germany 1980	EC	1	4.8	--	53 67 81	Root Root Root	1.8 ----- 0.5 ----- 0.2 -----	CH-724 -022
Bamberg Germany 1980	EC	1	4.8	-	42 56 70	Root Root Root	0.9 ----- 0.3 ----- 0.1 -----	CH-724 -022
Frankfurt Germany 1980	EC	1	4.8	-	60 74 88	Root Root Root	1.2 ----- 0.6 ----- 0.3 -----	CH-724 -022
Geisenheim Germany 1980	GR	1	5.0	-	49 63 77	Root Root Root	1.9 ----- 0.4 ----- 0.2 -----	CH-724 -023
Bamberg Germany 1980	GR	1	5.0	-	42 56 70	Root Root Root	0.7 ----- 0.3 ----- 0.1 -----	CH-724 -023
Frankfurt Germany 1980	GR	1	5.0	-	56 70 84	Root Root Root	<0.02 ----- <0.02 ----- <0.02 -----	CH-724 -023
Frankfurt Germany 1989	EC	1	4.8	1.2	70 77 84	Root Root Root	0.05 ----- 0.03 ----- <0.02 -----	CH-724 -024
Bonn Germany 1989	EC	1	4.8	1.2	42 49 63	Root Root Root	0.2 ----- 0.2 ----- 0.2 -----	CH-724 -024
München Germany 1989	EC	1	4.8	1.2	84 91 105	Root Root Root	<0.02 ----- <0.02 ----- <0.02 -----	CH-724 -024
Hannover Germany 1989	EC	1	4.8	1.2	63 70 84	Root Root Root	0.3 ----- 0.1 ----- 0.04 -----	CH-724 -024
Buttelborn Germany 1990[3]	EC	1	4.8	0.48	11 44 60 89 110	whole plant root root root root	21 ---- 0.1 ---- 0.06 ---- <0.04 ---- <0.04 ----	Anon 1995
	EC	1	4.8	0.48	9	whole plant	5.5 ----	

Location Country, year	Application				PHI, days	Sample	Residues, mg/kg Parent Met	Ref.
	Form	No.	kg ai/ha	kg ai/hl				
					42	root	0.8 ----	
					59	root	0.9 ----	
					92	root	0.7 ----	
					101	root	0.5 ----	
Wulfsdorf Germany 1990[3]	EC	1	4.8	0.48	42	root	1.1 ----	Anon 1995
					60	root	0.2 ----	
					91	root	0.05 ----	
					117	root	0.04 ----	
Braunschweig Germany 1990[3]	EC	1	4.8	0.48	20	whole plant	15 ----	Anon 1995
					42	root	0.3 ----	
					61	root	0.2 ----	
					89	root	0.09 ----	
					170	root	0.07 ----	
Saarlouis Germany 1990[3]	EC	1	4.8	0.48	28	whole plant	3 ----	Anon 1995
					42	root	0.6 ----	
					61	root	0.09 ----	
					90	root	<0.04 ----	
München Germany 1990[3]	EC	1	4.8	0.48	25	whole plant	3.5 ----	Anon 1995
					42	root	0.7 ----	
					60	root	0.3 ----	
					90	root	<0.2 ----	
Rastede Germany 1990[3]	EC	1	4.8	0.48	26	whole plant	2 ----	Anon 1995
					41	root	0.4 ----	
					60	root	0.1 ----	
					90	root	0.09 ----	
					102	root	0.08 ----	
Moos Germany 1990[3]	EC	1	4.8	0.48	28	whole plant	0.6 ----	Anon 1995
					42	root	0.2 ----	
					61	root	0.05 ----	
					90	root	0.04 ----	
					110	root	0.05 ----	
Lubeck Germany 1990[3]	EC	1	4.8	0.48	25	whole plant	3.3 ----	Anon 1995
					41	root	0.8 ----	
					60	root	0.1 ----	
					90	root	0.05 ----	
					94	root	0.08 ----	
Bonn Germany 1990[3]	EC	1	4.8	0.48	42	root	0.04 ----	Anon 1995
					60	root	0.05 ----	
					90	root	0.04 ----	
Germany 1964[4]	GR	1	2	-	119	Root	0.02 ----	CH-601-001
			4	-	119	Root	0.02 ----	
			8	-	119	Root	0.12 ----	
	EC	1	2	-	119	Root	<0.02 ----	
			4	-	119	Root	0.03 ----	
			8	-	119	Root	<0.02 ----	
Netherlands 1964[1]	WP	1	3	-	91	Root	<0.05 -----	CH-724 -001
	WP	1	4	-	91	Root	0.1 -----	
	WP	1	5	-	91	Root	0.07 -----	
Noordwijk Netherlands 1966[1]	GR	1	3	-	343	Root	0.2 -----	CH-724 -002
	GR	2	3	-	252	Root	0.9 -----	

Location Country, year	Application				PHI, days	Sample	Residues, mg/kg Parent Met	Ref.
	Form	No.	kg ai/ha	kg ai/hl				
	GR	1	4	-	343	Root	0.3 -----	
	GR	2	4	-	252	Root	1.0 -----	
	WP	1	3	-	343	Root	0.2 -----	
	WP	2	3	-	252	Root	3.8 -----	
	WP	1	4	-	343	Root	0.2 -----	
	WP	2	4	-	252	Root	1.6 -----	
	GR	1	3	-	343	Root	0.2 <0.05	
	GR	2	3	-	252	Root	1.1 <0.05	
	GR	1	4	-	343	Root	0.3 <0.05	
	GR	2	4	-	252	Root	1.1 <0.05	
	WP	1	3	-	343	Root	0.2 <0.05	
	WP	2	3	-	252	Root	3.8 <0.05	
	WP	1	4	-	343	Root	0.1 <0.05	
	WP	2	4	-	252	Root	2.0 <0.05	
Alkmaar Netherlands 1974[5]	WP	1	4	0.2	103	Root	0.08 ----- 0.07 ----- 0.04 ----- 0.06 -----	Anon 1996c
Alkmaar Netherlands 1974[5]	WP	1	4	0.2	60	Root	0.13 ----- 0.14 ----- 0.13 ----- 0.13 -----	Anon 1996c
Wageningen Netherlands 1977[5]	EC	1	5.3	1.06	93	Root	0.03 ----- 0.12 ----- 0.09 ----- 0.05 -----	Dorlijn, 1977
Twello Netherlands 1977[5]	EC	1	5.3	1.06	89	Root	0.28 ----- 0.25 ----- 0.33 ----- 0.37 -----	Dorlijn, 1977
Wieringerwerf Netherlands 1978[5]	GR	1	1.6	-	184	Root	<0.02 -----	Ten Broeke, 1979
Wieringerwerf Netherlands 1978[5]	GR	1+ 1	0.32+ 0.5	-	184	Root	<0.02 -----	Ten Broeke, 1979
Wieringerwerf Netherlands 1978[5]	GR	1+ 1	1.26+ 1.0	-	184	Root	<0.02 ----- <0.02 ----- <0.02 ----- 0.03 -----	Ten Broeke, 1979
Wieringerwerf Netherlands 1978[5]	GR	1+ 1	2.0+ 2.0	-	184	Root	0.05 ----- <0.02 -----	Ten Broeke, 1979

Location Country, year	Application				PHI, days	Sample	Residues, mg/kg Parent Met	Ref.
	Form	No.	kg ai/ha	kg ai/hl				
							0.03 ----- 0.04 -----	
Zwaagdizk Netherlands 1986[5]	EC	2	4	0.4	103	Root	0.17 ----- 0.25 ----- 0.17 ----- 0.45 ----- 0.18 ----- 0.33 ----- 0.25 ----- 0.17 -----	Greve, 1987
Zwaagdizk Netherlands 1986[5]	EC	2	4	0.4	72	Root	0.15 ----- 0.2 ----- 0.15 ----- 0.22 -----	Greve, 1987
Zwaagdizk Netherlands 1986[5]	EC	1+ 1	4+ 2	0.4+ 0.2	103	Root	0.15 ----- 0.19 ----- 0.07 ----- 0.08 ----- 0.07 ----- 0.1 ----- 0.1 ----- 0.14 -----	Greve, 1987
Zwaagdizk Netherlands 1986[5]	EC	1+ 1	4+ 2	0.4+ 0.2	72	Root	0.16 ----- 0.11 ----- 0.04 ----- 0.06 -----	Greve, 1987
Philippolis South Africa 1972[1,6]	EC	2	1.0	-	42	Root Pulp	0.1 ----- 0.07 -----	CH-724 -008
	EC	7	1.0	-	0	Root Pulp	1.0 ----- 0.5 -----	
	EC	2	2.0	-	42	Root Pulp	0.3 ----- 0.1 -----	
	EC	7	2.0	-	0	Root Pulp	2.3 ----- 1.0 -----	
Seville Spain 1970[1]	GR	1	2	-	119	Root Pulp	<0.02 0.09 <0.02 <0.02	CH-724 -013
	GR	1	3	-	119	Root Pulp	<0.02 0.1 <0.02 0.07	
	GR	1	4	-	119	Root Pulp	<0.02 0.40 <0.02 0.2	
Seville Spain 1972[1]	GR	1	4	-	140	Root	0.4 0.2	CH-724 -016
	GR	1	8	-	140	Root	2.9 0.6	
Seville Spain 1973[1]	GR	1	4	-	511	Root	<0.02 <0.02	CH-724 -019
	GR	2	4	-	140	Root	0.1 0.05	
	GR	1	8	-	511	Root	<0.02 <0.02	

Location Country, year	Application				PHI, days	Sample	Residues, mg/kg Parent	Met	Ref.
	Form	No.	kg ai/ha	kg ai/hl					
	GR	2	8	-	140	Root	0.2	0.2	
Seville Spain 1974[1]	GR	1	4	-	882	Root	<0.02	-----	CH-724 -021
	GR	3	4	-	182	Root	0.3	-----	
						Pulp	0.1	-----	
	GR	1	8	-	882	Root	<0.02	-----	
	GR	3	8	-	182	Root	0.5	-----	
						Pulp	0.4	-----	
Sweden 1966[1]	GR	1	1.5 kg per 20,000 m	-	98	Peel	1.5	-----	CH-724 -004
						Peel	0.9	-----	
						Peel	0.9	-----	
						Pulp	0.06	-----	
						Root	0.3	-----	
	GR	1	1.5 kg per 20,000 m	-	98	Peel	0.9	-----	
						Peel	1.2	-----	
						Pulp	0.04	-----	
						Root	0.3	-----	
	GR	1	1.5 kg per 20,000 m	-	175	Root	0.2	-----	
						Root	0.2	-----	
						Root	0.3	-----	
						Root	0.2	-----	
	GR	1+ 1	1.5+ 2 both kg per 20,000 m	-	84	Root	0.9	-----	
						Root	1.0	-----	
						Root	1.4	-----	
						Root	1.3	-----	
						Root	1.5	-----	
						Root	1.5	-----	
						Root	1.7	-----	
Eggensil Switzerland 1974[1]	GR	1	2	-	84	Root	0.01	-----	CH-724 -020
	GR	1	4	-	84	Root	0.02	-----	
Reichenberg Switzerland 1974[1]	GR	2	1.5	-	140	Root	0.01	-----	CH-724 -020
	GR	2	2	-	140	Root	0.01	-----	
	GR	2	3	-	105	Root	0.04	-----	
	GR	2	4	-	105	Root	0.1	-----	
Switzerland undated[4]	EC	1	1.5	-	49	Root	<0.02	----	CH-601-001
Shell Station Trinidad 1971[1]	EC	1	4	-	140	Root	<0.02	<0.05	CH-790 -027
	EC	1	8	-	140	Root	<0.02	<0.05	
Shell Station Trinidad 1972[1]	EC	1	4	-	448	Root	<0.02	<0.02	CH-790 -030
	EC	2	4	-	112	Root	<0.02	<0.02	
	EC	1	8	-	448	Root	<0.02	<0.02	
	EC	2	8	-	112	Root	<0.02	<0.02	

Location Country, year	Application				PHI, days	Sample	Residues, mg/kg Parent Met	Ref.
	Form	No.	kg ai/ha	kg ai/hl				
Shell Station Trinidad 1973[1]	EC	3	4	-	112	Root	<0.02 <0.02 <0.02 <0.02	CH-790 -032
	EC	3	8	-	112	Root	<0.02 <0.02 <0.02 <0.02	
Kent UK 1963[4]	EC	1	4.48	-	203	Root	0.02 ----	CH-601-001
UK undated[4]	GR	1	4	-	98	Root	0.1 ----	CH-601-001
Suffolk UK undated[4]	GR	1	4.48	-	183	Root	0.04 ----	CH-601-001
			8.96	-	183	Root	0.09 ----	
	EC	1	4.48	-	183	Root	<0.02 ----	
			8.96	-	183	Root	0.04 ----	
Peterborough UK undated[4]	GR	1	4.48	-	161	Root	0.03 ----	CH-601-001
			8.96	-	161	Root	0.06 ----	
	EC	1	4.48	-	161	Root	0.01 ----	
			8.96	-	161	Root	0.04 ----	
UK 1967[1,7]	EC	1	4.4	-	273	Root	<0.02 -----	CH-724-003
Faversham UK 1969[1]	GR	1	4	-	1274	Root	<0.02 -----	CH-790 -026
	GR	4	4	-	182	Root	<0.02 -----	
East Anglia UK 1971[1]	EC	5	1.1	-	98	Root	1.5 <0.05	CH-724 -007
						Pulp	0.8 -----	
						Pre-boiled	0.8 -----	
						Boiled	0.1 -----	
	EC	5	1.1	-	98	Root	1.00 <0.05	
						Pulp	0.5 -----	
						Pre-boiled	0.5 -----	
						Boiled	0.06 -----	
	EC	10	1.1	-	98	Root	2.6 <0.05	
						Pulp	1.0 -----	
						Pre-boiled	1.0 -----	
						Boiled	0.1 -----	
Feltwell UK 1992/3[6]	EC	1	4.7	-	90	Root	<0.02 <0.1	CH-724 -077
	EC	2	4.7	-	19	Root	0.03 0.1	
	EC	2	4.7 + 2.4	-	41	Root	0.05 0.1	
	EC	3	4.7+2x2.4	-	20	Root	0.05 <0.1	
	EC	3	4.7+2x2.4	-	42	Root	0.05 <0.1	
	EC	7	4.7+6x0.78	-	20	Root	<0.02 <0.1	
	EC	7	4.7+6x0.78	-	41	Root	<0.02 <0.1	
	GR	1	4.5	-	90	Root	<0.02 <0.1	
	Gr/Ec	2	4.5+1.2	-	19	Root	0.02 <0.1	
	Gr/Ec	3	4.5+2x1.2	-	23	Root	<0.02 <0.1	
	Gr/Ec	4	4.5+3x1.2	-	20	Root	0.01 <0.1	
	Gr/Ec	4	4.5+3x1.2	-	42	Root	0.01 <0.1	
Friday Bridge UK 1992/3[8]	EC	1	2.4	-	92	Root	<0.02 <0.1	CH-724 -077
	EC	2	2.4	-	22	Root	0.3 <0.1	
	EC	2	2.4	-	42	Root	0.05 <0.1	
	EC	3	2.4	-	20	Root	0.2 <0.1	
	EC	3	2.4	-	42	Root	0.08 <0.1	
	GR	1	2.3	-	92	Root	<0.02 <0.1	
	Gr/Ec	2	2.3+1.2	-	22	Root	0.09 <0.1	

Location Country, year	Application				PHI, days	Sample	Residues, mg/kg		Ref.
	Form	No.	kg ai/ha	kg ai/hl			Parent	Met	
	Gr/Ec	3	2.3+2x1.2	-	23	Root	0.07	<0.1	
	Gr/Ec	4	2.3+3x1.2	-	20	Root	0.02	<0.1	
	Gr/Ec	4	2.3+3x1.2	-	42	Root	0.09	<0.1	
	EC	1	2.4	-	89	Root	0.06	<0.1	
	EC	2	2.4	-	22	Root	0.5	<0.1	
	EC	2	2.4	-	42	Root	0.2	<0.1	
	EC	3	2.4	-	20	Root	0.2	<0.1	
	EC	3	2.4	-	42	Root	0.1	<0.1	
	EC	7	2.4+6x0.78	-	20	Root	0.04	<0.1	
	EC	7	2.4+6x0.78	-	41	Root	0.05	<0.1	
	GR	1	2.3	-	89	Root	0.02	<0.1	
	Gr/ec	2	2.3+1.2	-	22	Root	0.2	<0.1	
	Gr/ec	3	2.3+2x1.2	-	23	Root	0.07	<0.1	
	Gr/ec	4	2.3+3x1.2	-	20	Root	0.2	<0.1	
	Gr/ec	4	2.3+3x1.2	-	42	Root	0.05	<0.1	
Kirton End	EC	1	2.4	-	21	Root	0.2	<0.1	CH-724
UK	EC	1	2.4	-	42	Root	0.2	<0.1	-077
1992/3	EC	2	2.4	-	19	Root	0.3	<0.1	
	EC	2	2.4	-	40	Root	0.3	<0.1	
Cawood	EC	1	2.4	--	22	Root	0.2	<0.1	CH-724
UK	EC	1	2.4	-	42	Root	0.2	<0.1	-077
1992/3	EC	2	2.4	-	22	Root	0.2	<0.1	
	EC	2	2.4	-	42	Root	0.4	<0.1	
Ely	EC	1	2.4	-	24	Root	0.3	<0.1	CH-724
UK	EC	1	2.4	-	43	Root	0.3	0.2	-077
1992/3[8]	EC	2	2.4	-	28	Root	0.4	0.2	
	EC	2	2.4	-	42	Root	0.3	0.2	

Results underlined once or twice are considered comparable with the 4 kg ai/ha EC or WP spray post-planting GAP in The Netherlands
Double underlined residues are from maximum GAP treatments and have been used for estimating the STMR

[1] Duration of sample storage unspecified
[2] Some results were missing from the submitted report
[3] Only the JMPR residue trial summary sheets were supplied (no study report with further information).
[4] No detailed study report; only very brief details of the trial and analyses were available.
[5] Information is taken from residue trial summary sheets submitted by The Netherlands. Full study reports were submitted but were in Dutch
[6] Residues of apparent chlorfenvinphos in control carrots were 0.03 mg/kg
[7] High analytical recovery, >120%
[8] Residues of apparent "acetophenone" in control carrots were 0.02-0.03 mg/kg

Met = 1-(2,4-dichlorophenyl)ethanol

Table 27. Residues of chlorfenvinphos in commercially grown over-wintered field carrots of known treatment history during 1989-92 in the UK. All EC formulations. Roots analysed (Anon., 1989-92).

Soil type	Application		PHI, months	Chlorfenvinphos, mg/kg
	No.	kg ai/ha[1]		
Organic	2	2.4	6	0.20
Silty loam	2	1.2	7	0.20
Sandy loam	1	2.35	5	<0.02
Sandy loam	1	2.4	6	<0.02
Sandy loam	1	2.4	9	0.05
Sandy loam	2	2.4+0.84	6	0.09
Sandy loam	1	2.36	3	0.12
Sandy loam	1	2.35	6	0.13
Sandy loam	1	0.6	5	0.15
Sandy loam	1	2.4	9	0.20
Sandy loam	1	2.35	5	0.36
Sandy loam	1	2.36	5	0.83
Sandy loam	1	2.4	9	1.04
Sandy loam	2	2.4	6	1.30
Peaty loam	1	2.36	3	<0.01
Peaty loam	1	2.4	3	<0.01
Peaty loam	2	2.4+N/S	5	<0.02
Peaty loam	1	2.35	3	0.02
Peaty loam	1	2.4	3	0.04
Peaty loam	2	2.4	6	0.05
Peaty loam	2	2.35	3	0.10
Peaty loam	2	2.4+N/S	5	0.17
Peaty loam	2	2.35	3	0.19
Peaty loam	2	2.4	5	0.19[2]
Peaty loam	2	2.4+N/S	5	0.29
Peaty loam	2	2.4+N/S	5	0.31
Peaty loam	1	2.4	6	0.38
Peaty loam	2	2.4	5	1.4[2]
Peaty loam	2	2.4	5	1.6[2]
Unknown	2	2.4	5	0.01
Unknown	1	2.4	6	0.01
Unknown	1	2.4	6	0.20

[1] Approved in the UK as a spray application up to 2.35 kg ai/ha
[2] Mean of duplicate results
N/S Not specified

Parsley root. No GAP was reported for parsley root (i.e. Hamburg parsley) although summarized reports of residue trials were available from Germany.

Table 28. Supervised field trials on parsley root, Germany, 1979. All single granular applications, 5.0 kg ai/ha (Anon., 1995).

Location	PHI, days	Sample	Chlorfenvinphos, mg/kg
Stuttgart	93	leaves	<0.02
	128	leaves	<0.02
	170	leaves	0.08
	170	root	0.2
Buttelborn	78	leaves	0.2
	161	leaves	<0.02
	78	root	1.7
	161	root	0.2
Lübeck	132	leaves	0.1
	152	leaves	0.1
	138	root	1.3
	152	root	1.5
Münster	83	leaves	0.03
	111	leaves	<0.02
	83	root	0.4
	111	root	0.3
Hurthfischenich	50	leaves	0.05
	85	leaves	<0.02
	115	leaves	<0.02
	85	root	0.08
	115	root	0.03
	128	leaves	0.02
	128	root	0.21

Only the JMPR residue trial summary sheets were supplied (no study report with further information).

Parsnip. GAP was reported for The Netherlands and the UK. The UK provided government-generated data on residues in overwintered commercial parsnips of known treatment history. Two residues were from treatments according to UK GAP (2.35 kg ai/ha). The residues were 0.14 and 0.16 mg/kg.

Table 29. Residues of chlorfenvinphos in commercially grown overwintered field parsnips of known treatment history during 1989-92 in the UK. All EC. Roots analysed (Anon., 1989-92).

Soil type	Application		PHI, months	Chlorfenvinphos, mg/kg
	No.	kg ai/ha		
Peat	1	4.8	5	<0.02
Flinty sand	1	0.59	3	0.07
Sand	1	2.35	7	0.14
Sand	1	2.36	5	0.16
Sand	N/S	N/S	N/S	0.35

Double underlined residues are from maximum UK GAP treatments (spray application up to 2.35 kg ai/ha) and have been used for estimating the STMR
N/S Not specified

Potatoes. There are registered uses in The Netherlands and Poland.

Residue trials were carried out in the UK, Spain, Australia and Poland, but they were very old and poorly reported with few details.

Table 30. Supervised field trials on potatoes. Tubers analysed.

Location, Country, Year	Application			PHI, days	Residues, mg/kg Parent	Met	Ref.
	Form.	No.	kg ai/ha				
Kent UK 1963	EC	1	4.5 soil application	112	<0.02	<0.05	CH-601-001
Kent UK 1966	EC	1	0.25 foliar spray	65	<0.02	<0.05	CH-601-001
Spain 1966	EC	1	0.25 foliar spray	13	<0.02	<0.05	CH-601-001
Seville Spain 1965	EC	1	1 foliar spray	28	<0.02	<0.05	CH-601-001 & CH-640-002
Australia undated	EC	8	0.25 foliar spray	5	0.01	-----	CH-601-001
Poland undated	FSD	1	0.5 foliar spray	69	0.02	-----	CH-601-001
Poland undated	EC	1	0.24 foliar spray	69	0.02	-----	CH-601-001

No detailed study reports; only very brief details of the trials and analyses were available.
Met = 1-(2,4-dichlorophenyl)ethanol

Radishes. GAP was reported for Germany, The Netherlands and the UK.

Residue trials (Table 31) were in Germany and Switzerland. Several of the trials were very old and none were reported in detail. In addition the UK provided government-generated data on residues (four results) in overwintered commercial radishes of known treatment history (Table 32). The residues following applications close to GAP were all <0.1 mg/kg.

Table 31. Supervised field trials on radishes. All single applications.

Location Country, year	Application		PHI, days	Portion analysed	Chlorfenvinphos, mg/kg	Ref.
	Form.	kg ai/ha				
Germany 1964[1]	GR	4	63	root	<0.02	CH-601 -001
	GR	8	63	root	<0.02	
	EC	4	63	root	<0.02	
	EC	8	63	root	<0.02	
	GR	4	56	root	<0.02	
	GR	8	56	root	0.05	
Oldenburg Germany	GR	4.0	27	whole plant	0.12	Anon 1995
			33	root	0.08	

Location Country, year	Application		PHI, days	Portion analysed	Chlorfenvinphos, mg/kg	Ref.
	Form.	kg ai/ha				
1983[2]			40	root	0.06	
Braunschweig Germany 1983[2]	GR	4.0	29 42 57	whole plant root root	1.1 0.07 <0.02	Anon 1995
Germany 1965[1]	GR	2	28	root	0.95	CH-601-001
Germany 1966[1]	GR GR	2 3	35 35	root root	<0.04 <0.05	CH-601 -001
Switzerland 1966[1]	GR	2	17	root	<0.02	CH-601-001

Residues underlined once or twice are considered comparable with the German GAP for granular applications
Double underlined residues are from maximum GAP treatments and have been used for estimating the STMR
[1] No detailed study report; only very brief details of the trial and analyses were available.
[2] Only the JMPR residue trial summary sheets were supplied (no study report with further information provided)

Table 32. Residues of chlorfenvinphos found in commercially grown field radishes of known treatment history during 1989-92 in the UK, 1989-92. All granular applications at 2.24 kg ai/ha. Roots analysed (Anon., 1989-92).

PHI, months	1	1	1	1
Chlorfenvinphos, mg/kg	<0.1	<0.1	<0.1	<0.1

UK GAP is a granule application up to 2.0 kg ai/ha

Swedes and turnips. GAPs for swedes and turnips was reported for The Netherlands and the UK.

One field trial in the UK on swedes and three in the UK or USA on turnips were reported, but the analytical recovery was high (>120%) in the trial on swedes and the others were old and poorly described with no detailed study reports. The Meeting also received reports of six German trials on swedes or turnips in which the commodity was described as "turnip cabbage". This was an error in translation from the original German and the correct description was "swede/turnip". These trials did not comply with UK or Netherlands GAP.

Table 33. Supervised field trials on swedes and turnips.

Crop, Location Country, year	Application			PHI, days	Sample	Chlorfenvinphos, mg/kg	Ref.
	Form	No.	kg ai/ha				
SWEDE							
Wellesbourne UK 1964[1,2]	GR GR GR GR EC	1 1 1 1 1	2.8 2.8 2.8 2.8 2.8	109 109 126 126 99	root root root root root	<0.05 <0.05 <0.05 <0.05 <0.05	CH-724 -065
TURNIP							
Kent UK undated[3]	GR GR EC	1 1 1	4.5 4.5 4.5	112 112 112	root root root	<0.02 <0.02 <0.02	CH-601 -001
Wellesbourne UK	EC	1	0.84	0 0	foliage root	14 <0.02	CH-640-002

Crop, Location Country, year	Application			PHI, days	Sample	Chlorfenvinphos, mg/kg	Ref.
	Form	No.	kg ai/ha				
1965[3]				10 18 30 30	root root foliage root	<0.02 <0.02 <0.02 <0.02	
USA undated[3]	GR GR+ EC GR+ EC	1 1+ 3 1+3	1.12 1.12+ 1.12 1.12 1.12	70 21 56	root root root	<0.05 <0.21 0.08	CH-601 -001
SWEDE or TURNIP							
Geisenheim Germany 1980[1]	EC	1+2	4.88 0.144	0 7 14 21 28	root	0.09 <0.02 <0.02 <0.02 <0.02	CH-721 -013
Bamberg Germany 1980[1]	EC	1+2	4.88 0.144	0 7 14 21 28	root	0.5 <0.02 <0.02 <0.02 <0.02	CH-721 -013
Frankfurt Germany 1980[1]	EC	1+2	4.88 0.144	0 7 14 21 28	root	0.2 0.05 <0.02 <0.02 <0.02	CH-721 -013
Geisenheim Germany 1980[1]	GR GR	1 1	0.1 kg/m^2 0.1 g/plant	49 56 70 49 56 70	root	0.10 0.04 0.02 0.5 0.2 0.1	CH-721 -016
Bamburg Germany 1980[1]	GR GR	1 1	0.1 kg/m^2 0.1 g/plant	49 63 70 49 63 70	root	0.2 0.02 <0.02 0.7 0.1 0.06	CH-721 -016
Frankfurt Germany 1980[1]	GR GR	1 1	0.1 kg/m^2 0.1 g/plant	49 60 70 49 60 70	root	0.10 0.02 <0.02 1.6 0.6 0.2	CH-721 -016

[1] Duration of sample storage unspecified
[2] High analytical recovery (>120%)
[3] No detailed study report; only very brief details of the trial and analyses were available.

Sweet potatoes. No GAP was reported although reports of residue trials in Trinidad were submitted.

Table 34. Supervised field trials on sweet potatoes in Trinidad. All EC applications. Tubers analysed. Duration of sample storage was not specified.

Location, year	Application		PHI, days	Residues, mg/kg		Ref.
	No.	kg ai/ha		Parent	Met	
Shell Station	1	4	168	<0.02	<0.05	CH-790
Trinidad 1971	1	8	168	<0.02	<0.05	-027
Sell Station	1	4	532	<0.02	<0.02	CH-790
Trinidad	2[1]	4	196	<0.02	<0.02	-030
1972	1	8	532	<0.02	<0.02	
	2[1]	8	196	<0.02	<0.02	
Shell Station	1	4	868	<0.02	<0.02	CH-790
Trinidad	3[1]	4	154	<0.02	<0.02	-032
1973	1	8	868	<0.02	<0.02	
	3[1]	8	154	<0.02	<0.02	

[1] Only one application was made in any one year. Met = 1-(2,4-dichlorophenyl)ethanol

Celery. There is a registered use in The Netherlands.

One group of residue trials was reported, at an unspecified location. It was poorly described, with no detailed study report.

Table 35. Supervised field trials on celery (undated). Stems analysed.

Application				PHI, days	Residues, mg/kg Parent Met	Ref.
Form.	No.	kg ai/ha	kg ai/hl			
GR	1	2	-	112	0.2 ND	CH-601
GR	1	2	-	112	0.02 ND	-001
GR	1	1	-	91	0.03 ND	
GR	1	2	-	91	0.05 ND	
undated	1	17 mg/plant	root dip	77	0.5 ND	

No detailed study report; only very brief details of the trial and analyses were available.
Met = 1-(2,4-dichlorophenyl)ethanol

Rape seed. GAP for rape was reported for Austria, Germany, The Netherlands and Poland.

Several field trials were carried out in France and Germany. Six German trials complied with German GAP for EC spray. Residues in all the trials were <0.02 mg/kg. There were no trials with the broadcast application of granules at 3 kg ai/ha used in The Netherlands, although in two French trials with an application rate of 1 kg ai/ha the residues were <0.02 mg/kg.

Table 36. Supervised field trials on rape.

Location, Country, year	Application			PHI, days	Sample	Residues, mg/kg Parent Met	Ref.
	Form.	No.	kg ai/ha				
Mornay France 1988	GR	1	1.0	322	Seed	<0.02 -----	CH-750-011
Saulz-le-Duc France 1988	GR GR	1 1	1.0 1.0	336 322	Seed Seed	<0.02 ----- <0.02 -----	CH-750 -011
Villefargeu France 1991	EC	1	0.6	126	Seed	<0.02 -----	CH-750 -013
Buscieres sur Are France 1991	EC	1	0.6	105	Seed	<0.02 -----	CH-750-013
Saulay France 1991	EC	1	0.6	133	Seed	<0.02 -----	CH-750-013
Le Mee France 1991	EC	2	0.6	147	Seed	0.09 -----	CH-750-013
Lübeck Germany 1973	EC	1	0.144	77	Seed	≤0.02 ≤0.02	CH-750-007
Ansbach Germany 1974	EC	1	0.192	70 77	Seed Seed	<0.02 ----- <0.02 -----	CH-750 -008
Frankfurt Germany 1980	EC	2	0.144	35	Seed	≤0.02 ≤0.02	CH-750-009
München Germany 1989	EC	2	0.144	0 34	plant plant	3.12 ----- 0.025 -----	CH-750 -012

Location, Country, year	Application			PHI, days	Sample	Residues, mg/kg Parent	Met	Ref.
	Form.	No.	kg ai/ha					
				44	Seed	≤0.02	-----	
Solms Oberbiel Germany 1989	EC	2	0.144	0 38 50	plant plant Seed	1.68 0.02 ≤0.02	----- ----- -----	CH-750 -012
Hanau Germany 1989	EC	2	0.144	0 28 39	plant plant Seed	2.74 0.055 ≤0.02	----- ----- -----	CH-750 -012
Bad Segeberg Germany 1989	EC	2	0.144	0 50 62	plant plant Seed	2.22 <0.02 ≤0.02	----- ----- -----	CH-750 -012

Duration of sample storage was not specified.
Results underlined once or twice are considered comparable with German GAP for EC sprays.
Double underlined residues are from maximum GAP treatments and have been used for estimating the STMR
Met = 1-(2,4-dichlorophenyl)ethanol

Parsley. There are registered uses in The Netherlands and the UK with WP or EC spray applications.

Summarized reports of residue trials were available from Germany, but all the trials were with granular formulations whereas the reported GAP applications are by spraying.

Table 37. Supervised field trials on parsley in Germany. All single GR applications at 5.0 kg ai/ha. Leaves analysed (Anon., 1995).

Location, year	PHI, days	Chlorfenvinphos, mg/kg
Oldenburg 1979	89 96 104	0.1 0.04 0.04
Berlin 1979	69 79 90 128	0.07 0.04 0.04 <0.02
Nahermittenhausen 1979	83	<0.02
Hurthfischenich 1979	50 85	0.06 <0.02
Buttelborn	70 70	0.2 0.03
Münster 1975	88	0.01
Stenkamp Asche 1975	96	0.03

Only the JMPR residue trial summary sheets were supplied (no study report with further information provided).

Maize. GAP was reported for The Netherlands.

Residue trials were carried out in France but were very old and poorly described with no detailed study reports.

Table 38. Supervised field trials on Maize in France. All EC applications. Cobs analysed.

Location, year	Application		PHI, days	Residues, mg/kg Parent	Met	Ref.
	No.	kg ai/ha				
Sauveterre	1	1	14	<0.02	-----	CH-640-002
1965	1	2	14	<0.02	-----	
1965	1	1	98	<0.02	<0.02	CH-601-001
1966	2	0.6	45	<0.02	<0.02	CH-601-001

There were no detailed study reports; only very brief details of the trials and analyses were available.
Met = 1-(2,4-dichlorophenyl)ethanol

Wheat. There are registered uses in the UK. Two residue trials in the UK were very old and poorly reported with inadequate detail.

Table 39. Supervised field trials on wheat in the UK. Single applications. Grain analysed. Undated.

Location	Application		PHI, days	Residues, mg/kg Parent	Met	Ref.
	Form.	kg ai/ha				
Lincolnshire	GR	1.75	310	<0.02	<0.02	CH-601-
	DS	22.8kg/ tonne seed		<0.02	<0.02	001
Cambridgeshire	GR	1.75	310	<0.02	<0.02	CH-601-
	DS	22.8kg/ tonne seed		<0.02	<0.02	001

Met = 1-(2,4-dichlorophenyl)ethanol

A limited number of poorly reported trials on pasture, sorghum, peanuts, cotton seed, apples, tangerines and sugar beet were also submitted (Anon undated; Beynon, 1966). They have not been reviewed as no GAP is reported for these crops.

Residues in following crops

Lettuce. No GAP was reported for lettuce, but measurable residues could occur in lettuce planted as a following crop as a result of treatment of the primary crop.

Rotational crop trials on lettuce and lamb's lettuce were reported from Germany. The lettuce or lambs lettuce was planted 1-4 months after the treatment of radishes as the primary crop at 4 kg ai/ha. The dates of harvest of the radish crop and the residue levels in the soil were not recorded. The residues in lamb's lettuce at harvest were <0.04 (4) and 0.19 mg/kg, and in lettuce <0.04 (5), 0.05, 0.07 and 0.11 mg/kg. The trials data were submitted in JMPR summary format only with no accompanying study reports.

German GAP for radishes is a “spreading” application at 3 kg ai/ha (field) or 4 kg ai/ha (glass). Similar GAP for soil treatment was reported at comparable application rates for several other crops in a number of countries.

Table 40. Residues in lettuce and lamb's lettuce planted in the field as rotational crops following a single treatment of radishes as the primary crop with granules at 4.0 kg ai/ha. Leaves analysed. Germany, 1983 (Anon., 1995).

CROP Location	PHI, days[1]	Chlorfenvinphos, mg/kg
LAMB'S LETTUCE		
Oldenburg	168	<0.04
	189	0.19
	217	0.16
Braunschweig	144	<0.04
	161	<0.04
	179	<0.04
München	71	<0.04
	90	<0.04
	105	<0.04
Hurth-Fischenich	183	<0.04
	190	<0.04
	197	<0.04
Mainz-Bretzenheim	118	<0.04
HEAD LETTUCE		
Mainz-Bretzenheim	118	<0.04
Hurth-Fischenich	126	<0.04
	134	<0.04
	141	<0.04
Lübeck	118	0.11
	127	<0.04
	135	<0.04
München	36	<0.04
	50	<0.04
	64	<0.04
Freiburg	91	<0.04
	105	0.05
	114	<0.04
Frankfurt	69	<0.04
	82	<0.04
	90	<0.04
	62	<0.04
	75	<0.04
	83	<0.04
Stuttgart	73	<0.04
	84	<0.04
	93	<0.04
Oldenburg	75	0.06
	84	0.07
	92	<0.04

Only the JMPR residue trial summary sheets were supplied (no study report with further information provided).
Residues underlined once or twice are considered to reflect possible commercial practice.
Double underlined residues are from maximum GAP treatments and have been used for estimating the STMR

[1] The PHIs are from the last treatment of the radish crop to the harvesting of the secondary lettuce or lamb's lettuce crop.

Livestock feeding or topical treatment trials

In a 1966 Australian study designed to find out whether residues occur in the milk of cattle grazing on treated pasture, chlorfenvinphos was applied once to grass at 0.42 kg ai/ha and lactating cows were admitted to the pasture two days after treatment (Elgar, 1966e). The mean residues of chlorfenvinphos in the grass four days, 1 week, 2 weeks and 3 weeks after treatment were 17, 5.7, 4.4 and 2.5 mg/kg respectively.

No residues of chlorfenvinphos (<0.01 mg/kg), 2,4-dichorophenacyl chloride (<0.002 mg/kg), 1-(2,4-dichlorophenyl)ethanol (<0.01 mg/kg) or 2,4 -dichloroacetophenone (<0.005 mg/kg) were found in milk samples taken from the cows at these times.

In a briefly reported study (Schroder, 1984), two heifers and two steer calves were dipped in an unspecified formulation containing 0.037 kg ai/hl of chlorfenvinphos. At the time of dipping, the dipwash had been in the tank for up to 57 weeks. Tissue samples were taken 7 days after dipping. The residues were all below the LOD in liver (<0.1 mg/kg), muscle (<0.05 mg/kg) and kidney (<0.05 mg/kg). In ‘fat’ the residues were in the range <0.1 to 0.27 mg/kg.

FATE OF RESIDUES IN STORAGE AND PROCESSING

In storage

No data were submitted.

In processing

The distribution of chlorfenvinphos in carrots with incurred residues following EC treatments was investigated as part of a UK government research programme (Anon., 1989-92). The results are given in Table 41.

The highest concentrations of chlorfenvinphos were in the crowns of the carrots. The distribution varied but the data indicate that most consumers would remove 30% of the residue during preparation.

The results of a preliminary study of the distribution of residues between the core and peel of carrots and the effects of cooking topped but unpeeled carrots are given in Table 42. The effect of peeling and taking the top portion (crown and next 1 cm) from the roots was to remove 97-99% of the residue.

The effect of cooking topped (but not peeled) roots had, at most, a moderate effect on the concentration of chlorfenvinphos.

Table 41. Mean distribution of chlorfenvinphos residues along 7 average-sized carrot roots taken from samples of commercially grown crops.

Sample No	Crown			1 cm slice below crown			Remainder		
	sample wt., g	Residue, mg/kg	Residue, %[1]	Sample wt., g	Residue mg/kg	Residue, %[1]	sample wt., g	Residue, mg/kg	Residue, %[1]
1	4	11.3	19	25	3.0	34	389	0.27	47
2	4	1.6	24	21	0.33	29 26	450	0.03	48 50
3	6	3.2	39	42	0.32	27 25	621	0.03	34 36
4	5	2.2	34 37	30	0.11	9	391	0.05	57
5	6	9.4	14	28	1.5	11	986	0.29	75
6	2	3.7	21	26	0.51	31	429	0.05	47
7	3	11.6	61	29	0.45	27	363	0.02	12
Mean		1			6			94	
		30			24			46	

[1] % of total residue in carrot

Table 42. The effects of peeling and boiling on residues of chlorfenvinphos in carrots.

Part of root and process	Sample wt., g uncooked	% of carrot wt.	Sample wt., g cooked	chlorfenvinphos, mg/kg	µg in sample	% of residue
Sample No 1 whole root, uncooked	491			0.20		
calc. topped root before cooking[1]	309			0.21	66	
topped root after cooking	408		370	0.19	70	
top slice taken from root, uncooked	4	1		7.7	32	33
peel, uncooked	64	21		0.98	63	64
peeled core, uncooked	245	79		0.01	3	3
Sample No 2 whole root, uncooked	708			0.20		
calc. topped root before cooking[1]	746			0.37	280	
topped root after cooking	738		683	0.11	75	
top slice taken from root, uncooked	8	1		10.4	79	22
peel, uncooked	91	12		3.1	278	77
peeled core, uncooked	655	88		0.003	2	1

[1] Calculated from sum of uncooked peel and uncooked peeled core. Note that the peeled and boiled carrots were different sub-samples, hence results are unlikely to correspond exactly

Carrots - commercial cooking. In a study carried out in 1966 (Elgar, 1966a), carrots grown in soil treated with 'Birlane' were used to investigate the effect that cooking (specifically the process used commercially in preparing baby foods) had on chlorfenvinphos residues. The raw carrots, containing residues of either 0.05 or 0.07 mg/kg, were made into cooked purée by blanching in water, diluting with brine and macerating, then cooking under steam pressure for 35 minutes at 120°C. Samples were analysed for residues of chlorfenvinphos, 2,4-dichloroacetophenone and 2,4-dichlorophenacyl chloride after extraction with acetone and petroleum spirit. The acetone was removed and the petroleum extracts dried by filtering through anhydrous sodium sulfate. After clean-up on Florisil, the residues were determined by GLC with an ECD. Where recoveries were low, an enzyme-inhibition method was used

for the determination of residues, the details of which were not given.

The final chlorfenvinphos residue in the cooked purée from both batches of carrots was 0.02 mg/kg. It was stated that the reduction in the residue from raw carrots to cooked purée was due to two factors, the addition of brine and the cooking. No residues (<0.01 mg/kg) of the metabolites 2,4-dichloroacetophenone or 2,4-dichlorophenacyl chloride were detected in the raw or cooked purée.

Carrots - canning. Carrots treated in June 1966 with 'Birlane' at 2.24 kg ai/ha and harvested in the following December were made into a purée and canned (Elgar, 1967c). Six cans of carrots were analysed by GLC with EC detection. No residues of chlorfenvinphos (<0.01 mg/kg) were found. The treated carrots were not analysed before canning.

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

National monitoring data were supplied by Australia, The Netherlands, Poland, and the UK.

The results of monitoring analyses of samples taken randomly from export and domestic sources undertaken by Australia from 1 July 1993 to 31 December 1995 are shown in Table 43 (Anon, 1996b).

Table 43. Australian monitoring data for chlorfenvinphos.

Commodity	Australian MRL, mg/kg	Reporting limit	Total samples	No. with residues
Beef fat	0.2	0.01	7151	0
Buffalo fat	0.2	0.1	15	0
Deer fat		0.1	65	0
Emu fat		0.1	10	0
Game goat fat	0.2	0.1	176	0
Goat fat	0.2	0.1	198	0
Game pig fat		0.1	240	0
Horse fat		0.1	259	1 (0.39%)[1]
Kangaroo fat		0.1	223	0
Ovine fat	0.2	0.1	6146	0
Porcine fat		0.1	2060	0
Poultry fat		0.1	244	0
Barley whole		0.01	711	0
Bran from wheat		0.01	129	0
Canola whole		0.01	19	0
Faba beans whole		0.01	9	0
Flour from wheat whole		0.01	129	0
Lupins whole		0.01	184	0
Oats whole		0.01	67	0
Peas whole		0.01	67	0
Sorghum whole		0.01	16	0
Wheat whole	0.05	0.01	2563	0

[1] Determined residue was described as being in the range "Reporting limit - <0.2 x Reporting limit"
Samples described as fat are portions of adhering fat taken from animal carcases

The results of monitoring in The Netherlands in 1991-1994 are shown in Tables 44 and 45 (Olthof, 1996).

Table 44. Monitoring data for chlorfenvinphos in The Netherlands, 1991-93.

Commodity	Samples analysed	Samples without residues (LOD 0.05 mg/kg)	Samples with residues < MRL	Samples with residues ≥ MRL	Mean, mg/kg	MRL, mg/kg
CITRUS FRUIT						1
Lemons	181	160	19	2	0.09	1
Tangerines	523	504	19	0	<0.05	1
Oranges	958	937	21	0	<0.05	1
MISC. FRUIT						0.05*
Kiwifruit	309	307	2	0	<0.05	0.05*
ROOT AND TUBER VEGETABLES						0.5
Carrots	609	497	106	6	<0.05	0.5
BULB VEGETABLES	106					0.5
Onions		104	2	0	0.05	0.5
BRASSICA VEGETABLES						0.1
Red cabbage	134	131	3	0	<0.05	0.1
STEM VEGETABLES						
Celery	807	805	2	0	<0.05	0.5

Residues <LOD are assumed to be at half the LOD for the calculation of the mean

Table 45. Monitoring data for chlorfenvinphos in The Netherlands, 1994.

Commodity	Samples analysed	Samples without residues (LOD 0.05 mg/kg)	Samples with residues < MRL	Samples with residues ≥ MRL	Mean, mg/kg	MRL, mg/kg
CITRUS FRUIT						1
Grapefruit	111	109	2	0	<0.05	1
Lemons	102	90	12	0	0.09	1
Tangerines	215	208	7	0	<0.05	1
Oranges	348	342	6	0	<0.05	1
STONE FRUIT						0.05*
Peaches	113	112	1	0	<0.05	0.05*
BERRIES AND SMALL FRUIT						0.05*
Grapes	336	335	1	0	<0.05	0.05*
ROOT AND TUBER VEGETABLES						0.5
Carrots	141	94	47	0	0.05	0.5
STEM VEGETABLES						
Celery	84	78	6	0	<0.05	0.5
BRASSICA VEGETABLES						0.1
Kale	47	45	2	0	<0.05	0.1
LEAF VEGETABLES AND FRESH HERBS	1277					0.1
Lettuce	511	1276	1	0	<0.05	0.1
Endive		510	1	0	<0.05	0.1
CEREALS						0.05*
Maize	19	18	1	0	<0.05	0.05*

Residues <LOD are assumed to be at half the LOD for the calculation of the mean.

In 1994, 120 samples of glasshouse and 20 samples of field-grown cucumbers were analysed for chlorfenvinphos residues in Poland (Anon 1996a). No measurable residues were found although the LOD was not reported.

Monitoring in the UK gave the results shown in Table 46 (Anon, 1989-92).

Table 46. Residues of chlorfenvinphos reported during routine UK monitoring in retail samples during 1989-92.

Commodity	Source	No. analysed	LOD, mg/kg	Below LOD, No	%	Residues above LOD, mg/kg
citrus, soft (satsumas, clementines, mandarins and tangerines)	EC	26	0.05	24	92	0.2, 0.3
	Other	41		41	100	
	Unknown	1		1	100	
grapefruit	EC	2	0.05	2	100	
	Other	22		22	100	
	Unknown	1		1	100	
limes	Other	12	0.02	12	100	
lemons	EC	9	0.05	8	89	0.4
	Other	3		2	67	0.2
carrots	EC	13	0.05	12	92	0.3
	Other	2		2	100	
fresh immature carrots	Unknown	32		30	94	0.3 (UK), 0.7
canned immature carrots	Unknown	10	0.05	10	100	
frozen immature carrots	Unknown	14	0.05	14	100	
radishes	Unknown	7	0.1	7	100	
parsnips	UK	20	0.05	20	100	
	Unknown	3		3	100	
sweet corn	UK	15	0.1	15	100	
	EC	3	0.1	3	100	
	Other	1	0.1	1	100	
	Unknown	1	0.1	1	100	
mushrooms	UK	29	0.05	29	100	
	EC	10		10	100	
chicken	UK	90	0.02	90	100	
	EC	7	0.02	7	100	
	Unknown	21	0.02	21	100	
lamb	UK	6	0.02	6	100	
	Other	103	0.02	103	100	
	Unknown	3	0.02	3	100	
paté	UK	11	0.05	11	100	
	EC	23	0.02	23	100	
	Unknown	3	0.02	3	100	
sausages (pork)	Unknown	4	0.05	4	100	

Commodity	Source	No. analysed	LOD, mg/kg	Below LOD, No	%	Residues above LOD, mg/kg
sausages (beef)	Unknown	12	0.05	12	100	
pies and pasties	UK	191	0.05	191	100	
canned meat	UK	13	0.2	13	100	
	EC	15	0.02	15	100	
	Other	8	0.02	8	100	
	Unknown	1	0.02	1	100	
rabbit	UK	7	0.05	7	100	
	Other	11	0.05	11	100	
	Unknown	16	0.05	16	100	
sheep kidney	UK	55	0.02	55	100	
cattle meat	Unknown	41	0.02	41	100	
pig meat	Unknown	37	0.02	37	100	
cattle kidney fat	UK	81	0.02	81	100	
pig kidney fat	UK	77	0.02	77	100	
sheep kidney fat	UK	82	0.02	82	100	

NATIONAL MAXIMUM RESIDUE LIMITS

The national MRLs for chlorfenvinphos shown below were reported.

Country	Crop	MRL, mg/kg	Reference
Australia	broccoli	0.05	Anon 1996b
	Brussels sprouts	0.05	Anon 1996b
	cabbages, head	0.05	Anon 1996b
	carrot	0.4	Anon 1996b
	cattle, edible offal of	0.2	Anon 1996b
	cattle meat (in the fat)	0.2	Anon 1996b
	cauliflower	0.1	Anon 1996b
	celery	0.4	Anon 1996b
	cotton seed	0.05	Anon 1996b
	egg plant (aubergine)	0.05	Anon 1996b
	goat, edible offal of	0.2	Anon 1996b
	goat meat (in the fat)	0.2	Anon 1996b
	horsemeat	0.1	Anon 1996b
	leek	0.05	Anon 1996b
	maize	0.05	Anon 1996b
	milks (in the fat)	0.2	Anon 1996b
	mushrooms	0.05	Anon 1996b
	onion, bulb	0.05	Anon 1996b
	peanut	0.05	Anon 1996b
	potato	0.05	Anon 1996b
	radish	0.1	Anon 1996b
	rice	0.05	Anon 1996b
	sheep, edible offal of	0.2	Anon 1996b

Country	Crop	MRL, mg/kg	Reference
	sheep meat (in the fat)	0.2	Anon 1996b
	swede	0.05	Anon 1996b
	sweet potato	0.05	Anon 1996b
	tomato	0.1	Anon 1996b
	turnip, garden	0.05	Anon 1996b
	wheat	0.05	Anon 1996b
Austria	carrot	0.5	Anon 1996c
	celery	0.4	Anon 1996c
	citrus	1	Anon 1996c
	coffee	0.4	Anon 1996c
	milk	0.05	Anon 1996c
	parsley	0.5	Anon 1996c
	potato	0.1	Anon 1996c
	rape	0.1	Anon 1996c
	sugar beet	0.1	Anon 1996c
Belgium	cabbage	0.1 to 0.5	Anon 1996c
	carrot	0.5	Anon 1996c
	leek	0.1	Anon 1996c
	onions	0.5	Anon 1996c
	potato	0.05	Anon 1996c
France	asparagus	0.5	Anon 1996c
	bean	0.1	Anon 1996c
	cabbage	0.1	Anon 1996c
	carrot	0.5	Anon 1996c
	celery	0.5	Anon 1996c
	cereals	0.05	Anon 1996c
	corn salad	0.1	Anon 1996c
	courgette	0.1	Anon 1996c
	cress	0.1	Anon 1996c
	eggplant	0.1	Anon 1996c
	garlic	0.5	Anon 1996c
	gherkin	0.1	Anon 1996c
	melon	0.1	Anon 1996c
	mushrooms	0.05	Anon 1996c
	onions	0.5	Anon 1996c
	parsley	0.5	Anon 1996c
	potato	0.5	Anon 1996c
	radish	0.5	Anon 1996c
	rape	0.02	Anon 1996c
	shallot	0.5	Anon 1996c
	soya bean	0.1	Anon 1996c
	spinach	0.1	Anon 1996c
	turnip	0.5	Anon 1996c
Germany	cabbage	0.5	Anon 1996c
	carrot	0.5	Anon 1996c
	celery	0.5	Anon 1996c
	citrus	1	Anon 1996c

Country	Crop	MRL, mg/kg	Reference
	citrus juice	0.05	Anon 1996c
	coffee	0.5	Anon 1996c
	cucumber	0.1	Anon 1996c
	leek	0.5	Anon 1996c
	onions	0.5	Anon 1996c
	parsley	0.5	Anon 1996c
	potato	0.05	Anon 1996c
	radish	0.5	Anon 1996c
	rape	0.1	Anon 1996c
	root/tuber veg	0.5	Anon 1996c
	shallot	05	Anon 1996c
	sugar beet	0.1	Anon 1996c
	turnip	0.5	Anon 1996c
Ireland	carrot	0.5	Anon 1996c
	parsnip	0.5	Anon 1996c
Italy	cabbage	0.1	Anon 1996c
	carrot	0.5	Anon 1996c
	celery	0.5	Anon 1996c
	maize	0.05	Anon 1996c
	mushrooms	0.05	Anon 1996c
	potato	0.1	Anon 1996c
	rape	0.05	Anon 1996c
	sugar beet	0.1	Anon 1996c
Japan	apricot	0.5	Anon 1996c
	broccoli	0.05	Anon 1996c
	cabbage	0.2	Anon 1996c
	cauliflower	0.1	Anon 1996c
	chestnut	0.2	Anon 1996c
	citrus	3 to 5	Anon 1996c
	cucumber	0.2	Anon 1996c
	eggplant	0.2	Anon 1996c
	kidney bean	0.2	Anon 1996c
	maize	0.05	Anon 1996c
	onions	0.05	Anon 1996c
	peanuts	0.05	Anon 1996c
	pears	0.2	Anon 1996c
	persimmon	0.2	Anon 1996c
	potato	0.1	Anon 1996c
	radish	0.1	Anon 1996c
	rice	0.05	Anon 1996c
	soya bean	0.02	Anon 1996c
	sugar cane	0.05	Anon 1996c
	sweet pot	0.05	Anon 1996c
	wheat	0.05	Anon 1996c
Luxembourg	carrot	0.5	Anon 1996c
	maize	0.05	Anon 1996c
	onions	0.5	Anon 1996c

Country	Crop	MRL, mg/kg	Reference
	potato	0.05	Anon 1996c
Netherlands (manufacturer's submission)	bulb veg	0.5	Anon 1996c
	celery	0.5	Anon 1996c
	cereals	0.05	Anon 1996c
	citrus	1	Anon 1996c
	meat	0.2	Anon 1996c
	milk	0.008	Anon 1996c
	mushrooms	0.05	Anon 1996c
	parsley	0.5	Anon 1996c
	peanuts	0.05	Anon 1996c
	potato	0.05	Anon 1996c
	root/tuber veg	0.5	Anon 1996c
	tea	0.2	Anon 1996c
Netherlands (country submission)	citrus	1	Olthof 1996
	root and tuber vegetables	0.5	Olthof 1996
	bulb vegetables	0.5	Olthof 1996
	parsley	0.5	Olthof 1996
	celery leaves	0.5	Olthof 1996
	celery	0.5	Olthof 1996
	other vegetables	0.1	Olthof 1996
	tea	0.2	Olthof 1996
	meat	0.2	Olthof 1996
	milk	0.008	Olthof 1996
	other food commodities	0.05*	Olthof 1996
Poland	citrus fruit	1.0	Anon 1996a
	fruits (other than citrus)	0.05	Anon 1996a
	root vegetables	0.5	Anon 1996a
	potato	0.05	Anon 1996a
	vegetables, other	0.05	Anon 1996a
	mushroom	0.05	Anon 1996a
	rapeseed	0.2	Anon 1996a
	cereal grains	0.05	Anon 1996a
Portugal	brassica	0.1	Anon 1996c
	bulb veg	0.5	Anon 1996c
	citrus	1	Anon 1996c
	fruity veg	0.1	Anon 1996c
	grapes	0.05	Anon 1996c
	leafy veg	0.1	Anon 1996c
	legumes	0.1	Anon 1996c
	mushrooms	0.05	Anon 1996c
	pome fruit	0.05	Anon 1996c
	root/tuber veg	0.5	Anon 1996c
	stem veg	0.5	Anon 1996c
	stone fruit	0.05	Anon 1996c
Spain	brassica	0.1	Anon 1996c
	bulb veg	0.5	Anon 1996c

Country	Crop	MRL, mg/kg	Reference
	citrus	1	Anon 1996c
	fruity veg	0.1	Anon 1996c
	grapes	0.05	Anon 1996c
	leafy veg	0.1	Anon 1996c
	legumes	0.1	Anon 1996c
	mushrooms	0.05	Anon 1996c
	pome fruit	0.5	Anon 1996c
	root/tuber veg	0.5	Anon 1996c
	stem veg	0.5	Anon 1996c
	stone fruit	0.05	Anon 1996c
Switzerland	cabbage	0.1	Anon 1996c
	carrot	0.3	Anon 1996c
	onions	0.01	Anon 1996c
	radish	0.1	Anon 1996c
UK	citrus fruit	1.0	Anon 1994a
	oranges	1.0	Anon 1994a
	apples	0.05	Anon 1994a
	pears	0.05	Anon 1994a
	peaches and nectarines	0.05	Anon 1994a
	plums	0.05	Anon 1994a
	grapes	0.05	Anon 1994a
	strawberries	0.05	Anon 1994a
	raspberries	0.05	Anon 1994a
	blackcurrants	0.05	Anon 1994a
	bananas	0.5	Anon 1994a
	carrots	0.5	Anon 1994a
	swedes	0.5	Anon 1994a
	turnips	0.5	Anon 1994a
	onions	0.5	Anon 1994a
	tomatoes	0.1	Anon 1994a
	cucumbers	0.1	Anon 1994a
	cauliflower	0.1	Anon 1994a
	Brussels sprouts	0.1	Anon 1994a
	cabbage	0.1	Anon 1994a
	lettuce	0.1	Anon 1994a
	beans	0.1	Anon 1994a
	peas	0.1	Anon 1994a
	celery	0.5	Anon 1994a
	leek	0.1	Anon 1994a
	mushrooms	0.05	Anon 1994a
	milk	0.008	Anon 1994a
	meat, fat and preparations of meat	0.2	Anon 1994a

Only the residue definition applying in the UK, The Netherlands and Poland was specified. In these countries the definition is “the sum of *Z*- and *E*- isomers of chlorfenvinphos”.

In 1994 the US EPA proposed to revoke the tolerances in or on certain raw agricultural commodities, processed foods and animal feeds for 17 pesticide chemicals including chlorfenvinphos. The EPA stated that they were initiating this action for those pesticides which have no food use (national) registrations (Anon, 1994b).

APPRAISAL

Chlorfenvinphos is a contact and soil-applied organophosphorus insecticide available as granules, EC or WP sprays and seed-treatment formulations. It is used for the control of various pests, including wheat bulb fly, cabbage root fly and carrot fly, on a range of crops.

Chlorfenvinphos is present in the form of two configurational isomers and is liquid at 25°C. Data on physico-chemical properties were provided only for the technical material. The data on the solubility of chlorfenvinphos in water, fat and organic solvents and the octanol-water partition coefficient, were not supported by full study reports and have therefore not been included in the evaluation.

In briefly reported studies on humans, rats and dogs, chlorfenvinphos was extensively metabolized, and a number of metabolites were identified.

A number of briefly reported metabolism studies on ruminants were submitted in which cows were treated by injection or spraying, but none in which cattle were treated by oral ingestion. A number of metabolites were identified and a metabolic pathway proposed in which it was postulated that incorporation of some of the metabolites took place by conjugation with glucuronide. Most of the radioactive residue was found in the omental or renal fat, with little or no residue in the liver, kidney or other tissues even at high doses. However, these studies were old and briefly reported with limited experimental detail. The Meeting considered that new data on metabolism in lactating ruminants and/or laying poultry to meet modern standards are required if significant residues occur in relevant feed items. In addition, data on the ruminant metabolism of chlorfenvinphos applied externally are required to support the approved use for dipping in Australia.

In plants two main investigations were conducted, one with foliar applications to potatoes, cabbages and maize and the other with soil applications to cabbages, carrots and onions. Significant residues of parent chlorfenvinphos remained in crops sampled several weeks after treatment. The main metabolite from foliar applications was the conjugate of 1-(2,4-dichlorophenyl)ethanol. Traces of desethyl-chlorfenvinphos were also detected. After soil applications the metabolite 2,4-dichloroacetophenone was identified together with some polar unextractable material. These metabolism studies were old and briefly reported with limited experimental detail: the full metabolic pathway in plants was not elucidated. Although the data appeared to show that chlorfenvinphos was the major component of the residue the Meeting considered that new data on metabolism and translocation in plants according to modern standards are required to confirm this.

In a laboratory study of degradation in soil a number of products were identified and a degradation pathway was proposed. Chlorfenvinphos was the major single compound identified although 1-(2,4-dichlorophenyl)ethanol, the sodium salt of desethyl-chlorfenvinphos, and 2,4-dichloroacetophenone were present in significant concentrations. Degradation was slower in organic than in mineral soils. In the field, half-lives of chlorfenvinphos were 14-84 days in mineral soils and more than 150 days in peat soil.

The analysis of crop and soil samples for chlorfenvinphos and its metabolites was based on GLC with FP, EC or NP detection. The reported limits of determination were 0.01-0.05 mg/kg. Only limited data on validation of the methods were presented.

A definition of the residue as "chlorfenvinphos, sum of (*E*)- and (*Z*)- isomers" was recommended, but the Meeting agreed that the definition might have to be reconsidered when new data on plant and animal metabolism have been reviewed.

The information on GAP supplied by the manufacturer was incomplete. No copies of the product labels were submitted, only summary sheets.

Reports of residue trials on leeks, onions, head cabbage, Savoy cabbage, cauliflower, mushrooms, kale, carrots, parsley root, parsnips, potatoes, swedes, sweet potatoes, radishes, turnips, celery, rape seed, parsley, maize, and wheat were submitted, but as no GAP was reported for parsley root or sweet potatoes the Meeting could not estimate maximum residue levels for these commodities. No residue trials were reported on several crops for which GAP and/or CXLs exist, and the Meeting recommended withdrawal of the unsupported CXLs.

Many of the trials were very old with no detailed study reports. Details such as the method of analysis, the duration of sample storage, analytical recoveries and plot size were lacking. The Meeting agreed that such data were inadequate for the estimation of maximum residue levels. In many other trials the duration of sample storage before analysis was not reported and the Meeting agreed that although the data could be used to estimate maximum residue levels, such levels could not be recommended as MRLs because data on the stability of residues in stored analytical samples of representative substrates were required to confirm the validity of the results.

Onions. GAP was reported for several countries. A number of residue trials on bulb onions together with one on spring onions were reported. Four French trials with residues of <0.02 mg/kg complied with the granular application rate in France, but a PHI of 15 days was reported by the manufacturer as French GAP, whereas the PHIs in the trials were 133-182 days. One German trial according to GAP for pre-planting spray treatment in Belgium and The Netherlands gave residues below 0.02 mg/kg after 175 days (shorter PHIs were not considered to accord with GAP). A further five German trials were considered to comply with GAP for pre-planting granular treatments in Belgium, Denmark, Germany and The Netherlands: all residues were below the LOD (<0.02 mg/kg). Two replicated Japanese trials reflected Japanese foliar GAP (which has a low application rate), with residues of <0.02 mg/kg 7-8 days after treatment. The only measurable parent residues reported were from the higher application rate of 4.8 kg ai/ha in a German spray trial (0.04 mg/kg, at a 60-day PHI) and in one UK trial (0.07 mg/kg, PHI of 61 days) which was very old and poorly described with no detailed study report. These trials were not comparable with any reported GAP.

The Meeting estimated an STMR of 0.02 mg/kg and a maximum residue level of 0.02* mg/kg. These estimates were based partly on trials which lacked information on the duration of sample storage.

Cabbage. Registered uses on head cabbage were reported in Belgium, Denmark, France, Germany, Ireland, Italy, Japan, The Netherlands, Sweden, Switzerland, and the UK, and on Savoy cabbage in Germany and The Netherlands. Residue trials on head cabbage were reported from the UK, Germany, the USA and India, and on Savoy cabbage from Germany. Seven German trials on head cabbage and three on Savoy cabbage complied with GAP for pre-planting soil treatments at 0.1 kg ai/m^2. Six further trials on head cabbage reflected the German granular seedbed GAP of 2 g/100 plants and three trials on Savoy cabbage the German 2 kg ai/ha GAP. All residues in all these trials were below 0.02 mg/kg. The German granular treatment at 0.1 g/plant (in some cases in combination with an earlier pre-planting soil treatment at 0.1 kg ai/m^2) was represented by four acceptable trials on Savoy cabbage and one on head cabbage with residues of 0.02, 0.03, 0.15, 0.3 and 0.9 mg/kg. One UK trial complied with UK Gap for pre-emergence sprays but was very old and poorly reported without details. No trials were considered comparable with the GAP for foliar treatments reported in several countries, which have shorter PHIs. The Meeting agreed that there were insufficient data to estimate a maximum residue level on the basis of the German 0.1 g/plant granular treatment. However in view of the many trials conforming to German GAP for pre-planting and seedbed applications, all with residues below 0.02 mg/kg, the Meeting estimated an STMR of 0.02 mg/kg and a maximum residue level of 0.02* mg/kg. The trials on which these estimates were based included some which lacked information on the duration of sample storage and others for which this information was not clear to the reviewer because the study was not reported in the working language of the Meeting.

Cauliflower. GAP was reported for Germany, Ireland, The Netherlands and the UK. Residue trials were carried out in Germany, India, the USA and the UK. There were three German trials according to each of three different German GAP treatments: 2 g/100 plants nursery granular, the 0.1 g/plant single bed treatment and the 2 kg ai/ha granular “spreading” application. The UK and Dutch spray treatment (ca. 4-5 kg ai/ha) at the time of drilling or transplanting was reflected by four German trials. All the residues in these trials were <0.02 mg/kg.

The Meeting estimated an STMR of 0.02 mg/kg and a maximum residue level of 0.02* mg/kg. Again some of the trials had no information on the duration of sample storage and others were not reported in English.

Mushrooms. GAP was reported only for the UK as either compost or casing incorporation. Only one trial was available which was poorly described with no detailed study report. There were insufficient data to estimate an STMR or maximum residue level and the Meeting recommended that the existing CXL of 0.05 mg/kg should be withdrawn.

Kale. There are registered uses in Germany, The Netherlands, Portugal and Spain, but residue trials were available only from Germany. Five trials were according to the Dutch GAP for spray treatments at planting or before sowing. Residues were all <0.02 mg/kg. In one of these trials the residue of dichlorophenylethanol was 0.07 mg/kg. Three further trials complied with the German granular single plant treatment, and in two others this treatment was combined with soil treatment according to German GAP. Residues in these trials were <0.02 (2), 0.02, 0.07 and 0.09 mg/kg. There were insufficient data to estimate an STMR or maximum residue level.

Carrots. GAP was reported for Belgium, Denmark, France, Germany, Ireland, Italy, Luxembourg, The Netherlands, Switzerland and the UK. Residue trials were available from Canada, France, Germany, The Netherlands, South Africa, Spain, Sweden, Switzerland, Trinidad and the UK. In addition the UK government provided data on residues in overwintered commercial carrots whose treatment history had been recorded. The highest residues resulted from post-planting EC or WP sprays at *c*. 4 kg ai/ha which corresponds to GAP in The Netherlands and France. Similar treatments at *c*. 2.5 kg ai/ha are GAP in Ireland and the UK. The PHIs reported for these countries ranged between 21 and 60 days which reflects second generation carrot fly control. French GAP was also reported to include an EC spray at 5 kg ai/ha with a PHI of 15 days, but the Meeting was informed that the use in practice was at the time of sowing. Several trials in France, Germany and The Netherlands complied with the higher rate GAP, with residues of <0.02, 0.05, 0.08, 0.12, 0.14, 0.2(3), 0.22, 0.3, 0.37, 0.45, 0.9, 1.2, 1.8, 2.0, and 3.8 mg/kg. In the overwintered commercial carrots treated in accordance with UK GAP the residues were <0.02-1.6 mg/kg.

The Meeting estimated an STMR of 0.22 mg/kg and a maximum residue level of 5 mg/kg. This estimation was based in part on trials for which no information on the duration of sample storage was reported.

Parsnips. GAP was reported for The Netherlands and the UK. The UK provided government-generated data on residues in overwintered commercial parsnips of known treatment history. Two residues were from treatments according to UK GAP (2.35 kg ai/ha). The residues were 0.14 and 0.16 mg/kg. The estimates of the STMR and maximum residue level for carrots are based on the post-planting EC or WP spray at 4 kg ai/ha reported as GAP in The Netherlands. Since GAP for parsnips in The Netherlands is the same as for carrots the Meeting agreed that the data on carrots could be used to estimate maximum and mean residue levels for parsnip by extrapolation.

The Meeting estimated an STMR of 0.22 mg/kg and a maximum residue level of 5 mg/kg. The estimates were based in part on trials for which there was no information on the duration of sample storage.

Potatoes. There are registered uses in The Netherlands and Poland. Residue trials were carried out in the UK, Spain, Australia and Poland, but they were very old and poorly reported with few details. There were insufficient data to estimate an STMR or maximum residue level and the Meeting recommended that the existing CXL of 0.05 mg/kg should be withdrawn.

Radishes. GAP was reported for Germany, The Netherlands and the UK. Residue trials were in Germany and Switzerland. Several of the trials were very old and none were reported in detail. In addition the UK provided government-generated data on residues (four results) in overwintered commercial radishes of known treatment history. The residues following applications close to GAP were all <0.1 mg/kg. There were insufficient data to estimate an STMR or maximum residue level and the Meeting recommended that the existing CXL of 0.1 mg/kg should be withdrawn.

Swedes and turnips. GAP for swedes and turnips was reported for The Netherlands and the UK. One field trial in the UK on swedes and three in the UK or USA on turnips were reported, but the analytical recovery was high (>120%) in the trial on swedes and the others were old and poorly described with no detailed study reports. The Meeting also received reports of six German trials on swedes or turnips in which the commodity was described as "turnip cabbage". This was an error in translation from the original German and the correct description was "swede/turnip". These trials did not comply with UK or Netherlands GAP.

There were insufficient data to estimate an STMR or maximum residue level and the Meeting recommended that the existing CXLs of 0.05 mg/kg should be withdrawn.

Celery. There is a registered use in The Netherlands. One group of residue trials was reported, at a unspecified location. It was poorly described, with no detailed study report.

There were insufficient data to estimate an STMR or maximum residue level and the Meeting recommended that the existing CXL of 0.4 mg/kg should be withdrawn.

Rape seed. GAP for rape was reported for Austria, Germany, The Netherlands and Poland. Several field trials were carried out in France and Germany. Six German trials complied with German GAP for EC spray. Residues in all the trials were <0.02 mg/kg. There were no trials with the broadcast application of granules at 3 kg ai/ha used in The Netherlands, although in two French trials with an application rate of 1kg ai/ha residues were <0.02 mg/kg.

The Meeting estimated an STMR of 0.02 mg/kg and a maximum residue level of 0.02* mg/kg. The estimates were based on trials without information on the duration of sample storage.

Parsley. There are registered uses in The Netherlands and the UK with WP or EC spray applications. Summarized reports of residue trials were available from Germany, but all the trials were with granular formulation whereas the reported GAP applications are by spraying.

There were insufficient data to estimate an STMR or maximum residue level.

Maize. GAP was reported for The Netherlands. Residue trials were carried out in France but were very old and poorly described with no detailed study reports.

There were insufficient data to estimate an STMR or maximum residue level and the Meeting recommended that the existing CXL of 0.05 mg/kg should be withdrawn.

Wheat. There are registered uses in the UK. Two residue trials in the UK were very old and poorly reported with inadequate detail.

There were insufficient data to estimate an STMR or maximum residue level and the Meeting recommended that the existing CXL of 0.05 mg/kg should be withdrawn.

Lettuce and lamb's lettuce as rotational crops. Trials were carried out in Germany, but the data were submitted in JMPR summary format only with no accompanying study reports.

The lettuce or lamb's lettuce was planted 1-4 months after the treatment of radishes as the primary crop at 4 kg ai/ha. The dates of harvest of the radish crop and the residue levels in the soil were not recorded. The residues in lamb’s lettuce at harvest were <0.04 (4) and 0.19 mg/kg, and in lettuce <0.04 (5), 0.05, 0.07 0.11 mg/kg. German GAP for radishes is a “spreading” application at 3 kg ai/ha (field) or 4 kg ai/ha (glass). Similar GAP for soil treatment was reported at comparable application rates for several other crops in a number of countries.

Although no GAP was reported for chlorfenvinphos on lettuce or lamb’s lettuce, the trials demonstrated that significant residues may occur in these crops when grown in rotation following soil applications of chlorfenvinphos. Since the trials were reported only in summary form, the Meeting agreed not to estimate a maximum residue level for lamb’s lettuce or head lettuce.

Livestock. In a briefly reported trial calves were dipped in a chlorfenvinphos solution at a concentration of 0.037 kg ai/hl. Residues in liver, muscle and kidney were below the LODs of 0.1, 0.05 and 0.05 mg/kg respectively, but residues in the fat were in the range <0.1-0.27 mg/kg. In a trial in which cattle were grazed on treated pasture containing residues of 2.5-17 mg/kg the residues of chlorfenvinphos in the milk were all below 0.01 mg/kg.

The Meeting concluded that there were insufficient data on residues in ruminant feed items to estimate maximum residue levels for the meat, milk or edible offal of ruminants and that the existing CXLs for meat and milk should be withdrawn.

Domestic preparation and processing trials indicated that most of the residue in carrots treated with an EC spray is associated with the crown and the top 1 cm of the root. Removal of the crown alone was reported to lead to the loss of approximately 30% of the residue. Domestic boiling was found to have only a moderate effect on residues, but when carrots were peeled and the top of the roots (crown and next 1 cm) removed only 1-3% of the total residue remained. In a further study residues of 0.07 mg/kg in raw carrots were reduced to 0.02 mg/kg by commercial cooking, which included the addition of brine.

National monitoring data were supplied from Australia, Poland, The Netherlands and the UK.

The Meeting agreed that in view of the lack of studies according to modern standards on metabolism, the stability of residues in stored analytical samples, the mobility of chlorfenvinphos in soil and the residues found in following crops, the estimated maximum residue levels could not be recommended as MRLs. For any further future consideration of MRLs, submission of data on such studies would be needed.

RECOMMENDATIONS

1. The Meeting estimated the following maximum residue levels and STMRs, but the maximum residue levels are not recommended for use as MRLs.

Definition of the residue for compliance with MRLs and for estimation of dietary intake: chlorfenvinphos, sum of (*E*)- and (*Z*)- isomers.

The residue is fat-soluble.

Commodity		Maximum residue level, mg/kg	STMR, mg/kg
CCN	Name		
VB 0041	Cabbages, head	0.02*	0.02
VR 0577	Carrot	5	0.22
VB 0404	Cauliflower	0.02*	0.02
VA 0385	Onion, Bulb	0.02*	0.02
VR 0588	Parsnip	5	0.22
SO 0495	Rape seed	0.02*	0.02

2. The Meeting recommended that the following existing CXLs should be withdrawn.

Commodity		Existing CXL, mg/kg
CCN	Name	
VB 0400	Broccoli	0.05
VB 0402	Brussels sprouts	0.05
VB 0041	Cabbages, Head	0.05
VR 0577	Carrot	0.4
VB 0404	Cauliflower	0.1
VS 0624	Celery	0.4
FC 0001	Citrus fruits	1
SO 0691	Cotton seed	0.05
VO 0440	Egg plant	0.05
VR 0583	Horseradish	0.1
VA 0384	Leek	0.05
GC 0645	Maize	0.05
MM 0095	Meat (from mammals other than marine mammals)	0.2 (fat) V
ML 0107	Milk of cattle, goats and sheep	0.008 F V
VO 0450	Mushrooms	0.05
VA 0385	Onion, Bulb	0.05
SO 0697	Peanut	0.05
VR 0589	Potato	0.05
VR 0494	Radish	0.1
GC 0649	Rice	0.05
CM 1205	Rice, polished	0.05
VR 0497	Swede	0.05
VR 0508	Sweet potato	0.05
VO 0448	Tomato	0.1
VR 0506	Turnip, Garden	0.05
GC 0654	Wheat	0.05

FURTHER WORK OR INFORMATION

Desirable

1. The following physico-chemical properties of the pure active ingredient:
vapour pressure, melting point, octanol/water partition coefficient, solubility in organic solvents, solubility in water, specific gravity.

2. If significant residues occur in relevant feed items, a study of metabolism and distribution in a lactating ruminant and/or in laying poultry carried out according to modern standards in which treatment is made through oral ingestion.

3. Data on metabolism in a ruminant after the external application of chlorfenvinphos to support the reported approved dipping use in Australia.

4. Plant metabolism and translocation studies carried out according to modern standards.

5. Studies on the stability of pesticide residues in representative analytical samples stored for at least two years. These would help to support data evaluated by the Meeting on residue trials for which the duration of sample storage was not reported.

6. Studies to assess the nature and levels of residues in representative rotational crops other than lettuce and lamb’s lettuce.

7. If significant residues are found in animal feed, a transfer study on ruminants according to modern standards (see 1993 JMPR report, Section 2.7).

8. A study of the mobility of chlorfenvinphos in soil, including leaching, adsorption and desorption, according to modern standards.

9. Copies of the product labels supporting the information submitted on GAP.

10. The full reports of the rotational crop studies on lamb's lettuce and lettuce.

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DDT (021)

EXPLANATION

DDT was first evaluated in 1966 and has been reviewed several times since. At the 22nd (1990) and 23rd (1991) Sessions of the CCPR countries were requested to supply monitoring and other relevant data on DDT (ALINORM 91/24, para 77; ALINORM 91/24A, para 77). At the 23rd Session the existing Extraneous Maximum Residue Limits (ERLs) for DDT were converted to temporary limits awaiting evaluation by the 1993 JMPR. The 1993 JMPR proposed ERLs for carrots, eggs, meat and milks and the 1994 JMPR confirmed the existing TERL for cereal grains on the basis of the available data. The 1995 CCPR was informed that additional data on meat were available from Australia, New Zealand and the USA and decided to keep the proposal for meat at Step 3 pending the evaluation of these data by the 1996 JMPR. The 28th (1996) Session of the CCPR advanced all ERLs except that for meat to Step 8 (ALINORM 97/24, para 85).

The Meeting received national residue survey data on DDT in animal products from Australia (Anon., 1995a), Germany (Anon., 1995b), New Zealand (Anon., 1994; Jowett and Viggers, 1995), Norway (Anon., 1996), Thailand (Anon., 1995c) and the USA (Anon., 1995d) and on DDT in food of plant origin from Norway (Anon., 1996) and The Netherlands (Anon, 1995e). The Netherlands also submitted information on analytical methods and national MRLs (Anon., 1995e). Poland provided information on national MRLs (Anon., 1995f). The British annual report of the Working Party on Pesticides Residues for 1994 included information on DDT residues (Anon., 1995g). Residue data and information on the dietary intake of DDT were made available by the Global Environment Monitoring System - Food Contamination Monitoring and Assessment Programme (GEMS/Food) of WHO (WHO, 1996; Moy, 1996a,b).

USE PATTERN

No information was supplied by governments on registered or recommended uses of DDT in agriculture. An evaluation by the Pesticide Action Network (PAN, 1991) showed that DDT is banned (all uses prohibited by final regulatory action owing to health or environmental hazards) in 29 countries, severely restricted (most uses prohibited owing to health or environmental hazards, certain specific uses remain authorized) in 23 countries, and unregistered (no registered uses, but not explicitly banned) in 4 countries.

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

The presentation of the data received differed from country to country, and the layouts in the Tables are consequently different. With the exception of the Australian data all the residues are expressed as the sum of *p,p*¢DDT, *o,p*¢DDT, *p,p*¢DDE and *p,p*¢TDE (*p,p*¢DDD), in conformity with the Codex definition. In the Australian survey the residues of DDT, DDE and TDE were reported separately.

Monitoring of meat in Australia (Table 1). Residues of *p,p′*- and *o,p′*-DDT, *p,p′*-DDE and *p,p′*-TDE were reported for the period January 1989-December 1994 by the National Residue Survey of Australia. The reporting limit was 0.1 mg/kg for each compound.

Table 1. Residues of DDT in meat in Australia, 1989-1994 (Anon., 1995a).

Commodity	Compound	No. of samples	No. of residue-free samples	No. of samples with trace only[1]	No. of samples with residues, mg/kg, in ranges 0.1-1	1.1-2.5	2.6-5	5.1-10	>10
Beef (fat)	DDT	39854	39730	60	61	1	1		1
	DDE	39854	37149	1283	1394	24	3	1	
	TDE	39854	39752	47	53	2			
Buffalo (fat)	DDT	432	432						
	DDE	432	428	1	3				
	TDE	432	438	1	1				
Deer (fat)	DDT	110	110						
	DDE	110	106	3		1			
	TDE	110	110						
Emu (fat)	DDT	9	9						
	DDE	9	7	2					
	TDE	9	9						
Game goat (fat)	DDT	87	87						
	DDE	87	87						
	TDE	87	87						
Goat (fat)	DDT	927	925		2				
	DDE	927	912	4	7				
	TDE	927	924	3					
Horse (fat)	DDT	1939	1926	4	9				
	DDE	1939	1837	31	70	1			
	TDE	1939	1936		2	1			
Kangaroo (fat)	DDT	482	480	2					
	DDE	482	473	3	6				
	TDE	482	482						
Sheep (fat)	DDT	29270	29169	59	41				
	DDE	29270	25604	1336	2314	13			
	TDE	29270	29208	33	28	1			
Porcine (fat)	DDT	15900	15761	62	74	2		1	
	DDE	15900	15257	427	210	5	1		
	TDE	15900	15814	44	40	2			
Poultry (fat)	DDT	2167	2161	4	2				
	DDE	2167	2007	151	9				
	TDE	2167	2165	1	1				
Rabbit (fat)	DDT	570	570						
	DDE	570	566	4					
	TDE	570	562	7	1				

[1] Trace only: unquantifiable amount between the limit of detection and the reporting limit (0.1 mg/kg)

Monitoring of meat in Germany (Table 2). Information was supplied on residues of DDT from monitoring carried out in 1993. Residue data were expressed on the fat and raw product basis with a limit of determination of 0.001 mg/kg. The commodities were classified according to Council Directive of 24 July 1986, 86/363/EEC, modifying Council Directive 93/57/EEC of 29 June 1993 (EEC, 1986, 1993).

Table 2. Residues of DDT in meat in Germany, 1993 (Anon., 1995b).

Commodity according to 86/363/EEC	No. of samples	No. of samples with DDT residues , mg/kg in range <0.001	0.001 - 0.01	0.002 - 0.015	0.011 - 0.02	0.016 - 0.05	0.021 - 0.1	0.051 -0.2	0.11 -0.5	0.21 -1	0.6 -2	1.1 -5	2.1	max., mg/kg
Meat, except sheep (fat) ex 02.01	777	128		87	54	102	230	119	39	17	1			0.5
Meat, except	1080	618	192	221	36	7	3	2		1				0.42

Commodity according to 86/363/EEC	No. of samples	No. of samples with DDT residues , mg/kg in range												max., mg/kg
		<0.001	0.001 - 0.01	0.002 - 0.015	0.011 - 0.02	0.016 - 0.05	0.021 - 0.1	0.051 -0.2	0.11 -0.5	0.21 -1	0.6 -2	1.1 -5	2.1	
sheep (fresh substance) ex 02.01														
Sheep, meat (fat) ex 02.01	87	6		2	4	14	18	11	24	6	1	1		1.01
Sheep, meat (fresh substance) ex 02.01	30	8	3	9	2	5	3							0.033
Poultry, meat and edible offal (fat) 02.02	74	17		3	13	19	15	4	3					0.19
Poultry, meat and edible offal (fresh substance) 02.02	63	42	10	11										0.005
Poultry, liver (fresh substance) 02.03	14	11		3										0.003
Rabbit etc., meat (fat) ex 02.04	15				2	2	3	3	1	1	3			0.79
Rabbit etc., meat (fresh substance) ex 02.04	140	39	35	44	9	8	4	1						0.078
Bacon (pork fat), poultry fat ex 02.05	671	220		37	74	147	131	50	8	2			2	3.9
Meat salted, meat dried (fat) 02.06	29	17			6	3	3							0.045
Meat salted, meat dried (fresh substance) 02.06	6	4	1	1										0.002

Commodities according to Council Directive 86/363/EEC:
ex 02.01: Meat and edible offal of horses, asses, mules,bovine animals, swine, sheep and goats, fresh, chilled or frozen
02.02: Dead poultry and edible offal thereof (except liver), fresh, salted or in brine
02.03: Poultry liver, fresh, chilled, frozen, salted or in brine
ex 02.04: Other meat and edible offal, fresh, chilled or frozen, of domestic pigeons, domestic rabbits and game
ex 02.05: Pig fat and poultry fat, fresh, chilled, frozen, salted, in brine, dried or smoked
02.06: Meat and edible offal (except poultry liver), salted, in brine, dried or smoked

Monitoring of meat in New Zealand. The results of residue monitoring from July 1990 to June 1994, including routine testing (Table 3) and specifically targeted sampling from regions with a known DDT history (Table 4) were submitted.

Table 3. Residues of DDT in meat in New Zealand, 1990-1994 (Anon., 1994).

Commodity, Year	No. of samples analysed	No. positive[1]	No. of samples with residues, mg/kg fat, in range					DDT, mg/kg		
			0.02 -0.5	0.51 -1.0	1.01 -2.0	2.1 -5.0	>5	Mean	Median	Max.
Lambs										
1990-1991	244	123	119	1	3			0.13	0.05	1.4
1991-1992	159	95	84	4	7			0.24	0.11	1.7
1992-1993	261	138	127	7	2	2		0.19	0.07	3.7
1993-1994	301	178	161	13	4			0.19	0.08	1.5
Sum	965	534	491	25	16	2				
% of no. analysed		55.5	51	2.6	1.7	0.2				
Adult sheep										
1990-1991	203	85	77	5	1	2		0.21	0.07	3.3
1991-1992	158	87	78	5	4			0.2	0.07	1.5
1992-1993	84	37	34	2	1			0.2	0.09	1.2
1993-1994	103	68	61	3	2	2		0.24	0.06	2.6
Sum	548	277	250	15	8	4				
% of no. analysed		50.5	46	2.7	1.5	0.7				
Adult bovine										
1990-1991	202	70	69	1				0.085	0.04	0.73
1991-1992	261	125	117	5	3			0.14	0.06	1.3
1992-1993	132	47	47	1				0.08	0.04	0.89
1993-1994	164	77	71	4	2			0.16	0.08	1.4
Sum	739	319	304	11	5					
% of no. analysed		43	41	1.5	0.68					
Suckling calves										
1990-1991	306	201	175	18	8			0.23	0.11	1.3
1991-1992	309	246	206	26	7	7		0.31	0.13	4.1
1992-1993	310	193	188	5				0.13	0.09	0.98
1993-1994	301	217	199	9	6	2	1	0.29	0.15	5.2
Sum	1211	857	768	58	21	9	1			
% of no. analysed		71	63	4.8	1.7	0.74	0.08			
Pigs										
1990-1991	232	88	85	3				0.07	0.03	0.62
1991-1992	305	180	175	1	2	2		0.12	0.045	3.1
1992-1993	288	170	161	4	3	1	1	0.17	0.04	6.2
1993-1994	100	69	66	2	1			0.12	0.05	1.45

Commodity, Year	No. of samples analysed	No. positive[1]	No. of samples with residues, mg/kg fat, in range					DDT, mg/kg		
			0.02 -0.5	0.51 -1.0	1.01 -2.0	2.1 -5.0	>5	Mean	Median	Max.
Sum	925	507	487	10	6	3	1			
% of no. analysed		55	53	1.1	0.65	0.32	0.11			
Deer										
1990-1991	10	6	4	1						
1991-1992	17	4	3	1						
1992-1993	15	6	6					0.17	0.16	0.32
1993-1994	185	102	91	9	2			0.17	0.07	1.2
Sum	227	118	104	11	2					
% of no. analysed		52	46	4.8	0.88					
Goats										
1990-1991	7	2	2							
1991-1992	17	4	4							
1992-1993	13	4	4							
1993-1994	30	1	1							
Sum	67	11	11							
% of no. analysed		16	16							

[1] ≥0.02 mg/kg

Table 4. Residues of DDT in meat from lambs in New Zealand from a region with known DDT history, 1992-3 (Anon., 1994).

	No. of samples analysed	No. positive[1]	No. of samples with residues, mg/kg fat, in range					DDT, mg/kg		
			0.02-0.5	0.51-1.0	1.01-2.0	2.1-5.0	>5	Mean	Median	Max.
% of no. analysed	403	396	183	82	60	58	13	1.2	0.64	13
		98	45.4	20.3	14.9	14.4	3.2			

[1] ≥0.02 mg/kg

Monitoring of meat in Norway (Table 5). In total, 1568 samples of meat were analyzed in the period from 1990 to 1994. Residues were detected (LOD 0.02 mg/kg) in only 2 samples, both from 1990: 1 sample of pig meat and 1 sample of cattle meat contained 0.023 and 0.315 mg/kg of *p,p*¢DDE in the fat respectively.

Table 5. Residues of DDT in meat in Norway, 1990-1994 (Anon., 1996).

Commodity	No. of samples	No.of samples with DDT residues (mg/kg fat)	
		<0.02	0.02-0.5
Bovines (fat)	537	536	1
Pigs (fat)	537	536	1
Sheep (fat)	149	149	
Hens (fat)	145	145	
Reindeer (fat)	31	31	
Moose (fat)	169	169	

Monitoring of meat in Thailand (Table 6). Data on residues of DDT in meat during the period 1993-1994 were submitted by Thailand without reference to the expression of residues. However since the Codex commodity numbers were given for chicken meat (PM0840), duck meat (PM0841), pig meat (MM0818) and cattle meat (MM0812), it could be assumed that the residues were expressed on a fat basis, corresponding to the Codex expression of the residues of fat-soluble pesticides.

Table 6. Residues of DDT in meat in Thailand, 1993 and 1994 (Anon., 1995c).

Commodity, year	No. of samples	No. of samples with DDT residues, mg/kg, in range				
		<0.01	0.01-0.05	0.06-0.1	0.11-0.5	0.51-1
Chicken meat, 1993	9514	47	3618	3481	2342	26
Chicken meat, 1994	14650	457	9475	3669	1027	22
Duck meat, 1993	2291	27	1778	395	90	1
Duck meat, 1994	1810	19	1575	199	17	
Cattle meat, 1993	30	2	23	2	3	
Cattle meat, 1994	123	2	94	16	11	
Pig meat, 1993	65	1	48	10	6	
Pig meat, 1994	157	1	129	19	8	

Monitoring of meat in the USA. The 1993 JMPR evaluated only the data on DDT residues in imported meat. The present Meeting received new information on the monitoring of domestic and imported samples for the years 1991, 1992 and 1993, shown in Tables 7-10. All residue values are expressed on a fat basis. The reporting limit was 0.01 mg/kg.

Table 7. Residues of DDT in meat in the USA, 1991 (Anon., 1995d).

Species	No. of samples	No. of violations	No. of samples with DDT residues, mg/kg fat, in range								Total No. positive
			0.01- 0.1	0.11-0.2	0.21-0.3	0.31-0.5	0.51-1.0	1.01-2.5	2.51-5.0	>5.0	
Horse	106	0	1								1
Bull	25	0									0
Steer	1967	0	27	10	2	3	2	1			45
Beef cow	272	0	3	1	3	1					8
Heifer	1327	0	18	5	1		1	1			26
Dairy cow	217	0	4		1	1					6
Formula-fed calf	319	0									0
Non-formula calf	243	0	3	1			1				5
Heavy calf	280	0	3	3	1	1					8
Mature sheep	27	0									0
Lamb	320	0	2	1	3						6
Goat	94	1		1						1	2
Market hog	329	0	1		1		1				3
Boar or stag	61	0	1								1
Sow	253	0	3	1				1	1		6
Young chicken	3197	0	1	1							2
Mature chicken	338	0	1								1
Young turkey	3267	0	23	1							24
Mature turkey	205	0	2		1						3
Duck	108	0									0
Goose	62	0	4								4
Rabbit	81	0	1	1	1						3

Table 8. Residues of DDT in meat in the USA, 1992 (Anon., 1995d).

Species	No. of samples	No. of violations	No. of samples with DDT residues, mg/kg fat, in range								Total No. positive
			0.01-0.1	0.11-0.2	0.21-0.3	0.31-0.5	0.51-1.0	1.01-2.5	2.51-5.0	>5.0	
Horse	98	0	2		1		2				5
Bull	1	0									0
Steer	196	0	4	5	1	1	1				12
Beef cow	56	0	2	2		1					5
Heifer	131	0	3		1						4
Dairy cow	240	0	23	9	5		3				40
Formula-fed calf	334	0	3								3
Non-formula calf	293	0	3	7	2			1			13
Heavy calf	295	0	29	15	2	1	1		1		49
Mature sheep	25	0	2	1							3
Lamb	317	0	13	10	4	7	4	1			39
Goat	104	0	2	1			1	1			5
Market hog	3285	2	42	20	12	11	4	2		2	93
Boar or stag	60	0	1	2	3						6
Sow	259	0	8	3	1	1	2		1		16
Young chicken	415	0									0
Mature chicken	321	0	3		1						4
Young turkey	319	0	13								13
Mature turkey	203	0	12	1							13
Duck	109	0									0
Goose	38	0	1								1
Rabbit	71	0	1								1

Table 9. Residues of DDT in meat in the USA, 1993 (Anon., 1995d).

Species	No. of samples	No. of violations	No. of samples with DDT residues, mg/kg fat, in range								Total No. positive
			0.01- 0.1	0.11-0.2	0.21-0.3	0.31-0.5	0.51-1.0	1.01-2.5	2.51-5.0	>5.0	
Horse	425	0	17	14	2	6	3				42
Bull	555	0	13	12	7			2			34
Steer	523	0	13	10		3	3				29
Beef cow	651	0	23	13	4	5	1				46
Heifer	546	0	11	8	1	1	1				22
Dairy cow	272	0	15	17	11	7	2				52
Formula-fed calf	529	0	3			1					4
Non-formula calf	458	0	8	4	2	5	1	1			21
Heavy calf	498	0	52	18	7	3	2	3			85
Mature sheep	470	0	22	8	8	3	2	2			45
Lamb	574	0	39	29	7	4	2				81
Goat	533	0	17	10	2	2	1				32
Market hog	499	0	3	4	3	1					11
Boar or stag	452	1	7	2	3	1	1	1		1	16
Sow	537	0	12	6	4	3	1				26
Young chicken	498	0	3								3
Mature chicken	457	0	9	1							10
Young turkey	519	0	10			1					11

Species	No. of samples	No. of violations	No. of samples with DDT residues, mg/kg fat, in range								Total No. positive
			0.01- 0.1	0.11-0.2	0.21-0.3	0.31-0.5	0.51-1.0	1.01-2.5	2.51-5.0	>5.0	
Mature turkey	228	0	4								4
Duck	322	0		1							1
Goose	3	0									0
Rabbit	84	0	2	1							3

Monitoring data for the period January-December 1994 are summarized in Table 10. The reporting limit was 0.04 mg/kg. All residues are expressed on a fat basis.

Table 10. Residues of DDT in meat in the USA, 1994 (Anon., 1995d).

Species	No. of samples	No. of samples with DDT residues, mg/kg fat, in range								
		<0.04	0.04-0.1	0.11-0.2	0.21-0.3	0.31-0.5	0.51-1	1.01-2.5	2.51-5	>5
Cattle	3955	3657	151	66	39	31	7	2	1	1
Pigs	1457	1346	57	27	14	8	3	1		1
Horses	217	213	1	2						
Poultry	1990	1973	13	3		1				
Sheep and goats	900	692	91	55	27	15	18	2		

Monitoring of dairy products in New Zealand (Table 11). DDT residues found in the monitoring of dairy products carried out from June 1992 to May 1994 are shown in Table 11. In samples with a fat content of 2% or more "total DDT and metabolites" occurred only as DDE. Two anomalously high residues in cheese of 0.48 and 0.55 mg/kg in the fat, were both considered to be due to stress of the animals caused by an early summer snow storm. In addition to butter, cheese and fortified milk (baby food) the products analysed were anhydrous milk fat (99.9% fat), buttermilk powder (12% fat), whole milk powder (26% fat), whey protein concentrate (1.2% fat), skim milk powder (max. 1.2% fat), casein, caseinate, total milk protein (max. 1.2% fat), lactose and lactalbumin. In all, 2915 samples were analysed (Table 11, last line) using an analytical method with a limit of determination of 0.01 mg/kg.

Table 11. Residues of DDT in dairy products in New Zealand, June 1992 - May 1994 (Jowett and Viggers, 1995).

Commodity	No. of samples	No. of samples with DDT residues, mg/kg fat, in range					Max. residue, mg/kg fat
		<0.01	0.01-0.099	0.1-0.19	0.2-0.49	0.5-1.0	
Butter	180	29	142	9			0.13
Cheese	398	42	277	61	17	1	0.55
Fortified milk, Baby food	295	144	148	3			0.16
All product groups combined[1]	2915	966	1748	171	29	1	0.55

[1] Butter, cheese, fortified milk, anhydrous milk fat, buttermilk powder, whole milk powder, whey protein concentrate, skim milk powder, casein, caseinate, total milk protein, lactose, lactalbumin

Monitoring of foods of plant origin in Norway. In Norway, domestic and imported food is routinely

analysed for residues of DDT. In the period 1985-1995 12,682 samples of fruit, vegetables and potatoes were analysed with a limit of determination of 0.05 mg/kg (Anon., 1996). Residues were found only in a single sample of imported table grapes in 1994 which contained 1.2 mg/kg DDT, probably owing to illegal use. Results from the monitoring of cereal grains (wheat 411, rye 70, oats 15 samples) during the period 1990-1995 were also received. The residues were below the Norwegian national MRL (before 1994 0.1 mg/kg, since 1994 0.05 mg/kg).

Monitoring of food of plant origin in The Netherlands. The 1993 JMPR reported monitoring data on DDT residues for the period 1987-1991. The present Meeting received additional information for 1991-1993 and 1994 (Tables 12 and 13). In addition 167 samples of tea were analysed during 1991-1993: four samples contained residues of DDT above the LOD of 0.05 mg/kg but below the Dutch MRL of 1 mg/kg.

Table 12. Residues of DDT in foods of plant origin in The Netherlands, 1991-1993 (Anon., 1995e).

Product	No. of samples	No. of samples with DDT residues, mg/kg, in range		
		<0.05	0.05-0.09	0.1 or higher
Berries and small fruit: grapes	999	994	5	
Miscellaneous fruit: kiwifruit	309	307	2	
Root and tuber vegetables: carrots	609	606	3	
Fruiting vegetables: sweet peppers	1129	1127	2	
cucumbers	644	222	2	
Leaf vegetables and fresh herbs: lambs lettuce	310	305	5	
iceberg lettuce	557	553	4	

Table 13. Residues of DDT in foods of plant origin in The Netherlands, 1994 (Anon., 1995e).

Product	No. of samples	No. of samples with DDT residues, mg/kg, in range		
		<0.025	0.025-0.049	≥0.05
Berries and small fruit	103	101	2	
Root and tuber vegetables: carrots	144	143	1	
Stem vegetables: celery	209	208	1	
Fruiting vegetables: tomatoes	332	331	1	
melons	164	164		
Leaf vegetables and fresh herbs: iceberg lettuce	185	184	1	
parsley	149	148	1	
Pulses	23	22	1	

UK monitoring data. Table 14 shows the results of the 1994 UK monitoring programme for DDT residues (Anon., 1995g).

Table 14. Residues of DDT in foods in the UK, 1994 (Anon., 1995g).

Commodity	No. of samples	Residue or range, mg/kg, and (no. of samples)	Basis	MRL, mg/kg	Remarks
Bread	53	<0.01 (53)			
Milk	202	<0.0008 (201), 0.002 (1)	whole product	0.04	residue present as *p,p*¢DDE
Apple, dessert Apple, cooking Apple, juice	25 UK 48 imported 29 UK 40 UK 9 imported	<0.05 (73) <0.05 (29) <0.05 (49)			
Asparagus	6 UK 20 imported	<0.01 (26)			
Lettuce imported UK produced	 31 56	 <0.01 (30), 0.02 (1, France) <0.01 (56)		0.05	residue present as *p,p*¢DDE
Infant cereals Wheat germ Wheat bran	12 8 11	<0.01 (12) <0.01 (8) <0.01 (11)			
Cream UK produced unknown origin	 30 6	 <0.04 (30) <0.04 (6)	 fat fat		
Chocolate UK produced imported	 62 2	 <0.02 (62) <0.02 (2)	 fat fat		
Honey UK produced imported blended	 20 60 20	 <0.008 (20) <0.008 (59), 0.009 (1, Mexico) <0.008 (20)	 whole product		residue present as *p,p′*-DDT and *o,p′*-DDT
Vegetable oils imported unknown origin	 57 16	 <0.02 (62) <0.02 (2)	fat		
Goat cheese UK produced imported unknown origin Goat milk	 11 11 6 8	 <0.01 (11) <0.01 (11) <0.01 (6) <0.004 (8)	 fat fat fat whole product		
Pig kidney UK produced imported unknown origin	 11 1 11	 <0.008 (11) <0.008 <0.008 (11)	 whole product		
Lamb kidney UK produced imported unknown origin	 11 6 8	 <0.008 (9), 0.009 (2) <0.008 (6) <0.008 (8)	 whole product	0.1	1st sample:0.009 *p,p′*-DDE, 2nd sample: 0.003 *p,p′*-TDE and 0.006 *p,p′*-DDE
Ox kidney UK produced unknown origin	 15 16	 <0.008 (15) <0.008 (16)	 whole product		
Lamb, imported	74	<0.01 (38), 0.01-0.09 (23) 0.1-0.3 (12), 0.8	fat	1	residue present as *p,p′*-DDE
Lamb liver UK produced imported	 12 5	 <0.008 (12) <0.008 (5)	 whole product		

Commodity	No. of samples	Residue or range, mg/kg, and (no. of samples)	Basis	MRL, mg/kg	Remarks
unknown origin	7	<0.008 (7)			
Chicken liver UK produced unknown origin	 24 7	 <0.008 (24) <0.008 (6), 0.01	whole product	0.1	residue present as *p,p′*-DDE
Ox liver UK produced unknown origin	 10 14	 <0.008 (10) <0.008 (14)	whole product		
Cattle, kidney fat UK produced	 116	 <0.01 (108), 0.01-0.05 (8)	fat		residue present as *p,p′*-DDE
Pig, kidney fat UK produced	 114	 <0.01 (108), 0.01-0.05 (6)	fat		residue present as *p,p′*-DDE
Sheep, kidney fat UK produced	 114	 <0.01 (96), 0.01-0.06 (18)	fat		residue present as *p,p′*-DDE
Pheasant UK produced unknown origin	 46 22	 <0.008 (42), 0.009-0.01 (4) <0.008 (21), 0.008	whole product	0.1	residue present as *p,p′*-DDE
Rabbit UK produced imported unknown origin	 29 32 (China) 1	 <0.004 (26), 0.007-0.008 (3) <0.008 (23), 0.009, 0.01-0.04 (8) 0.09	whole product	0.1	residue present as *p,p′*-DDE
Wood pigeon UK produced unknown origin	 17 13	 <0.004 (8), 0.004-0.007 (8), 0.5 <0.004 (7), 0.004-0.002 (6)	whole product	0.1	0.5 mg/kg: 0.01 *p,p′*-DDE, 0.04 *p,p′*-TDE, 0.02 *p,p′*-DDT
Fish paté UK produced imported unknown origin	 19 1 22	 <0.008 (12), 0.01-0.09(7) <0.008 <0.008 (10), 0.009-0.05 (12)	whole product		residue present as *p,p′*-DDE, *p,p′*-TDE and *p,p′*-DDT
Fish paste UK produced imported unknown origin	 16 1 (Italy) 26	 <0.004 (8), 0.004-0.009 (6), 0.01-0.02 (2) 0.02 <0.004 (15), 0.004-0.009 (9), 0.01 (2)	whole product		residue present as *p,p′*-DDE, *p,p′*-TDE and *p,p′*-DDT
Eel (common) UK produced unknown origin Eel (conger) unknown origin	 15 (14 jellied, 1 fresh) 27 (26 jellied, 1 fresh) 3	 <0.008 (2), 0.01-0.3 (13) <0.008 (2), 0.01-0.2 (25) <0.008 (2), 0.02	whole product		residue present as *p,p′*-DDE, *p,p′*-TDE and *p,p′*-DDT
Herring, smoked, unknown origin	 2	<0.006 (2)	whole product		
Mackerel, smoked	40	<0.006 (39), 0.02	whole product		residue present as *p,p′*-DDE

Residues of DDT in foodstuffs from GEMS/Food database (Table 15). Data on residues of DDT in various foods from the GEMS/Food database were available from WHO (Moy, 1996a). Most of the data (Table 15) are too summarized for the estimation of ERLs. Supplementary information on the number of samples, if available was supplied by Moy (1996b).

Table 15. DDT residues in foodstuffs from various countries (GEMS/Food; Moy, 1996a,b).

Commodity/Country	Year	Residues, mg/kg				Remarks and {no. of samples}
		Median	Mean	90th percentile	Range	
Cereals/Australia	1990-1992		0.0082		0.00002-0.0023	
Cereals/Canada	1989-1990	<0.01 <0.01		<0.01 <0.01	<0.01-0.02 <0.01-0.01	Breakfast cereal {50} Infant cereal {50}
Cereals/China (P.R.)	1992 1990	0.019	0.027 0.0012	0.095	<0.001-0.095 0.0009-0.0014	
Cereals/India	1990 (R) 1990 (R) 1990 (R) 1989		0.12 0.026 0.31 0.0035		max.0.52 max.0.085 max 1.3 0.0017-0.0054	Wheat Rice Maize
Cereals/Former Soviet Union	1991 (R) 1991 (R)		1.3 1.2			*o,p′*-DDT in wheat *p,p′*-DDT in wheat
Cereals/Spain	1990-1991 1990-1991		<0.001 <0.001		<0.001-0.003 <0.001-0.003	Bread, Basque country Cereals, Basque country
Cereals/Qatar	1989-1991		<0.01			Wheat, barley, maize, rice, flour {233}
Cereals/Vietnam	1990-1991		0.002		0.001-0.0033	Rice
Chicken/Brazil	1991 1990 1989	<0.001 <0.001 <0.001		0.07 0.04 0.07	<0.001-0.3 <0.001-0.22 <0.001-0.16	Sao Paulo {30} Sao Paulo {22} Sao Paulo {36}
Chicken/Canada	1984-1989 1986-1988 1986-1988		0.001 0.0014 0.0009		max. 0.003 max.0.0013	Chicken meat, domestic DDE, Avian, broiler DDE, Avian, turkey
Chicken/Denmark	1986-1991		0.003		0.002-0.003	Poultry meat, domestic
Chicken/Japan	1992-1993		0.001			
Chicken/Kenya	1988 (R)		0.68			
Chicken/Poland	1992		0.056 0.02			
Chicken/Spain	1995 (R)		0.00063		0.0004-0.00087	DDE, fresh poultry sausage
Eggs/Canada	1986-1988		0.008		max. 0.009	DDE, extractable fat, Ontario
Eggs/China (P.R.)	1992 1990	0.02	0.03 0.041	0.095	0.01-0.095 0.013-0.072	
Eggs/Cuba	1985-1988		0.61			Fat
Eggs/Denmark	1986-1991		0.02		0.02-0.03	
Eggs/Finland	1994 (R)		0.0016		0.0005-0.024	Imported (fat)
Eggs/Netherlands	1990-1992				0.11-0.20	Egg powder (fat)
Eggs/Spain	1990-1991		<0.001			Basque country
Fish/Arabian Gulf	1987 (R) 1987 (R)				0.002-0.011 0.005-0.045	NW Arabian Gulf Hor-al-Hammar
Fish/Australia	1990-1991		0.022		0.00014-0.23	
Fish/Canada	1984 1984		2 3			Cod, Admiralty Intel Cod, Barrow Strait
Fish/China (P.R.)	1992 1990	0.024	0.046 0.14	0.095	max. 0.31 0.0097-0.44	Fish uncooked Aquatic food
Molluscs/Croatia	1976-1990 1976-1990 1976-1990	0.0051 0.0051 0.006	0.0079 0.0084 0.0063			Bivalves Mussels Oysters
Fish/Cuba	1985-1988		0.075			
Fish/Egypt	1990 (R) 1990 (R) 1990 (R) 1990 (R) 1986-1988 1986-1988 1986-1988		0.0049 0.076 0.047 0.015 0.73 0.23 0.057			*o,p′*-DDE *p,p′*-DDT *o,p′*-DDT *p,p′*-DDT *o,p′*-DDT, Red Sea gov. *p,p′*-DDE, Ismailia gov. *p,p′*-DDT, Ismailia gov.

Commodity/Country	Year	Residues, mg/kg				Remarks and {no. of samples}
		Median	Mean	90th percentile	Range	
	1986-1988		0.01			*o*,*p*′-DDE, Suez gov.
Fish/Finland	1979				0.7-2.2	Herring muscle (fat)
	1986				0.3-1.0	Herring muscle (fat)
Molluscs/France	1989 (R)				<0.0004-0.59	dry wt., Mussels
	1989 (R)				0.001-0.73	dry wt., Oysters
Fish/India	1989-1993		0.015		0.00086-0.14	
	1989		0.015		0.00086-0.14	
Fish/Indonesia	1989-1993		0.028		0.00066-0.076	
Fish/Japan	1992-1993		<0.001			Tuna
	1992-1993		0.0087			Sea bream
	1992-1993		0.0091			Horse mackerel
	1992-1993		0.0089			Salmon
	1992-1993		0.041			Young yellowtail
	1992-1993		0.00016			Shrimp
	1992-1993		0.00016			Short-necked clam
	1992-1993		0.0024			Salted salmon
	1992-1993		0.0032			Semi-dried horse mackerel
	1992-1993		<0.001			Canned tuna
	1992-1993		0.0011			Seasoned tuna
	1992-1993		0.0036			Fish paste products
	1992-1993		<0.0001			Fish sausage
	1990	0.002		0.0048	<0.001-0.005	Fishes, flat, fresh {11}
	1990	0.002		0.009	<0.001-0.018	Horse mackerel, fresh {12}
Fish/Qatar	1989		<0.01		<0.01	Greasy grouper
	1989		<0.01		<0.01	Whitespotted-spinefoot
	1989		<0.01		<0.01	Gold toothless-trevally
	1989		<0.01		<0.01	Starry pigface bream
Fish/Spain	1990-1992		0.081		0.003-1	wet wt., Catalonia
	1990-1992		9.9		0.033-183	lipid wt., Catalonia
	1990-1991		0.002		<0.001-0.003	wet wt., Basque country
Fish/Thailand	1989-1993		0.0062		0.00048-0.019	wet wt.
Fish/ Molluscs/UK	1988		0.016			wet wt., Mackerel, liver
	1988		0.016			wet wt., Herring, liver
	1988		0.031			wet wt., Monkfish, liver
	1988		0.12			wet wt., Dogfish, liver
	1988		0.014			wet wt., Crab, hepato-pancreas
	1988		0.002			wet wt., Mussels, whole
Fish/ Molluscs/USA	1986-1989		0.42			*p*,*p*′-DDE, Carp
	1986-1989		0.08			*p*,*p*′-DDE, White sucker
	1986-1989		0.63			*p*,*p*′-DDE, Channel catfish
	1986-1989		0.06			*p*,*p*′-DDE, Largemouth bass
	1986-1989		0.03			*p*,*p*′-DDE, Smallmouth bass
	1986-1989		0.03			*p*,*p*′-DDE, Walleye
	1990 (R)		0.0054		<0.005-0.016	wet wt., Oysters
	1990 (R)		0.030		<0.005-0.18	wet wt., Crabs
Fish/Vietnam	1990-1991		0.026		0.0039-0.076	wet wt., Fish
	1990-1991		0.0017			wet wt., Prawn
	1990-1991		0.0072			wet wt., Shellfish
	1990-1991		0.078			wet wt., Crab
	1990-1991		0.090		0.050-0.12	wet wt., Caviar
Fruits/Australia	1990-1992		0.00013		0.00002-0.00024	wet wt.
Fruits/Canada	1992-1993	<0.01		<0.01	<0.01-0.01	Cherry
	1984-1989		0.02		0.01-0.03	Grapes, domestic
	1984-1989		0.03			Grapes, imported
	1984-1989		0.08		0.04-0.12	Pear, imported
Fruits/China (P.R.)	1992	0.002	0.035	0.095	<0.001-0.095	
	1990		0.003		0.0007-0.006	
Fruits/India	1990 (R)		0.003		max. 0.005	Guava
	1990 (R)		0.004		max. 0.007	Apple
	1990 (R)		0.004		max. 0.006	Grape

Commodity/Country	Year	Residues, mg/kg				Remarks and {no. of samples}
		Median	Mean	90th percentile	Range	
Fruits/Spain	1990-1991		<0.001			Basque country
	1990-1991		<0.0002			Sugars, Basque country
Fruits/USA	1991-1992				max. 0.01	Sugar beet
Meat/Australia	1990-1992		0.013		0.00016-0.083	wet wt., Meat and fat
Meat/Canada	1984-1989		0.14		0.10-0.17	Pig meat (fat), domestic
	1986-1988		0.030		max. 0.41	DDE, Beef (fat)
	1986-1988		0.008		0.008	DDE, Goat (fat)
	1986-1988		0.055		max. 0.15	DDE, Rabbit (fat)
	1986-1988		0.029		max. 0.20	DDE, Lamb, Mutton (fat)
	1986-1988		0.024		max. 0.08	DDE, Pork (fat)
	1985-1987				0.00025-0.0038	highest in Pork
Meat/China (P.R.)	1992	0.095	0.16	0.48	<0.001-0.6	DDT, Meat, uncooked
	1990		1.2		0.12-4.1	Meats, fat
Meat/Cuba	1985-1988		0.43			Pork fat, Havana
Meat/Denmark	1986-1991		0.04		0.02-0.09	Cattle meat (fat), domestic
	1986-1991		0.04		0.02-0.10	Pig meat (fat), domestic
Meat/Egypt	1993 (R)				1-5	Beef carcases, Cairo
	1993 (R)				1-7	Beef carcase muscle, Cairo
	1993 (R)				4	Beef carcase fat, Cairo
	1993 (R)					Buffalo carcases, Cairo
	1993 (R)				0	Buffalo carcase muscle, Cairo
						Buffalo carcase fat, Cairo
	1993 (R)				4	Buffalo carcase liver, Cairo
	1993 (R)				9.6	Mutton carcases, Cairo
	1993 (R)					Mutton carcases muscle, Cairo
	1993 (R)				4	Mutton carcase fat, Cairo
	1993 (R)				1-7	Mutton carcase liver, Cairo
	1993 (R)				5	
Meat/India	1989		0.5		0.00065-4.1	wet wt., (fat)
Meat/Japan	1992-1993		0.016			Beef fat
	1992-1993		0.0086			Pork fat
	1992-1993		0.075			Sausage fat
Meat/Mexico	1995 (R)		0.14			Bovine kidney fat, Vera Cruz
Meat/Netherlands	1990-1992				0.11-0.50	Calf (fat), domestic
	1990-1992				0.11-0.50	Pig (fat), domestic
	1990-1992				0.01-0.05	Sheep (fat), domestic
	1990-1992				0.01-0.05	Goat (fat), domestic
	1990-1992				0.01-0.10	Horse (fat), domestic
Meat/New Zealand	1990-1991	<0.03			<0.03	Beef (fresh) {48}
	1990-1991	<0.03			<0.03	Meat products {48}
						p,*p*′- DDT, *o*,*p*¢DDT
Meat/Poland	1990-1993		0.87			*p*,*p*′-DDE, Wild boar
	1990-1993		0.73			DDT, Wild boar
	1990-1993		0.26			*p*,*p*′-DDE, Roe-deer
	1990-1993		0.28			DDT, Roe-deer
	1990-1993		0.12			*p*,*p*′-DDE, Stag
	1990-1993		0.14			DDT, Stag
	1990-1993		0.12			*p*,*p*′-DDE, Elk
	1990-1993		0.14			DDT, Elk
	1991 (R)				0.00034-0.0018	Muscle, Horse
	1991 (R)				0.022-0.12	Fat, Horse
	1987-1988				0.045-0.084	Fat, Ruminants
	1987-1988				0.079-0.14	Fat, Rabbits, Swine, Turkeys, Geese
	1987-1988				0.40-0.44	Fat, Duck, Wild boar
	1986-1989		0.18			Fat, Wild boar
	1986-1989		0.063			Fat, Roe-deer
	1986-1989		0.048			Fat, Stag
	1986-1989		0.049			Fat, Elk
	1986		0.10			Fat, Pig
	1986		0.032			Liver, Pig

Commodity/Country	Year	Residues, mg/kg				Remarks and {no. of samples}
		Median	Mean	90th percentile	Range	
	1986		0.0065			Meat, Pig
	1984-1985				0.18-0.32	Fat, Wild boar, Roe-deer, Red deer
	1984-1985				0.006-0.009	Brain, Wild boar, Roe-deer, Red deer
	1980-1983		0.23			Fat,Wild boar
	1980-1983		0.057			Fat, Roe-deer
	1980-1983		0.045			Fat, Stag
	1980-1983		0.0071			Fat, Elk
Meat/Former Soviet Union	1991 (R)		2.0			*o*,*p*¢DDT, Pork
	1991 (R)		2.0			*p*,*p*′-DDT, Pork
Meat/Spain	1994 (R)		25		max. 91	Lamb
	1990-1991		<0.003			Meat, Basque country
	1990-1991		<0.005		<0.005-0.007(2)	Meat products, Basque country
	1995 (R)					DDE, Pork, Cured sausage
	1995 (R)		0.0063		0.004-0.016	(fat)
	1995 (R)					DDE, Pork, Cured ham (fat)
	1995 (R)		0.007		0.004-0.015	DDE, Pork, Bologna (fat)
			0.016		0.009-0.03	DDE, Fresh sausage with beef
	1995 (R)		0.0066		0.004-0.013	and pork (fat)
						DDE, Fresh beef sausage (fat)
			0.0077		0.004-0.013	
Meat/USA	1991-1992				max. 0.50	Cattle (fat), imported
	1992				max. 0.19	Pig (fat), imported
	1992				max. 0.44	Sheep (fat), imported
	1991				max. 1.1	Sheep, lamb, goat (fat), imported
	1991				max. 0.27	Pig (fat), imported
Meat/Vietnam	1990-1991		0.0013		0.061-0.18	Wet wt., Fat
	1990-1991		0.048		0.01-0.086	Wet wt., Meat
Edible Oils/Australia	1990-1992		0.0021		0.00036-0.0053	Wet wt.
Edible Oils/China (P.R.)	1992	0.022	0.035	0.095	<0.007-0.095	
Edible Oils/India	1990 (R)		0.59		max. 0.73	Vegetable
	1990 (R)		2.4		max. 7.5	Mustard
	1990 (R)		1.5		max. 2.2	Groundnut
	1990 (R)		0.21		max. 0.65	Sesame
	1989		0.021		0.0018-0.057	Wet wt.
Edible Oils/Spain	1990-1991		<0.01			Fats and oils, Basque country
Edible Oils/Vietnam	1990-1991		0.067			Wet wt.
Pulses/Australia	1990-1992		0.0024		0.00002-0.0086	Wet wt.
Pulses/India	1990 (R)		0.022		max. 0.051	Arhar
	1990 (R)		0.016		max. 0.057	Moong
	1990 (R)		0.023		max. 0.086	Gram
	1990 (R)		0.063		max. 0.16	Lentil
	1990 (R)		0.039		max. 0.12	Black Gram
	1989		0.02		0.0011-0.04	Wet wt.
Pulses, Nuts/Spain	1990-1991		<0.001			Basque country
Pulses/Vietnam	1990-1991		0.0019		0.00034-0.003	Wet wt.
Vegetables/Australia	1990-1992		0.0033		0.00007-0.0089	Wet wt.
Vegetables/Canada	1984-1989		0.04		0.01-0.11	Carrots, domestic
	1984-1989		0.43		0.39-0.48	Carrots, imported
	1984-1989		0.05			Potatoes, domestic
	1984-1989		0.04		0.01-0.07	Potatoes, imported
	1984-1989		0.02			Cucumbers, imported
Vegetables/China (P.R.)	1992	0.019	0.024	0.095	<0.001-0.095	Fresh vegetable
	1990		0.0021		0.0015-0.0028	Legumes and nuts
	1990		0.0016		0.0004-0.0033	Potatoes
	1990		0.0009		0.0032-0.008	Vegetables
	1994 (R)		0.38			Vegetables (fatty food)
	1992 (R)				0.0012-0.11	Vegetables

Commodity/Country	Year	Residues, mg/kg				Remarks and {no. of samples}
		Median	Mean	90th percentile	Range	
Vegetables/Egypt	1990 (R)		0.002		max. 0.005	Spinach
Vegetables/Spain	1990-1991		<0.001			Basque country
Vegetables/USA	1991-1992				max. 0.17	Carrots, domestic
	1991-1992				max. 0.13	Carrots, imported
	1991-1992				max. 0.03	Tomatoes, imported
Dairy/Argentina	1994		0.00096			Butter, Santa Fe, Rosario
Dairy/Australia	1990-1992		0.0059		0.0016-0.018	Wet wt., Dairy products
Dairy/Brazil	1989-1991	0.020	0.025	0.05	<0.01-0.14	Cow whole Milk, Sao Paulo {184}
Dairy/Bulgaria	1993		<1.0		max. 1.8	Ewe Milk-cheese (one sample)
Dairy/Canada	1986		0.00064			Cow milk (fat basis)
	1984-1989				0.02-0.1	Butter, domestic
	1984-1989		0.03		0.01-0.06	Cheese, domestic
	1984-1989		0.04		0.01-0.11	Cheese, imported
Dairy/China (P.R.)	1992	0.025	0.033	0.095	<0.008-0.095	Whole fluid Milk (uncooked)
	1990		0.0028		0.0005-0.0063	Milk
Dairy/Cuba	1985-1988		0.0049			Butter, Havana
	1985-1988		0.024			Whole product, Milk, Havana
	1985-1988		0.030			Whole product, Cheese, Havana
Dairy/Denmark	1986-1991		0.02		0.02-0.03	Butter, domestic
	1986-1991		0.03		0.02-0.05	Cheese, domestic
	1986-1991		0.04		0.02-0.04	Cheese, imported
	1986-1991		0.03		0.03-0.03	Butter, imported
Dairy/Finland	1994 (R)		ND			Wet wt., Milk
	1994 (R)		0.00068		0.0002-0.0007	*p,p′*-TDE, domestic Cheese
	1994 (R)		0.00054		0.0002-0.0012	*p,p′*-DDE, imported Cheese
	1994 (R)		0.00014		0.0002-0.00076	*p,p′*-TDE, imported Cheese
Dairy/Greece	1992-1993		0.10			Northern Greece
Dairy/India	1994 (R)		0.63			Milk (fat)
	1993 (R)				0.042-0.38	Milk, Delhi
	1993 (R)		0.22			Buffalo Milk
	1993 (R)		0.19			Milk, condensed
	1993 (R)		0.34			Cheese
	1993 (R)		0.38			Cream
	1993 (R)		0.19			Curd
	1993		0.006			Cow milk
	1992 (R)				0.040	Bovine milk, Haryana
	1992 (R)				0.74	Baby milk, Haryana
	1990 (R)		0.058		max. 0.22	Milk
	1990 (R)		4.8		max. 9.8	Butter
	1990 (R)		3.8		max. 6.0	Deshi ghee
	1989		0.0014		0.17-5.2	Wet wt.
	1989		1.4		0.78-3.0	Wet wt.
	1988 (R)		0.78			Cheese, Punjab
Dairy/Israel	1986		0.0032			Cow milk
Dairy/Japan	1994	<0.02	<0.02	0.057	<0.02-0.076	Milk, cow's, pasteurized whole
	1994	<0.025	<0.025	<0.025	<0.025	fluid
	1992-1993		0.0026			Milk, cow's, raw whole fluid
	1992-1993		0.0023			Ice cream
	1992-1993		0.00021			Processed Cheese
	1993	<0.02	<0.02	0.029	<0.02-0.059	Cow milk
	1993	<0.02	<0.02	0.028	<0.02-0.081	Milk, cow's, raw whole fluid
	1992	0.013	0.014	0.023	<0.01-0.074	Milk, cow's, pasteurized whole
	1992	0.012	0.015	0.026	<0.01-0.056	fluid
	1991	0.013	0.022	0.054	<0.01-0.17	Milk, cow's, pasteurized whole fluid
	1991	0.019	0.032	0.058	<0.01-0.11	Milk, cow's, raw whole fluid
	1990	0.013		0.017	<0.01-0.053	Milk, cow's, pasteurized whole fluid

Commodity/Country	Year	Residues, mg/kg				Remarks and {no. of samples}
		Median	Mean	90th percentile	Range	
						{90} Milk, cow's, raw whole fluid {11} Milk, cow's, raw whole fluid {72}
Dairy/Mexico	1993		0.0023		0.00016-0.025	Cow milk
Dairy/Netherlands	1990-1992				0.0044-0.02	Dairy cow products, domestic
Dairy/Poland	1994		0.0026		0.00044-0.015	Milk
Dairy/Qatar	1990		<0.01		<0.01	Whole dried cow milk
Dairy/Slovakia	1987-1988				0.041	Milk products
Dairy/Spain	1994 (R) 1990-1991 1990-1991 1990		0.056 <0.001 <0.002 0.00084			Milk, sterilized Milk, Basque country Dairy products, Basque country Cow milk
Dairy/USA	1990-1991 1991-1992				max. 0.019 max. 0.0076	*p,p′*-DDE,whole product basis Milk, domestic
Dairy/Vietnam	1990-1991		0.0072		0.007-0.0073	Wet wt., Butter

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Dietary intake of DDT (Table 16). Data on adult dietary intakes of DDT were prepared by the GEMS/Food programme (WHO, 1996). The data are from studies of regular diets.

Table 16. Dietary intake of DDT by adults (WHO, 1996). ADI: 20 ì g/kg bw.

Country	Year	Daily intake, ì g/kg bw	% of ADI
Australia	1980 1987	0.39 0.026	1.95 0.13
Egypt	1988	13.7	68.5
Finland	1984 1986	0.041 0.026	0.21 0.13
Guatemala	1982 1984 1985 1988	0.26 0.2 0.065 0.031	1.3 1.0 0.33 0.16
India	1981 1983	3.9 3.6	19.5 18
Japan	1980 1982 1984 1986 1988	0.056 0.07 0.03 0.02 0.02	0.28 0.37 0.15 0.1 0.1
Netherlands	1984 1985	0.004 0.004	0.02 0.02
New Zealand	1982	0.003	0.015
Switzerland	1983	0.03	0.15
Thailand	1980 1987	1.6 0.0008	8 0.004
UK	1980 1981 1985	0.05 0.035 0.05	0.25 0.18 0.25
USA	1980 1982 1985 1986	0.36 0.033 0.036 0.019	1.8 0.17 0.18 0.1

Country	Year	Daily intake, ì g/kg bw	% of ADI
	1988	0.025	0.13

Dietary intake of DDT from human breast milk by infants (Table 17). Levels of DDT in breast milk can provide an assessment of the integrated exposure of women to DDT, which is largely due to food contamination. Breast milk is also the sole source for most infants and, consequently, levels of DDT are an important safety concern. Data provided by the GEMS/Food programme (WHO, 1996) from 39 countries show that DDT is present in virtually every sample of breast milk tested.

Table 17. Estimated dietary intakes of DDT for infants from breast milk (WHO, 1996).

Country	Year	Intake, ì g/kg bw	
		Median	Mean
Australia	1974	16.4	
	1978	9.1	
	1980	5.5	
	1981		5.0
	1982	5.0	
	1990	1.6	
	1991	3.6	
Belgium	1976		12.7
	1982		5.4
Brazil	1987 (Sao Paolo)		4.2
	1987 (Rural)		2.0
Canada	1967		17.4
	1970		9.2
	1975		5.3
	1981		4.7
	1982		4.5
	1982		4.5
	1986	1.0	1.4
China	1982		30.5
Colombia	1987(R)		9.0
Croatia	1981-1982		9.9
Slovakia	1971-1974		33.2
	1989-1992	6.4	8.4
Denmark	1982	4.3	
	1982	4.9	
Ethiopia	1994(R)		35.3
Egypt	1985-1986		6.9
Finland	1982		3.7
France	1974		13.6
	1986		9.9
Germany	1973-1974		11.6
(FRG/West)	1984		6.3
	1984-1987		3.7
	1988		3.1
	1989		3.2
	1990		2.6
	1991		2.4
	1992-1993		1.6
Germany	1979		12.3
(GDR/East)	1980-1981		23.4
	1984-1988		5.8
	1989-1990		12.4
	1990		11.7
	1990-1991		6.2
	1990-1991		10.8

Country	Year	Intake, ì g/kg bw	
		Median	Mean
Greece	1974-1975		41.3
	1983		0.2
Guatemala	1983	41.4	
Hong Kong	1985	40.0	47.2
Hungary	1975-1976		16.15
India	1980	45.6	
	1982	36.7	
	1983		29.8
	1986	62.0	
	1990(R)	17.3	
Iran	1992-1993	1.2	0.7
Iraq	1984(Baghdad)		19.2
Italy	1978		18.4
	1983-1984(Rome)		7.2
	1982-1985		6.6
	1985		6.1
Japan	1980	4.4	
	1981	4.7	
	1982	4.7	
	1983	3.5	
	1984	3.5	
	1985	2.9	
	1989	3.3	
	1991	2.9	3.6
	1992	2.4	1.9
Kenya	1983-1985		7.7-85.3
The Netherlands	1979		10.9
Nigeria	1987		17.4
Norway	1981-1982		4.1
	1986		2.6
	1988		3.7
Poland	1979		45.4
	1987(R)		25.0
Rwanda	1983		18.9
South Africa	1987		68.5
Spain	1979-1982(Madrid)		2.7
Sweden	1981		4.6
	1983	5.6	
	1983		5.6
	1986(Sundsvall)	2.5	2.6
	1986(Göteborg)	2.5	2.5
	1986(Uppsala)	2.5	3.0
	1987(Borlange)	2.0	2.4
Tunisia	1982		17.4
Turkey	1984-1985(Ankara)		16.7
	1984-1985(Adana)		48.1
	1984-1985(Kocaeli)		15.0
Uganda	1994(R)		14.7
USA	1979		8.6
	1983		9.5
	1984-1987(Hawaii)		2.5
UK	1980	3.7	
	1979-1980		8.6
	1982		7.8
	1988	0.025	0.1
Yugoslavia	1984-1985(Serbia)		7.6
Zimbabwe	1989(Harare)		26.9

R: report

NATIONAL MAXIMUM RESIDUE LIMITS

The Meeting was informed of the following national MRLs (next page) for DDT in animal products.

Definition of the residue: sum of *p,p′*-DDT, *o,p′*-DDT, *p,p′*-DDE and *p,p′*-TDE (*p,p′*-DDD).

Country	Commodity	MRL, mg/kg
Australia	Meat (fat)	5
European Union	Meat[1,2]	1
	Milk[3]	0.04
	Eggs[4]	0.1
Norway	Young bovine animal (fat)	1
	Pig (fat)	1
	Sheep (fat)	1
	Lamb (fat)	1
	Hen (fat)	1
	Reindeer (fat)	1
	Moose (fat)	1
Poland	Meat and meat products (fat)	1
	Milk and milk products	0.04
	Eggs	0.1

[1] In fat

[2] The maximum level of the residues in meat and meat products are expressed on the basis of fat. In the case of foodstuffs with a fat content of 10% or less by weight, the residue is related to the total weight of the boned foodstuff. In such cases, the maximum level is one-tenth of the value related to the fat content, but must be no less than 0.01 mg/kg.

[3] In determining the residues in raw cow's milk and whole cream cow's milk, a fat content of 4% by weight should be taken as a basis. For raw milk and whole cream milk of other animal origin the residues are expressed on the basis of the fat.

For other foodstuffs

- with a fat content of less than 2% by weight, the maximum level is taken as half that set for raw milk and whole cream milk,
- with a fat content of 2% or more by weight, the maximum level is expressed in mg/kg of fat. In such cases, the maximum level is 25 times that set for raw milk and whole cream milk

[4] For eggs and egg products with a fat content higher than 10% the maximum level is expressed in mg/kg fat. In such cases the maximum level is 10 times the maximum level for fresh eggs

APPRAISAL

DDT was first evaluated in 1966 and has been reviewed several times since. The JMPR in 1993 and 1994 proposed Extraneous Residue Limits (ERLs) for carrots, eggs, meat and milks and confirmed the previous temporary ERL proposed for cereal grains. The 1995 CCPR was informed that additional data on meat were available from Australia, New Zealand and the USA and decided to keep the proposal for meat (1 mg/kg) at Step 3 pending the evaluation of these data by the 1996 JMPR. The 28th (1996) Session of the CCPR advanced all ERLs except that for meat to Step 8. The existing CXL for meat, 5 mg/kg (fat), was converted to a temporary limit in 1993.

No information was supplied by governments on registered or recommended uses of DDT on crops or animals. Some countries allow its limited use for public health applications.

The Meeting received national residue survey data on DDT in meat from Australia, Germany,

New Zealand, Norway, Thailand, the UK and the USA.

The British annual report on residue monitoring for 1994, further data on dairy products from New Zealand and on commodities of plant origin from Norway and Spain were also provided. Information on residues and dietary intakes of DDT was made available by Global Environment Monitoring System - Food Contamination Monitoring and Assessment Programme (GEMS/Food) of WHO. Because no request was made for these data by the CCPR however, the Meeting could not conclude that a complete database was available to support re-evaluations of ERLs for commodities other than meat and proposed to postpone such evaluations to a later periodic review.

Definition of the residue: Sum of *p,p′*-DDT, *o,p′*-DDT, *p,p′*-DDE and *p,p′*-TDE (*p,p′*-DDD).

The residue is fat-soluble.

Meat. Residues of DDT and its metabolites in most of the many samples analysed were at very low levels, but in some samples of meat fat were higher than the proposed ERL of 1 mg/kg.
From Australia, results of 91,747 analyses of meat fat were available from 1989-1994, and 51 samples (0.06% of the total) of which 29 were beef, 1 deer, 1 horse, 13 sheep and 7 pork, contained residues above 1 mg/kg. In 3 samples the residues were higher than 5 mg/kg.

In Germany, 1653 samples of fat of meat were analysed in 1993. Only 1 sample of sheep fat and 2 samples of bacon (0.2% of the total number) contained residues in the ranges 1.1-2 and 2.1-5 mg/kg respectively.

Analyses of 1568 and 28,640 meat fat samples were available from Norway (1990-1994) and Thailand (1993-1994) respectively. All residues were lower than 1 mg/kg.

In the USA, 31 (0.08%) of 38,420 meat fat samples from 1991-1994 contained residues higher than 1 mg/kg (4 samples with residues above 5 mg/kg).

In UK monitoring in 1994 74 samples of lamb fat were analysed and none contained residues exceeding 1 mg/kg.

In a monitoring programme in New Zealand, from July 1990 to June 1994 analysis of a total of 4682 samples of meat fat from lambs, sheep, calves, cattle, pigs, deer and goats showed residues above 1 mg/kg in 1.6%, above 2 mg/kg in 0.43%, and above 5 mg/kg in 0.04% of the samples. In a separate survey of 403 lambs from a region with a history of DDT use 33%, 18% and 3% of the fat samples contained residues above 1,2 and 5 mg/kg respectively.

The following Table shows the distribution of DDT residues higher than the proposed ERL of 1 mg/kg.

Animal	No. of samples	Distribution of DDT residues in %		
		>1 mg/kg	>2 mg/kg	>5 mg/kg
Lambs	965	1.9	0.2	-
Adult sheep	548	2.2	0.7	-
Adult bovines	739	0.68	-	-
Suckling calves	1211	2.5	0.82	0.08
Pigs	925	1.1	0.43	0.11
Deer	227	0.88	-	
Goats	67	-	-	
Lambs from a region where DDT	403	33	17.6	3.2

Animal	No. of samples	Distribution of DDT residues in %		
was historically used				

In all, 162,102 samples of meat fat were analysed in Australia, Germany, Norway, Thailand, the UK and the USA, and residues above 1 mg/kg were found in 85 samples (0.05%). The samples from New Zealand belonged to a different population. Excluding the lambs from the region with a known DDT history, 1.6% of the 4682 samples analysed were higher than the proposed ERL of 1 mg/kg; 0.43% higher than 2 mg/kg and 0.04% higher than 5 mg/kg.

The GEMS/Food database for meat fat shows low levels of DDT residues in most countries, but maximum residues were found of 2 mg/kg in the former Soviet Union (1991), 4.1 mg/kg in China (1990), 7 mg/kg in Egypt (1993), 4.1 mg/kg in India (1989) and 91 mg/kg in Spain (1994). These figures are insufficient to support a revision of the proposed ERL, because some relevant information (e.g. number of samples analysed, explanation of extreme values) is not given.

RECOMMENDATIONS

On the basis of the residue data received from the government of New Zealand, the Meeting concluded that the ERL of 1 mg/kg for DDT in meat (fat) recommended by the 1993 JMPR should be increased to 5 mg/kg, thus confirming the existing temporary CXL.

Definition of the residue for compliance with MRLs and for estimation of dietary intake:

Sum of *p,p′*-DDT, *o,p′*-DDT, *p,p′*-DDE and *p,p′*-TDE (*p,p′*-DDD).

The residue is fat soluble.

Commodity		Recommended ERL, mg/kg	
CCN	Name	New	Previous
MM 0095	Meat	5 (fat)	1 (fat)

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DIAZINON (022)

EXPLANATION

Diazinon was first evaluated by the 1965 JMPR and has been reviewed several times since. In 1993 a periodic review was conducted and in 1994 a new MRL was recommended for hops. The 1993 JMPR recommended, among other items, an increase in the CXL for pome fruits from 0.5 to 2 mg/kg and the withdrawal of the CXLs for animal commodities in the absence of animal transfer studies and data from uses as an ectoparasiticide.

The CCPR in 1995 and 1996 endorsed most of the recommendations of the 1993 JMPR with the exception of the proposed MRL for pome fruits and the recommended withdrawal of the CXLs for milks and the meat of cattle, pigs and sheep. The CXLs for animal commodities were retained pending review by the 1996 JMPR of data on new animal feeding trials to be submitted by Australia and the manufacturer. The 1993 proposal for pome fruit was held at step 7C by the 1996 CCPR, mainly owing to concern at the potentially high dietary intake from this source.

The Meeting received (1) data on residues and information on GAP for uses of diazinon as an ectoparasiticide, together with animal transfer studies, from the manufacturer (2) summarized data on residues in pome fruit, plums and carrots from Germany (3) monitoring data and information on GAP and national MRLs from Poland (4) information on GAP for mushrooms in the UK and (5) an Australian submission on residues of diazinon in cattle resulting from ectoparasite control. Summary data on residues and information on GAP for the use of diazinon on rice in Thailand were also received. Summary data were not considered to be an adequate basis for estimating maximum residue levels.

Formulations

EC and WP formulations for crop protection were mentioned in the 1993 Evaluations. EC and WP formulations are also available for the treatment of animals for ectoparasites. The most important, and used in the ectoparasite control trials, were a 250 EC with the formulation code A-7182 (250 g ai/l, trade name Neocidol, a 600 EC with the formulation code A-3695 J (600 g ai/l, trade name Neocidol) and to a lesser extent a 60 EC with the formulation code A-139F (60% w/w, trade name Top Clip Gold Shield). The compositions of these formulations were available to the Meeting. When these three were applied as a spray to sheep at the same nominal (recommended) rate of 600 ppm no differences were found in the resulting residues in blood or fat (Morrison, 1994).

Other code numbers used for diazinon include G 24′480, CGA 31′331, OMS 469 and GNT 19507. Other trade names of formulations for animal health uses include Dimpygal, Nucidol, Sarnicida-Garrapaticida, Vetsarol, Clik, Spike, Protector and Kacador, and for other uses Banosan, Antigal, Galton, Gal-Wash, Galesan, Paragal and Eureka.

METABOLISM AND ENVIRONMENTAL FATE

The fate of diazinon in animals, plants and soil was described in the 1993 JMPR periodic review or in earlier JMPR evaluations, and only those studies not previously reviewed by the FAO Panel or details of which are needed to facilitate the review and understanding of the studies on animal transfer and ectoparasite control will be described in detail.

Animal metabolism

The fate of diazinon in rats, mice, guinea pigs, dogs, goats, sheep, cows and plants was described in the 1993 periodic review and a diagram of the proposed metabolic pathways was presented. Some of these studies were provided to the WHO Expert Group but not to the FAO Panel. For example, the 1993 Expert Group reviewed several animal disposition or metabolism studies which apparently were not provided to either the 1993 or the present FAO Panel. These included a cow metabolism study (Robins *et al*., 1957) the disposition of residues in goats (Simoneaux, 1988a) and chickens Simoneaux, 1988b), the identification of metabolites in hens and goats (Simoneaux *et al*., 1988), a supplementary report on metabolism in hens (Simoneaux *et al*., 1989), and the characterization of diazinon metabolites in chickens (Simoneaux, 1988c). Some studies reviewed by the 1993 JMPR were re-submitted to the present Meeting.

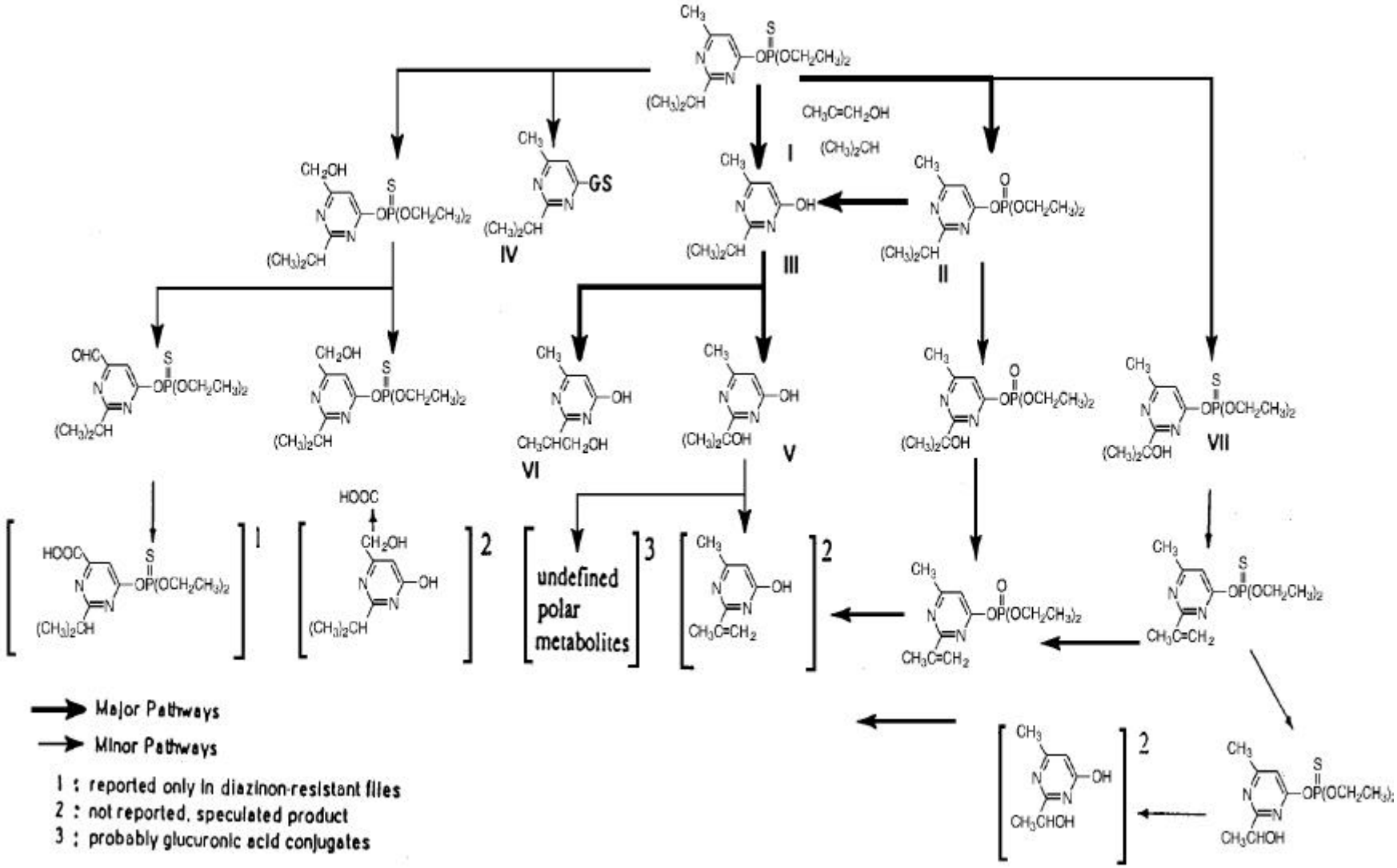
In mammals

In general terms, diazinon was reported in the 1993 Evaluations (Parts I and II) to be almost completely absorbed from the intestinal tract and easily absorbed dermally. Elimination was reported to be rapid in the urine and faeces, mainly the urine. In mammals metabolism was reported to progress primarily via hydrolysis of the ester linkage, yielding 4-hydroxy-2-isopropyl-6-methylpyrimidine (metabolite B1 or G-27550) followed by oxidation of the isopropyl group to give primary and tertiary alcohols, of which the latter may become conjugated. Another primary route is oxidation to diazoxon which may be hydrolysed to B1 or oxidized at the isopropyl group before hydrolysis. Other less important routes include oxidation of the methyl group. Ring cleavage was not reported in rats.

Unchanged diazinon was not a major residue in tissues although low residues of diazinon and diazoxon were reported, especially in fat. Because of the importance of these metabolic routes to the focus of this evaluation on residues in animal products, the proposed metabolic route in mammals presented in the 1993 Evaluations is repeated, slightly modified and expanded, as Figure 1.

In an early study on sheep dosed by stomach tube at 1 g/kg, three cholinesterase-inhibiting metabolites were identified in the urine and fat (Janes *et al.*, 1973). These were hydroxydiazinon (VII in Figure 1), its isomer formed by hydroxylation of the ring methyl group, and dehydro-diazinon, shown in Figure 1 as formed by dehydration of VII. This study was concerned with cholinesterase-inhibiting metabolites, but later studies on metabolism in mammals produced for food have focused on the major routes of metabolism irrespective of cholinesterase inhibition.

Figure 1. Proposed metabolic pathways of diazinon in mammals.



A more recent study (Simoneaux, 1988) was reviewed by the 1993 JMPR (Evaluations Part II - Toxicology). A more detailed description follows. Tissues and milk were analysed after dosing goats by capsule for four consecutive days with [^{14}C]diazinon at a rate equivalent to 100 ppm in the diet. TLC analysis showed that 94% of the radioactivity was extracted from milk. At least 94% of the total ^{14}C was generally extractable from the tissues, but only 79% from liver. Over 80% of the extractable ^{14}C was in the organic phase except that from liver (58%) and kidney (68%). Table 1 indicates the identity and distribution of the residues.

Table 1. Distribution of ^{14}C in tissues and milk of goats dosed for 4 consecutive days with [^{14}C]diazinon at a rate equivalent to 100 ppm in the diet (Simoneaux, 1988).

Sample & total ^{14}C expressed as diazinon	% of organoextractable ^{14}C present as				
	diazinon	diazoxon	hydroxydiazinon	hydroxypyrimidine	Metab. 31144*
Liver 1.6 mg/kg	0.2	0.3	0.2	19.2	19
Kidney 3.0 mg/kg	<0.1	0.3	<0.1	19.8	30.6
Omental fat 0.4 mg/kg	67.8	4.1	12.8	9.3	6.8
Perirenal fat 0.4 mg/kg	64	0.8	12.3	4.3	4.2
Tenderloin 0.4 mg/kg	6.2	1.0	1.4	26	39.4
Leg muscle 0.5 mg/kg	1.6	<0.1	0.4	35.3	40.4
Milk (day 4) 0.7 mg/kg	0.2	0.2	0.1	39.3	37.3

* Metabolite GS-31144 = hydroxy derivative of hydroxypyrimidine
(-OH on tertiary isopropyl carbon, see Figure 1).

The 1993 JMPR also reviewed a study in which residues in tissues were characterized or identified after the dermal treatment of sheep with [^{14}C]diazinon (Capps and Sumner, 1990). The sheep were treated daily for three days with an acetone solution of [^{14}C]diazinon at 40 mg/kg bw approximating the maximum drench treatment, applied to a shaved area of *c*.10% of the back. The sheep were slaughtered 6 hours after the last treatment and tissues were analysed by TLC and HPLC. Over 90% of the ^{14}C was extracted from all the tissues. The distribution of the residues as determined by HPLC is shown in Table 2. Results were similar by TLC for all samples except muscle, which was not analysed by TLC.

Table 2. Distribution of ^{14}C residues in sheep tissues after dermal treatment for three days with 40 mg/kg bw [^{14}C]diazinon as determined by HPLC (Capps and Sumner, 1990)

Sample & total ^{14}C expressed as diazinon[1]	% of organoextractable ^{14}C present as[2]				
	diazinon	Conj. of 3114 and hydroxypyrimidine[3]	Unknown polar compounds	hydroxy-pyrimidine	Metab. 3114*
Liver 4.4 mg/kg	3.7	13.8	10.9	41.4	18
Kidney 9.4 mg/kg	6.2	8.6	28	24.5	22.6
Back fat 7.3 mg/kg	85.2	--	--	1.6	--
Heart 4.4 mg/kg	55.9	--	--	16.4	12
Leg muscle 4.0 mg/kg	59.2	--	--	23.2	13

* Metabolite GS-31144 = hydroxy derivative of hydroxypyrimidine (-OH on isopropyl tertiary carbon, see Figure 1).
[1] Average of sheep 1 & 2
[2] Fat, heart and leg muscle from sheep 1. Kidney and liver average of sheep 1 & 2
[3] Identified as HPLC "region B", mainly conjugates of GS-3114 and hydroxypyrimidine.

In poultry

Poultry feeding (transfer) studies were provided to the Meeting, but poultry metabolism had not been reviewed by the FAO Panel in the 1993 periodic review. Studies of poultry metabolism were therefore provided on request to the present Meeting (Simoneaux, 1988c, 1989; Simoneaux *et al*., 1988, 1989).

Simoneaux (1988c) dosed 4 Leghorn hens with [^{14}C]diazinon by capsule for 7 consecutive days at a rate equivalent to 25 ppm in the diet. Residues were characterized in the excreta, eggs and tissues. More than 78% of the dose was excreted. Simoneaux *et al*. (1988) identified metabolites in goat urine in order to correlate the findings with residues found in the tissues of goats and hens. The identification of G-27550 and GS-31144 by GC-MS and LC-MS after the acid or enzymatic hydrolysis of aqueous fractions gave evidence of their conjugation.

Simoneaux (1989) and Simoneaux *et al*. (1989) provided further clarification of hen metabolism, and demonstrated improved extraction of residues and further identification of metabolites after the treatment of samples with protease. The results of analyses of eggs (day 7) and hen tissues by Simoneaux *et al*. (1989) are summarized in Tables 3-5.

diazinon

Table 3. Distribution and extractability of ^{14}C in eggs and tissues before and after treatment with protease (Simoneaux *et al*., 1989).

Sample	^{14}C		Extractable ^{14}C[1] as % of total		% of extractable ^{14}C in	
	mg/kg as diazinon	% of dose	Before protease	After protease	Organic phase[2]	Aqueous phase
Egg yolk	0.07	<0.01	67	88	88	12
Egg white	0.07	0.01	98		87	13
Liver	0.11	0.02	63	82	49	51
Kidney	0.15	0.01	76	98	48	52
Lean meat	0.03	0.05	64	94	49	51
Skin fat	0.02	0.01	44	100	61	39
Peritoneal fat	0.01	0.01	31	100	62	38

[1]Extracted with 9:1 methanol/water
[2]Methanol/water extract was concentrated and partitioned with hexane

Table 4. Characterization of organo-extractable ^{14}C in 7-day egg yolks and whites by TLC (Simoneaux *et al*., 1989).

Residue	Egg yolks		Egg whites	
	% of ^{14}C in yolk	mg/kg as diazinon	% of ^{14}C in white	mg/kg as diazinon
Diazinon	0.02	<0.001	0.03	<0.001
Hydroxydiazinon (CGA-14128)	0.06	<0.001	0.05	<0.001
Diazoxon (G-24576)	0.42	<0.001	1.3	<0.001
Pyrimidinol metabolite (G-27550)	11.1	0.007	9.4	0.006
Unknown[1]	2.9	0.002	--	--
Hydroxy derivative of G-27550 (GS-31144)[2]	18.6	0.012	33.3	0.022
Metabolite M3[3] + glucuronide & other conjugates	25	0.016	41.3	0.027

[1] Unresolved GS-31144 and G-27550 suspected
[2] 4-hydroxy-2-(1-hydroxy-1-methylethyl)-6-methylpyrimidine (Figure 1, structure V).
[3] 4-hydroxy-2-(2-hydroxy-1-methylethyl)-6-methylpyrimidine (Figure 1, structure VI).

Similar residues were identified by TLC in organic extracts of poultry tissues (Table 5).

Table 5. Distribution of residues in poultry tissues determined by TLC (Simoneaux *et al.*, 1989).

Residue	% of ^{14}C in sample/residue, mg/kg as diazinon				
	Liver	Kidney	Skin	Lean meat	Peritoneal fat
31144	3.5/0.004	3.7/0.006	4.2/0.001	6.5/0.002	3.1/0.001
27550	0.6/<0.001	2.3/0.003	2.6/<0.001	2/<0.001	0.7/<0.001
diazoxon	0.9/0.001	0.18/<0.001	1.3/<0.001	0.24/<0.001	0.77/<0.001
Unknown[1]	-----	-----	-----	-----	1/<0.001
Unknown[2]	-----	-----	6.3/0.001	-----	-----
hydroxydiazinon	<0.01/<0.001	0.11/<0.001	0.02/<0.001	<0.03/<0.001	1.4/<0.001
diazinon	0.03/<0.001	<0.08<0.001	<0.89/<0.001	<0.0.04/<0.001	2/<0.001
Metabolite M3	2/0.002	5.7/0.008	2.3/<0.001	----	-----
Glucuronide & other conjugates	23.5/0.026	24.6/0.04	9.7/0.002	----	-----
Metab. M3 + glucuronide & other conjugates	-----	-----	-----	22.4/0.006	9.7/0.001

[1] Unresolved CGA-14128 suspected
[2] Unresolved GS-31144 and G-27550 suspected

Plant metabolism

The fate of diazinon in plants was described in the 1993 periodic review and a diagram of the proposed metabolic pathways was presented. The metabolic route in plants is summarized briefly here for convenience. Metabolism in plants progresses, as in animals, primarily by hydrolysis of the ester linkage, yielding metabolite B1 (G-27550), followed by oxidation of the isopropyl group to primary and tertiary alcohols and/or oxidation of the methyl group to the alcohol. Glucose or malonylglucose conjugates are formed from the alcohols. Diazoxon was not reported as a significant plant metabolite although low levels were found in mammals.

The major residues reported in various crops in the 1993 evaluation in decreasing order for each crop are were as follows.

Apple	Beans	Maize forage	Lettuce	Potato foliage[1]
diazinon/ G-27550	G-27550 GS-31144 JAK-III-57[2] diazinon	G-27550 JAK-III-57 GS-31144 diazinon	diazinon GS-31144 GS-31144	G-27550 (CL-XIX-29[3]/ conjugate) JAK-III-57 GS-31144 G-27550 diazinon

[1]Diazinon is extensively metabolized in the tuber
[2]G-27550 with the methyl group oxidized to the alcohol, see 1993 evaluation, Figure 2
[3]G-27550 oxidized on the primary carbon of the isopropyl, see 1993 evaluation, Figure 2

Environmental fate in soil

This is described in the 1993 JMPR evaluations.

Environmental fate in water/sediment systems

No information was provided either to the 1993 or the present Meeting.

METHODS OF RESIDUE ANALYSIS

Analytical methods

The 1993 JMPR monograph describes methods which have been used for the residue analysis of samples from crops and animals. The methods summarized in the 1993 monograph and those submitted to the present Meeting are tabulated below. References are given in the text.

Method	Year	Limit of determination, mg/kg	Detector	Substrates
1993 JMPR				
REM 7a/73	1973	0.02	FID	Apple, lettuce, bean leaves
REM 15/82	1982	0.02	NP	Cherry, lettuce, cocoa seed
REM 119.01	1989	0.01	EC or NP	Kiwifruit, maize (whole plant)
AG-550A	1990	0.01-0.02[1] diazinon, diazoxon	FPD	(21 crops, almonds, corn oil, animal tissues
		0.02[1] hydroxydiazinon		
		0.05 ditto		Hops
	Generally 0.02 mg/kg may be a more practical limit of determination for diazinon and metabolites in animal products via AG-550A, although 0.01 mg/kg may be attainable in some cases.			
1996 JMPR				
Method 113	Undated	0.02	Thermionic	Sheep tissues and fat
Method 29/73	1973	0.02-0.05[1]	AFID	Meat and milk
Method 4/74	1974	0.02[1] diazinon	FPD	Animal tissues
		0.05[1] hydroxydiazinon		
		0.2[1] G-27550		
REM 21/86	1987	0.02[1]	NPD	Muscle, liver, kidney, fat
Netherlands Official Methods	1988	0.01-0.05	NPD	Crops
		0.01-0.04		Meat, tissues
		0.001-0.01		Milk
REM 128.02	1991	0.02[1]	NPD	Milk

Method	Year	Limit of determination, mg/kg	Detector	Substrates
Method 132A	1994	0.01	NPD-thermionic	Milk
Method 132B	1992	0.01-0.02[1]	N-P	Butter
Method 135	1994	0.01	thermionic	Muscle, liver, fat

[1] Estimate by the present Meeting (often twice the reported value). In all other cases the reported value could not be confirmed with information provided. LODs apply to diazinon unless otherwise indicated.

Method AG-550A, which has been used extensively for animal products, is discussed below. Other methods summarized by the 1993 JMPR will not be described again.

Method AG-550A (Hubbard *et al.*, 1990) involves extraction of crops and various animal tissues and milk with acetone/water, partitioning into petroleum ether/methylene chloride, concentration, dissolution in acetone and GLC with a flame-photometric detector. It was tested on 21 crops. Some variation is provided for selected samples. For hops the solvent is evaporated and the residue dissolved in hexane and partitioned into acetonitrile before evaporation of the acetonitrile and transfer to acetone for analysis. Corn oil is extracted directly with acetonitrile which is similarly evaporated and the residue transferred to acetone. Beef fat is extracted with hexane before partitioning into acetonitrile, otherwise its treatment is similar to corn oil. An alternative mini-Florisil column clean-up is provided to remove material which interferes with GLC.

Method AG-550A was used in most of the US trials. More importantly, it was the method used in the animal transfer studies reviewed here, in which it was used to determine diazoxon and hydroxydiazinon as well as diazinon. Generally a limit of determination of 0.01 mg/kg (0.05 mg/kg in hops) is reported to be achievable for diazinon and the metabolites with use the preferred capillary columns, and 0.025 mg/kg for diazinon and 0.05 mg/kg for the metabolites with packed columns.

The validations and sample chromatograms provided were generally consistent with the estimated limits of determination, with analytical recoveries generally ≥96% for all three compounds in meat, eggs, fat and milk at a fortification level of 0.01 mg/kg. The exception was 68% for the determination of diazoxon in beef liver. Recoveries were similar in the transfer studies, but again a little lower in liver and kidney. The method was also validated (Hubbard, 1990) for crops and animal tissues with [^{14}C]diazinon in fortified samples and in goat tissues from animal metabolism studies.

Representative chromatograms in the animal transfer study by Selman (1994a) also suggest that 0.01 mg/kg of all three compounds can probably be determined in milk and tissues, although the results were not as convincing with kidney and fat, especially for hydroxydiazinon for which 0.02 mg/kg would appear to be a more reasonable limit of determination. Also on the basis of sample chromatograms in poultry transfer studies (Selman, 1993) 0.02 mg/kg may be a more practical limit of determination, especially for hydroxydiazinon. Measurement with confidence at 0.01 mg/kg may be possible, however, at least for fat and eggs.

Method 113 (Anon., undated) determines diazinon in the fat and tissues of sheep and is based on extraction with hexane, sweep co-distillation and determination by GLC and a thermionic detector. A "method sensitivity" of 0.02 mg/kg was reported, but no information on validation or sample chromatograms were provided.

REM 29/73 (Formica, 1973) for diazinon involves extraction of meat with methanol and milk with acetone, partitioning into chloroform, clean-up on an alumina column and determination by GLC with either flame-photometric or alkali flame-ionisation detectors. The reported limit of detection was 0.01 mg/kg in meat and milk. Recoveries were ≥94% at fortification levels of 0.03 mg/kg in milk and 0.05 mg/kg in meat. Because sample chromatograms of controls showed no really quantifiable residues and since the method was not validated below 0.03 or 0.05 mg/kg, a limit of determination of 0.02-0.05 mg/kg should be achievable.

REM 4/74 (Formica, 1974) was developed for the determination of diazinon, diazoxon, hydroxydiazinon and 4-hydroxy-2-isopropyl-6-methylpyrimidine (G 27550) in animal tissues. For the determination of diazinon, hydroxydiazinon and G 27550, the tissues are macerated or extracted with methanol, the extract is diluted with 1 N HCl and diazinon and hydroxydiazinon are extracted with chloroform. G 27550 is likewise extracted with chloroform after neutralization of the HCl. Diazinon and hydroxydiazinon are cleaned up on an alumina column (or by TLC) before GLC analysis with an FPD. G 27550 is determined with a nitrogen-selective electrolytic conductivity detector. Diazoxon is determined by cholinesterase inhibition, but as this is not a currently acceptable method it will not be further described.

The lowest levels at which analytical recoveries were measured were 0.1 mg/kg of diazinon and hydroxydiazinon and 0.2 mg/kg of G 27550, at which levels recoveries were generally ≥75% from sheep muscle, liver and fat and sow kidney and liver, but only 62% of G 27550 from sheep fat. The limits of detection of diazinon and hydroxydiazinon were reported as 0.01 and 0.02 mg/kg respectively. For G 27550 an interfering GLC peak resulted in a reported limit of detection of 0.1 mg/kg. From sample chromatograms and the validation levels, reasonable limits of determination in muscle would appear to be about 0.02 mg/kg for diazinon, 0.05 mg/kg for hydroxydiazinon and 0.2 mg/kg for G 27550. In the absence of sample chromatograms, the Meeting could make no estimates for other tissues.

In REM 21/86 (Netherlands, 1988) diazinon is extracted from homogenized muscle, liver or kidney with methanol, partitioned into hexane, and cleaned up on a phenyl-coated solid-phase extraction column. Heated fat is extracted with acetonitrile, the extract partitioned with hexane for clean-up, and the acetonitrile rotary-evaporated. The extract is taken up into hexane and cleaned up on a cyano-coated solid-phase extraction column. Determination is by GLC with an NP detector.

Analytical recoveries were ≥85% at a fortification level of 0.02 mg/kg from liver, kidney and fat. The limit of determination was reported to be 0.01 mg/kg. Sample chromatograms suggest that 0.02 mg/kg may be a more practical limit for sheep muscle and fat. Sample chromatograms were not provided for liver or kidney, so the Meeting could not estimate LODs for them.

The Netherlands Official multi-residue GLC Sub-method 1 (1988) for diazinon in fruits and vegetables involves extraction with ethyl acetate and analysis by GLC with a phosphorus-specific detector without further clean-up. For crops recoveries of 80% (fortification level unspecified) and limits of determination of 0.01-0.05 mg/kg are reported. In Sub-method 2 for the determination of diazinon in animal tissues extraction with acetone/acetonitrile is followed by evaporation, partitioning into acetonitrile from hexane and determination by GLC with an NPD. Recoveries of 66-102% and limits of determination of 0.01-0.04 mg/kg are reported. In Sub-method 3 for diazinon in milk, extraction with ethyl acetate is followed by evaporation, dissolution in hexane, partitioning with acetonitrile, evaporation, solution in ethyl acetate and analysis by GLC with a phosphorus-specific detector. Recoveries of 75-100% and limits of determination of 0.001 to 0.01 mg/l are reported. Recoveries and limits of determination were not included in the submission to the JMPR.

REM 128.02 (1991) was developed for the determination of diazinon in blood and milk. The method for milk is based on Method AG-550A, but the initial acetone/water extract is cleaned up on a C-18 solid-phase cartridge before GLC determination with an NPD. The "lower practical level" for milk by this method was reported as 0.008 mg/kg. At the lowest fortification level of 0.02 mg/kg the average recovery from milk was 104%. A sample chromatogram in the report suggested a general practical limit of determination of 0.02 mg/kg, although 0.01 mg/kg might be achievable.

Method 132A (1994) for the determination of diazinon in milk is similar to AG-550A, but residues are extracted with acetone and transferred to methylene chloride, which is evaporated. The residue is taken up into hexane, partitioned into acetonitrile, concentrated into added methanol and finally cleaned up on an alumina column. A recovery of 86% is reported at the lowest fortification level in the report (0.1 mg/kg). The limit of determination is reported to be 0.01 mg/kg, although sample chromatograms of controls and fortified samples were not available for independent confirmation.

Method 132B (1992) determines diazinon in butter. The butter is dissolved in hot hexane and the residues partitioned into acetonitrile, which is evaporated to dryness. Clean-up is on an alumina column and determination by GLC with an NP detector. The average recovery was 90% at the lowest fortification level (0.02 mg/kg). A limit of determination of 0.01 mg/kg is reported and a sample chromatogram indicates that that should be achievable.

Method 135 (1994) determines diazinon in fat and animal tissues. Muscle and liver are macerated with methanol, which is diluted with water and extracted with methylene chloride. This is evaporated and the residue cleaned up on an alumina column. Fat is ground with sodium sulfate, extracted with hot hexane, and cleaned up by partitioning into acetonitrile. The acetonitrile is evaporated and the residue taken up in hexane and further cleaned up on an alumina column. Determination is by GLC with thermionic detection. Analytical recoveries of 84% from fat, 90% from muscle and 82% from liver were reported at the lowest fortification level of 0.1 mg/kg. The limits of detection and determination were reported to be 0.01 mg/kg, but the report did not include sample chromatograms of controls and fortified samples for independent confirmation.

Stability of pesticide residues in stored analytical samples

The studies of storage stability in crops and processed commodities (Beidler and Moore, 1991) and animal tissues (Schnabel and Formica, 1981) reviewed and described by the 1993 JMPR were re-submitted. From the latter study the 1993 JMPR noted that diazinon residues in animal tissues were stable for at least 8 months. The study was on samples from sheep which had been dipped in diazinon and contained initial residues of 0.05-2 mg/kg in muscle, ≤0.1 mg/kg in liver, 0.08-0.5 mg/kg in kidney and 2.8-5 mg/kg in fat. Samples were stored at -20°C. The study did not include information on the storage stability of metabolites in tissues, or of diazinon or metabolites in milk.

USE PATTERN

Information was provided on GAP for uses on both crops animals. Information on crop uses (Netherlands, 1995; Thailand 1995; Norway 1995) is not summarized here, since the emphasis is on residues in animal products.

The most important formulations for ectoparasite control in animals are listed above. WP formulations are also available. All are diluted with water for animal treatment by dipping, spraying,

wash or pour-on, and formulations as dusting powders and ear tags are also available. The most important treatment rates generally recommended are 600 mg/l for dipping cattle and for spraying cattle, sheep and goats, and 250 mg/l for dipping sheep and spraying goats.

Sheep are likely to incur the highest residues because of their wool coat and the high solubility of residues in wool grease (lanolin). Morrison (1994) demonstrated the bioequivalence of different EC formulations in sheep. This led the manufacturer to conclude that similar bioequivalence could be expected for these formulations in other species such as cattle, pigs and goats.

Although information on GAP for the use of diazinon in animal health was provided for over 55 countries and was consulted by the Meeting as needed, the summary in Table 6 below is largely confined to those countries in which supervised trials were conducted, and in some cases neighbouring countries.

Table 6. GAP for the use of diazinon for ectoparasite control on animals.

Animal/ Country	Form.	No. of treatments	Application rate			Pre-slaughter (S) or milking (M) interval	Comments[1]
			mg ai/kg bw (approx.)	l/animal	mg ai/l		
Wound dressings							
CATTLE Australia	Powder 20 g/kg	if needed	not defined	not defined	not defined	S 3	wound dusting S14
SHEEP Australia	Powder 20 g/kg	if needed				S 14	S14
	EC	--	--	--	600-1000	--	saturate wound area
Ear tags							
CATTLE Australia	Ear tags 200 g/kg 15 g/tag	two tags/animal	not defined	not defined	--	"Nil"	S17
Canada	Ear tags 200 g/kg 10.5 g/tag	1-2 tags/animal	2.1-4.2 g ai/animal[2]	not defined	--	"Nil"	S22
Backrubs							
CATTLE Australia	EC	pest-dependent	not defined	--	10 g/L oil	S 3	S16
Dips							
SHEEP Australia	EC	pest-dependent	not defined	not defined	100-200 (plunge/shower dip)	S 14	S15
Egypt[3]	EC	≤3	--	--	250	S 14 M 3	S2
France	EC	1-2/yr	--	--	250	S 14 M 3	S18
Ireland	EC	4-5 wk intervals	--	--	400	S 14 M no inf.	dip at least 1 min. S5
	EC	1-2? at 4-5 wk intervals	--	--	400 (winter dip)	S 14	dip at least 1 min. S5
Morocco	EC	≤3	--	--	250	S 21 M 4	S7
Netherlands	EC	≤3		--	250	S 28	S7

Animal/ Country	Form.	No. of treatments	Application rate			Pre-slaughter (S) or milking (M) interval	Comments[1]
			mg ai/kg bw (approx.)	l/animal	mg ai/l		
						Not for milk	
New Zealand	EC	pest-dependent	--	--	200-400 (shower or plunge dip)	S 21 ewes for milk not treated	S13
Norway	EC	≤3		--	250	S 21 Not for milk	S7
Portugal	EC	≤3		--	250	S 14	S7
Spain	EC	≤3		--	250	S 14 M 3	S7
Switzerland	EC	≤3	--	--	250	S 21 M 4	S7
UK	EC	1-3? (unclear)	--	--	250 dip or shower dip (winter dip)	S 14 ewes for milk not treated	dip at least 1/2 min. S5
CATTLE Austria	EC	≤3	6	--	600	M 5	S6
Egypt[3]	EC 600 g/L	≤3	6	--	600	S 14 M 3	S1
Morocco	EC	≤3	6	--	600	S 21 M 4	S6
Netherlands	EC	≤3	6	--	600	S 28 Not for milk	S6
Norway	EC	≤3	6	--	600	S 21 Not for milk	S6
Portugal	EC	≤3	6	--	600	S 14 M 3	S6
Spain	EC	≤3	6	--	600	S 14-15 M 3	S6
Switzerland	EC	≤3	6	--	600	S 21 M 4	S6
Sprays							
SHEEP Australia	EC	pest-dependent	8-24	1-3	400 jetting	S 14	S15
	spray-on93.3 g/kg	1 off-shear wide-band back spray-on	47	3 ml/kg bw of 1 in 7 dilution	13330	S 21	milk not for human consumption S15
Austria	EC	≤3	36	3	600	S 7 M 5	
Egypt[3]	EC	≤3	36	3	600	S 14 M 3	S2
France	EC	1-2/yr	--	--	550	S 14 M 3	S18
Morocco	EC	≤3	36	3	600	S 21 M 4	S7
Netherlands	EC	≤3	36	3	600	S 28 Not for milk	S7
New Zealand	EC	pest-dependent	500 max./ animal	0.5-1	500 jetting lamb treatment	S 21 Ewes for milk not treated	S13

Animal/ Country	Form.	No. of treatments	Application rate			Pre-slaughter (S) or milking (M) interval	Comments[1]
			mg ai/kg bw (approx.)	l/animal	mg ai/l		
Norway	EC	≤3	36	3	600	S 21 Not for milk	S7
Portugal	EC	≤3		--	600	S 14	S7
Spain	EC	≤3		--	600	S 14 M 3	S7
Switzerland	EC	≤3	36	3	600	S 21 M 4	S7
CATTLE Australia	EC	pest-dependent spray as needed	8	2.3	1000	S 3	Low volume S16
		spray as needed	8	4.5	500	S 3	High volume S16
		spray as needed	1.3	0.5	600-800 Back line spray	S 3	S16
Austria	EC	≤3	6	--	600	S 7	
Egypt[3]	EC	≤3	6	3	600	S 14 M 3	S1
France	EC	≤2/yr	6	--	550	S 14 M 3	S18
Morocco	EC	≤3	6	3	600	S 21 M 5	S6
Netherlands	EC	≤3	6	3	600	S 28 Not for milk	S6
Norway	EC	≤3	6	3	600	S 21 Not for milk	S6
Portugal	EC	≤3	6	3	600	S 14 M 3	S6
Spain	EC	≤3	6	3	600	S 14-15 M 3	S6
Switzerland	EC	≤3	6	3	600	S 21 M 4	S6
GOATS Australia	EC	pest-dependent	30	3	500	S 14	S16
France	EC	≤2/yr	2.5	1	550	S 14	S18
Egypt[3]	EC	≤3	36	3	600	S 14 M 3	S3
Morocco	EC	≤3	6	3	600	S 21 M 4	S8
Norway	EC	≤3	6	3	600	S 21 Not for milk	S8
Spain	EC	≤3	6	3	600	S 14 M 3	S8
Switzerland	EC	≤3	36	3	600	S 21 M 4	S8
PIGS Australia	EC	pest-dependent	5	1	500	S 14	S17
Austria	EC	≤3	6	1	600	S 7	S9
Egypt[3]	EC	≤3	2.5	1	250	S 14	S4
France	EC	2/yr	2.5	1	250	S 14	S19
Morocco	EC	≤3	6	1	600	S 21	S9
Netherlands	EC	≤3	6	1	600	S 28	S9

Animal/ Country	Form.	No. of treatments	Application rate			Pre-slaughter (S) or milking (M) interval	Comments[1]
			mg ai/kg bw (approx.)	l/animal	mg ai/l		
Norway	EC	≤3	6	1	600	S 21	S9
Portugal	EC	≤3	6	1	600	S 14	S9
Spain	EC	≤3	6	1	600	S 14	S9
Switzerland	EC	≤3	2.5	1	250	S 21	S9

[1]S1, S2, ... etc. are cross-references to the Table number of Ectoparasite Vol. 1 GAP Table summary submission from which, along with labels, the information is derived.
[2]Assumes 10.5 g product/tag containing 20% diazinon
[3]And several neighbouring countries

RESIDUES RESULTING FROM SUPERVISED TRIALS

CXLs are currently established at 0.7 mg/kg for the meat (fat) of cattle, goats and pigs and 0.02 mg/kg for milk. The 1993 JMPR reviewed residues resulting from the use of ear tags but not those from other uses on animals. Mainly because of the lack of animal transfer studies the 1993 JMPR recommended withdrawal of these CXLs, noting that data from veterinary uses were also desirable. The 1995 CCPR recommended retention of the limits pending the 1996 JMPR review of promised data on both transfer to animals and treatments against ectoparasites.

Animal feeding studies

The results of studies on cattle and poultry were available.

Cattle. Residues of diazinon, diazoxon (G-24576) and hydroxydiazinon (CGA-14128) were determined in the blood, milk and tissues of Holstein dairy cows dosed with technical diazinon after the evening milking for 28-30 consecutive days by gelatin capsule at rates equivalent to 40 ppm (1X), 120 ppm (3X) and 400 ppm (10X) in the diet (Selman, 1994a,b). Separate reports were provided of the analytical phase (Perez and Wetters, 1994) and the biological phase (Krautter, 1994) of the study, which was based on specified protocols (Selman, 1992a).

Three cows were dosed at each rate and one served as a control. The 1X rate was based on worst-case estimates of residues of 10 mg/kg in bean forage and pea vines each fed at 50% of the diet, converted to a dry-weight basis (34.3 ppm dietary burden) and the dosing took into account the mean dry-weight food consumption by the test animals.

Blood was sampled just before slaughter and composite samples of morning and evening milk were taken from each cow before dosing and 1, 3, 7, 14, 21 and 27 days after the first dose. Liver, kidney, round muscle, tenderloin muscle, and perirenal and omental fat were taken within 18-24 hours after slaughter. All samples were handled and stored satisfactorily (-20°C storage). Storage was for 4-5 months before analysis. As noted earlier, information on storage stability has been provided only for diazinon *per se* and only in tissues, not in milk.

The analytical method used was AG-550A. Results were corrected for procedural recoveries but not for control values. The results of recovery experiments were corrected for control values and diazoxon and hydroxydiazinon were expressed as diazinon (factors of 1.052 and 0.950 respectively). At the 0.01 mg/kg fortification level the percentage recoveries were:

	diazinon	diazoxon	hydroxydiazinon
Liver	101	77	111
Kidney	91	82	104
Muscle[1]	115	107	121
Fat[1]	109	106	113
Milk[1]	108	103	113
Blood	96	35	102

[1] mean recoveries

No residues of diazoxon or hydroxydiazinon were found in milk or any of the tissues at any interval or dosing level with the exception of hydroxydiazinon in omental and perirenal fat, which ranged from 0.01 to 0.06 mg/kg from the 10X dose. The residues of diazinon are shown in Table 7.

Table 7. Residues of diazinon in blood, milk and tissues from dosing diary cows with diazinon for 28-30 consecutive days at rates equivalent to 40, 120 and 400 ppm in the diet (Selman, 1994a,b).

Sample		Diazinon, mg/kg, from equivalent of		
		40 ppm (1X)	120 ppm (3X)	400 ppm (10X)
Liver		<0.01	<0.01	<0.01
Kidney		<0.01	<0.01	<0.01-0.01
Blood		<0.01	<0.01	<0.01
Muscle, round		<0.01	<0.01	<0.01-0.02
Muscle, tenderloin		<0.01	<0.01	0.01-0.02
Fat, perirenal		<0.02-0.03 (0.02)	0.05-0.08 (0.06)	0.15-0.58 (0.4)
Fat, omental		0.02-0.04 (0.03)	0.07-0.1 (0.08)	0.2-0.84 (0.6)
Milk	Days 1	<0.01	<0.01	<0.01-0.05 (0.02)
	3	<0.01	<0.01	0.01-0.06 (0.04)
	7	<0.01	<0.01	0.02-0.08 (0.04)
	14	<0.01	<0.01	<0.01-0.06 (0.03)
	21	<0.01	<0.01-0.01	<0.01-0.03 (0.02)
	27	<0.01	<0.01	<0.01-0.03 (0.02)

Poultry. A study on poultry described by Selman (1992b, 1993) was derived from supporting studies of the biological phase by March and Rezold (1992) and the analytical phase by Perez and Wetters (1992) in accordance with specified protocols (Selman, 1991).

White Leghorn hens (3 groups of five birds at each dose level) were dosed with technical grade diazinon by gelatin capsule at rates equivalent to 0, 0.5 (1X), 1.5 (3X) and 5 ppm (10X) for 28 consecutive days. Feed and water were *ad libitum*. Eggs were collected from all the birds at 0, 3, 7, 14, 21 and 28 days and the hens were killed after 28 days. Composite samples of breast and thigh muscle, skin and attached fat, peritoneal fat and liver were taken on the next day. Samples were transported in dry ice to the laboratory where they were stored at -20°C until analysis within approximately 5 months of sampling. The diazinon content of the capsules was confirmed by analysis.

The samples were analysed by AG-550A for diazinon, diazoxon (G-24576) and hydroxydiazinon (CGA-14128). Procedural recoveries at the 0.01 mg/kg fortification level were respectively 103, 103 and 99% (means) from eggs; 120, 126 and 123% from muscle; 95, 87, and 107% from skin with attached fat; 120, 81 and 117% from fat and 99, 63 and 89% from liver, with all controls <0.01 mg/kg. Sample chromatograms from fat and eggs fortified at 0.01 mg/kg suggest that although 0.01 mg/kg may be attainable, 0.02 mg/kg would be a practical limit of determination. Other sample chromatograms were from fortification levels of 0.1 mg/kg in muscle, 0.05 mg/kg in skin/fat, and 0.5 mg/kg in liver.

No residues (<0.01 mg/kg) of diazinon, diazoxon or hydroxydiazinon were found in any sample at any dose rate.

Uses as an ectoparasiticide

Eight trials were reviewed by the JMPR from 1967 to 1975 and 3 trials with ear tags were reviewed in 1993. All of these were re-submitted to the present Meeting, together with studies not previously reviewed: 9 with sprays (6 on cows, 2 on goats, 1 on pigs), 4 with cattle ear tags, 3 with sheep dips and one trial each on sheep dressing, sheep jetting and cattle backrubbing.

Dust application. In one very old briefly described study 3 cows were hand-dusted from a sprinkler can with 2% w/w diazinon dust (124 g/animal) and milk was analysed by GLC 2, 5, 9 and 24 hours after application (Chilwell *et al.*, 1967). Analytical recoveries averaged 79% at a fortification level of 0.1 mg/kg. The residues (mg/kg) were as shown below.

	2 h	5 h	9 h	24 h
Range	0.01-0.02 0.03-0.05	0.05-0.1	0.05-0.07	
Mean	0.01	0.04	0.09	0.06

No GAP was reported for this use.

Wound dressing. In an old Australian study residues of diazinon were determined in the fat, muscle and liver of fly-struck sheep 10 days after treatment (5 animals at each rate) with a 2% ai powder formulation applied as a wound dressing at 10 and 30 g/animal (Bull, 1974). The lower rate was reported to be according to GAP but this could not be confirmed with the label provided. Residues (mg/kg) were as shown below.

	g/sheep	Liver	Muscle	Omental fat
Range	10	<0.01-0.01	0.01-0.03 0.05-0.08	
Mean		<0.01	0.01	0.06
Range	30	<0.01-0.01	0.01-0.02 0.08-0.1	
Mean		<0.01	0.02	0.09

Backrubs. Two studies were available. The first (Bourne and Arthur, 1967) was a very old published US study in one part of which 56.6 g of 2% diazinon dust formulated on Pyrex ABB with 1% motor oil was hand-rubbed on the backs of 5 cows daily for 4 days. Half a day after the last treatment diazinon residues in the milk ranged from <0.05 to 0.52 mg/kg and after intervals of 1 to 15 days they were <0.05 mg/kg.

In the second part of the study 5 different cows were rubbed several times across the back with a burlap backrubber (1.2 m long X 10.2 cm diameter) impregnated with 0.45 kg of 2% diazinon dust. It was not clear whether the applications were daily but they were probably daily for four consecutive

days as in the other part of the study. Diazinon residues in the milk as determined spectrophotometrically ranged from <0.05 to 0.23 mg/kg 0.5 days after the last treatment to <0.05 mg/kg in all samples from 1 to 15 days after treatment. There was no information on relevant US GAP.

The second study was a recent Australian trial on 2- to 3-year old Brahman male cattle in which the backrubber was charged with 500 ml of 200 g/l diazinon EC per 10l of sump oil (i.e. 10 g ai/l oil). This is reported Australian GAP, which also includes a 3-day withdrawal period (Rose, 1995; Queensland and New South Wales, 1996). Groups of 5 cattle were exposed for 10 days and slaughtered 1 or 2 days after treatment, or for 19 days and slaughtered after 4, 7 or 10 days. Samples of renal and/or loin fat were taken from the 5 cattle in each group at each interval. The residues are shown in Table 8.

Table 8. Residues of diazinon in renal and loin (subcutaneous) fat of groups of 5 cattle 1 to 10 days after backrubber treatment at 10 g ai/l with EC formulation ((Rose, 1995; Queensland and New South Wales, 1996)

Diazinon, mg/kg, at interval, days, after treatment						
1[1]	2[1]	4[2]		7[2]		10[2]
Loin	Loin	Loin	Renal	Loin	Renal	Loin
0.07	0.08	0.66	0.26	0.1	0.08	0.03
0.04	0.05	0.24	0.16	0.05	0.08	0.03
<0.02	0.04	0.07	0.05	0.14	0.06	0.03
0.31	0.12	0.16	0.16	0.15	0.06	0.1
0.04	0.03	0.34	0.17	0.08	0.04	0.07

[1] Exposed for 10 days [2] Exposed for 19 days

Ear tags. Reports of three Canadian and four Australian trials were available. The results are shown in Table 9.

Table 9. Residues of diazinon in milk, milk fat and tissues of cows or calves treated with diazinon ear tags.

Country, year, ref., dose	Sample	Days after tag application	Residue, range and (mean)[1]
Canada 1987 Surgeoner, *et al.*, 1987a Vol 2 of 3 ref. 9 Two tags/cow 11% diazinon	whole milk	5 h through 1 day	ì g/kg this study only ND (= <0.5 ì g/kg) (ND)
		3	ND - 1.4 (0.64)
		7	1.2-1.7 (1.4)
		14	1.1-1.7 (1.4)
		21	ND-1.8 (0.53)
		28	0.73-1.4 (1.1) 3 cows

Country, year, ref., dose	Sample	Days after tag application	Residue, range and (mean)[1]
Canada 1987 Surgeoner *et al.*, 1987b Vol. 2 of 3 ref. 10 Two tags/steer, 9.6% diazinon	back fat (omental)	14 ≥100	0.032, ND (= ≤0.01 mg/kg) ND
	kidney fat (perirenal)	14 ≥100	0.035, ND ND
	muscle or liver	14 to ≥100	ND
	tongue	≥100	ND 3 cows
Canada 1989 Surgeoner *et al.*, 1989 Vol. 2 of 3 ref. 11 Two 20% diazinon tags/steer	back fat (centre, subcutaneous)	7 14	0.01 0.05 0.03, 0.02
	kidney fat (perirenal)	7 14 28	0.03 0.04 0.03, 0.03
	kidney, liver or muscle	7, 14, 28	all ND (= ≤0.01)
	tongue	7 14 28	ND 0.02 ND -0.02 4 steers
Australia, 1989 Bull and Wicker, 1989 Vol. 2 of 3, ref. 19 Two 18% diazinon tags/cow	Milk fat (butter)	1 7 14 28	<0.01-0.02 (<0.01) <0.01-0.01 (<0.01) <0.01-0.01 (<0.01) 0.01-0.02 (0.0125) 4 cows
	Milk (not analysed, but estimate based on the milk fat residue)	1-28	<0.01
Australia, 1990 Mawhinney, 1990 Vol. 2 of 3, ref. 18 One 18% diazinon tag/calf or cow (1/2 max. GAP)	fat (biopsy, subcutaneous tail butt)	calves 7 42 43	 <0.01-0.03 (0.02) 0.01-0.03 (0.02) <0.01-0.01
		cows 7 44	 <0.01-0.01 <0.01
Australia, 1992	milk fat (butter)	1 7 14	0.04-0.08 (0.06) 0.12-0.26 (0.19) 0.06-0.18 (0.13) 5 cows
Bull and Swindale, 1992 Vol. 2 of 3, ref. 15 Two 15 g 20% diazinon tags/cow	whole milk (highest residues assuming 4% fat)[1]	1 7 14	0.003 0.01 0.007
Australia, 1993 Strong and Bull, 1993 Vol. 2 of 3, ref. 16 Two 15 g 20% diazinon tags/cow	fat of milk	1 2 3 7 10 14 28 42 56	<0.01-0.02 (<0.01) <0.01-0.01 (<0.01) <0.01-0.02 (<0.01) 0.01-0.02 (0.02) 0.01-0.02 (0.02) 0.02(4) (0.02) 0.02(4) (0.02) 0.01-0.03 (0.02) 0.01-0.02 (0.02)

Country, year, ref., dose	Sample	Days after tag application	Residue, range and (mean)[1]
		84	<0.01-0.01 (<0.01)

[1] mg/kg unless otherwise indicated

The three Canadian trials were reviewed by the 1993 JMPR and will not be described in detail here. In the first (Surgeoner *et al.*, 1987a) the highest residues in milk during the 28 days of the trial were 0.0018 mg/kg (on day 21) with two 11% diazinon ear tags applied to each of three dairy cows. In the second study (Surgeoner *et al.*, 1987b) with two 9.6% diazinon ear tags on each of three steers, the maximum diazinon residues were 0.035 mg/kg in perirenal fat and 0.032 mg/kg in omental fat 14 days after the application of the tags. No residues (≤0.01 mg/kg) were found in muscle or liver. Residues in the fat decreased to <0.01 mg/kg after 100 days. In the third study (Surgeoner *et. al.*, 1989) with two 20% diazinon tags on each of 4 steers, the highest residues in the 28-day treatment occurred after 14 days with 0.45 mg/kg in back subcutaneous fat, 0.041 mg/kg in perirenal fat and 0.016 mg/kg in tongue. No residues (≤0.01 mg/kg) were reported in muscle, liver or kidney. This study appears to be in accordance with Canadian GAP (see Table 6).

In the first of four Australian trials two ear tags (18% diazinon, 4% Æ-cypermethrin; tag weights unspecified) were applied to each of three Fresian dairy cows and milk samples (two milkings/day) were taken 1, 7, 14 and 28 days after application (Bull and Wicker, 1989). Butter was immediately separated and stored at -15°C until analysis for diazinon by method 132A. The residues were <0.01-0.02 mg/kg on days 1 and 28 and <0.01-0.01 mg/kg on days 7 and 14. Mean procedural recoveries of diazinon from butter of 90% and a limit of determination of 0.01 mg/kg were reported, although sample chromatograms were not included.

In the second trial three *Bos indicus* calves (*c*. 200 kg) and three Brahman cows (400-660 kg) were each treated with a single ear tag impregnated with 18% diazinon and 4% Æ-cypermethrin (tag weights unspecified). Subcutaneous fat was taken by biopsy from fat pads on the side of the tail butt at 7, 42 and 83 days after treatment from the calves and after 7 and 44 days from the cows (Mawhinney, 1990). The highest diazinon residues in the fat from the calves were 0.03 mg/kg after 7 and 42 days and 0.01 mg/kg after 83 days. In the cows the residues were <0.01-0.01 mg/kg at 7 days and <0.01 at 44 days, with controls for both calves and cows <0.01 mg/kg.

The analytical procedure consisted in extraction of the fat with hexane, partitioning with acetonitrile, clean-up on a Florisil column and determination of diazinon by GLC with an NP detector. The reported limit of "detection" was 1 ng/g (0.001 mg/kg at a signal:noise ratio of 5:1). Sample chromatograms suggest that detection at 0.001 mg/kg is possible and that 0.01 mg/kg would probably be a reasonable limit of determination, although controls were not included among the sample chromatograms. At the 0.01 mg/kg fortification level procedural recoveries of diazinon averaged 82%. Diazinon in fat samples from treated animals was confirmed by mass spectrometry.

In the third Australian study two 15 g tags (20% diazinon) were applied to each of 5 Friesian cows and milk was sampled after 1, 7, and 14 days (Bull and Swindale, 1992). Cream was separated from the milk and stored in a refrigerator for 5 days. Butter was then made and stored at -15°C until analysis. The analytical phase was reported to be according to OECD GLP.

The highest residue in the butter of any one cow was 0.26 mg/kg on day 7 with mean residues for

the 5 cows of 0.06 mg/kg on day 1, 0.19 mg/kg on day 7 and 0.13 mg/kg on day 14.

The analytical method was 132B with procedural recoveries from butter ranging from 73 to 103% (n=4, mean 91.5%), excluding one recovery of 58% with fortification at 0.05 mg/kg. No sample chromatograms were provided.

In the fourth Australian trial (Strong and Bull, 1993) diazinon residues were determined in the milk fat of four Friesian cows treated with two 15g, 20% diazinon, ear tags for up to 3 months. Milk was sampled 1, 2, 3, 7, 10, 14, 28, 42, 56 and 84 days after attachment of the tags, immediately separated, and made into butter which was stored at -15°C until analysis. The study was started in October 1992 and the analyses were completed by January 1993: they were reported to be in accordance with OECD GLP.

The residues did not exceed 0.03 mg/kg in any sample. The mean residue for the four cows increased to 0.02 mg/kg by day 7 and remained at that level until day 56, decreasing to <0.01 mg/kg by day 84.

The method of analysis was again 132B with a mean procedural recovery of 87% at 0.02 mg/kg and a reported limit of determination of 0.01 mg/kg. No sample chromatograms were provided.

Dips. Six old trials (1962-1974, 3 on sheep, 3 on cattle) and two more recent trials on sheep (1986, 1989) were available. The results are given in Table 10.

Table 10. Residues of diazinon in the milk, tissues and fat of sheep and cattle following dipping in EC formulations.

Country, ref.	Rate, mg ai/l	No. of dips	Sample	Residues, mg/kg, range and (mean)	Slaughter interval (days)	Comments
SHEEP						
Australia, Hastie & Cavey, 1962a	200	1 for 20 sec	Meat Kidney fat Kidney fat Kidney fat Controls, meat & fat	<0.1 <0.1-1.4 (0.1) 1.1-1.4 (1.2) 0.5-1.4 (0.8) <0.1	1 1 7 14	3 animals per group for fat. Dip period reportedly twice recommended. Rate is GAP according to current label.
Switzerland, Formica, 1973b	200 (0.8 X GAP)	1 plunge dip	Whole milk Trial 1 Trial 2 Controls	Residues at day 6h 1 2 3 4 7 15 30 0.09 0.06 0.02 0.02 0.02 0.02 <0.01<0.01 0.09 0.03 0.01 0.01 0.01 0.01 <0.01<0.01 <0.01		
	400	1 plunge dip	Trial 3 Trial 4 Controls	Residues at day 6h 1 2 3 4 7 15 30 0.18 0.10 0.04 0.03 0.02 0.03 0.01 <0.01 0.16 0.07 0.04 0.02 0.02 0.03 <0.01<0.01 <0.01 Method REM 29/73, reported limit of detection 0.01 mg/kg, 92% recovery at 0.03 mg/kg		
Switzerland	750[1]	1	Muscle	0.21-0.37 (0.28)	10	Applic. rate 3X reported

Country, ref.	Rate, mg ai/l	No. of dips	Sample	Residues, mg/kg, range and (mean)	Slaughter interval (days)	Comments
Formica, 1974c			Liver Omental fat	<0.02 2.2-2.6 (2.3)		GAP Residues of hydroxydiazinon, diazoxon and pyrimidinol below LOD. Method REM 4/74, reported limit of detection 0.02 mg/kg parent; recovery 88% at 0.5 mg/kg
Australia Strong *et al.*, 1986a	250	1 plunge dip		Residues at day 1 3 7 14 21 muscle 0.15 0.08 0.05 0.03 0.01 0.13 0.05 0.04 0.01 0.02 liver 0.01 0.02 <0.01 <0.01 0.01 <0.01 <0.01 <0.01 0.01 0.01 kidney 0.04 0.03 0.02 0.02 0.01 0.03 0.03 0.01 0.01 0.01 kidney fat 2.6 2.2 1.6 0.67 0.29 1.2 2.1 1.0 0.63 0.24 Two sheep/interval. Method 114A, LOD 0.01 mg/kg; recovery ≥87% at 0.1 mg/kg		
UK Roberts and MacDonald, 1989	400 (1.6 X UK GAP, 1 X Ireland & N. Zealand GAP)	1 minute plunge		Residues at day 7 14[1] 21[2] 28 35 Omental fat 1.4 1.1 0.8 0.6 0.2 2.3 1.1 0.7 0.5 0.6 2.8 1.1 0.7 0.7 0.4 1.7 1.3 0.7 0.5 0.4 1.1 1.2 0.5 0.3 0.7 0.8 0.6 0.5 Mean (2.1) (1.1) (0.8) (0.6) (0.4) Subcut. fat --- 1.3 1.4 0.9 0.5 1.4 1.0 0.5 0.7 4.3 1.2 1.2 0.7 Mean (2.3) (1.2) (0.8) (0.6) Controls <0.005 mg/kg; 6 animals/interval (4 at 7 days) [1] Irish GAP PHI [2] New Zealand GAP PHI		
CATTLE						
Australia, Hastie, 1962	500	3	Kidney fat	3.2-3.9 (3.6)	18 h	4 animals. Swiss GAP is 600 mg ai/l and 21-day S.I. Same in Egypt & near countries, but 14 day S.I.
	EC[1]	2-3	Kidney fat	0.0-2.1 (1.5)	90 h	1 animal 3 dips 3 animals 2 dips
		2	Kidney fat	0.6-0.8 (0.7)	7	4 animals

Country, ref.	Rate, mg ai/l	No. of dips	Sample	Residues, mg/kg, range and (mean)	Slaughter interval (days)	Comments
			Control	0.1		
Australia Hastie 1963c	500	1	Kidney fat Subcut. fat Kidney fat Subcut. fat Kidney fat Subcut. fat Controls: Kidney fat Subcut. fat	0.4-1.5 (1) 0.2-0.3 (0.25) 0.4-0.6 (0.5) 0.15-0.2 (0.2) 0.3-0.6 (0.5) 0.4-0.7 (0.5) (0.2) <0.1	1 1 4 4 7 7	4 animals in each group
Australia Hastie 1963b	500	3 at 3 day interval	Kidney fat Subcut. fat Kidney fat Subcut. fat Kidney fat Subcut. fat	1.7-4 (2.7) 0.8-1.5 (1.2) 0.6-1.2 (0.8) <0.2-1.2 (0.7) 0.2-0.8 (0.5) 0.4	1 1 4 4 7 7	2 steers + 1 cow per group

[1] Formulation not stated but presumably EC

Hastie (1963a) reviewed several reports of studies on the dipping and spraying of sheep and cattle (Hastie, 1962, 1963b,c; Hastie and Cavey, 1962a,b, 1963). The reports were essentially brief summaries with little information on sample storage and handling, analytical methods or other details.

The other trials were on sheep dipping. Those by Formica (1973b, 1974c) are fairly well documented in terms of method, sample storage etc. The report by Strong *et al.* (1986) is reasonably detailed (e.g. sample storage at -15°C), although the periods from sampling to analysis are not provided. The trial by Roberts and MacDonald (1989) is the best documented and reported to be in accordance with recognized GLP. Details are given of all aspects of the trial, including sample storage and analysis of dip solutions.

Spraying. Reports of eighteen new or previously reviewed trials were provided: 13 on cattle, 2 on sheep, 2 on goats and 1 on pigs. The results are shown in Tables 11 (cattle) and 12 (sheep, goats and pigs).

Table 11. Residues of diazinon in the milk, fat and tissues of cattle after spraying.

Country, ref.	Formulation. Application rate, ai	No. of sprays	Sample	Residues, mg/kg, at withdrawal interval
Australia Hastie and Cavey, 1962a	Emulsifiable 1500 mg/l (3 X high vol. GAP) 9.5 l/animal (2 X high vol. GAP)	1	 meat kidney fat	1 — 7 — 14 days <0.1 - - 1.1 1.2 0.4 0.9 1.2 0.4 3.2 1.3 0.3 Residues in fat from 3 animals. Residue in meat from single animal. GAP S.I. (pre-slaughter interval) = 3 days
Australia Hastie and Cavey,	Emulsifiable 1000 mg/l	1	kidney fat	1 3 (GAP) days

Country, ref.	Formulation. Application rate, ai	No. of sprays	Sample	Residues, mg/kg, at withdrawal interval
1962b	(1 X low vol. or 2 X high vol. GAP)		(6 animals)	1.8, 1.2 1.0, 0.8 0.9, 1.1 0.7, 0.9 1.5, 1.1 0.9 --
	3.8 l/animal (High vol. GAP is 4.5 l)		subcut. fat (6 animals)	0.2, 0.1 0.3, 0.4 <0.1, 0.2 0.2, 0.7 0.3,<0.1 0.5, --
Australia Hastie and Cavey, 1963	Emulsifiable 1000 mg/l (2 X high vol. GAP) 7.6 l/animal (High vol. GAP)	1	milk (3 cows)	1 2 3 days 0.07 <0.01 Nil 0.05 <0.01 Nil 0.04 <0.01 Nil
	Emulsifiable 500 mg/l (High vol. GAP) 7.6 l/animal (High vol. GAP)	1	milk (3 cows)	0.03 <0.01 Nil 0.04 <0.01 Nil 0.04 <0.01 Nil
USA Claborn,*et al.*, 1963	WP 500 mg/l (1/2 max. low vol. GAP for Australia) 1-1.5 l/animal	16 at weekly intervals	omental fat Spray no.: 1	1 6 7 14 days <0.05, 0.06
			2	<0.05, 0.09
			6	0.2, 0.4 0.2, 0.2 0.5, 0.9 0.8,
			10	0.2, <0.08, 0.7
			11	0.4 <0.05
			16	
	WP 1000 mg/l (max. low vol. GAP for Australia)	16 at weekly intervals	1	0.7 0.5 0.6 0.5 0.5 0.8

Country, ref.	Formulation. Application rate, ai	No. of sprays	Sample	Residues, mg/kg, at withdrawal interval
				0.8 0.8 2.3 0.7 <0.05 2 6 10 16
USA Matthysse and Lisk, 1963	EC 600 mg/l 11.4 l/cow (high vol.)	2 10-day interval	Mil k 1st spray 2nd spray	0.5 1 2 3 4 7 days 0.3 0.09 0.03 <0.02 0.3 0.1 0.04 Residue at 4 days is mean of 2 cows. All others are means of 3cows.
UK Chilwell *et al*., 1967	Oil emulsion 200 mg/l 10 l/cow (high vol.)		Milk Cow 1 Cow 2 Cow 3 Mean	2 5 9 24 hours 0.01 0.06 0.07 0.02 0.02 0.06

Country, ref.	Formulation. Application rate, ai	No. of sprays	Sample	Residues, mg/kg, at withdrawal interval
				0.08 0.02 0.02 0.05 0.09 0.02 0.02 0.06 0.08 0.02 Each residue is mean of two analyses by GLC/thermionic. Recoveries 79% at 0.1 mg/kg.
Switzerland Blass, 1971	Formulation not stated 500 mg/l (600 mg/l is GAP) 10 l/cow 4 cows	4 (weekly intervals) (Max. 3 is Swiss GAP)	Milk Spray no. 1 Range Mean 2 Range Mean 3 Range Mean 4 Range Mean	0 3 4 (GAP) 6 days 0.2-0.4 <0.02-0.02 <0.02 (0.3) <0.02 (0.02) (<0.02) (<0.02) 0.1-0.2 <0.02-0.04 <0.02-0.02 (0.13) <0.02 (0.03) (0.02) (<0.02) 0.06-0.13 <0.02-0.03 <0.02-0.03 (0.09) <0.02 (0.02) (0.02) (<0.02 0.05-0.14 0.02-0.05 <0.02-0.04 (0.08) <0.02 (0.03)

Country, ref.	Formulation. Application rate, ai	No. of sprays	Sample	Residues, mg/kg, at withdrawal interval
				(0.03) (<0.02)
	1000 mg/l (1.7 X GAP) 10 l/cow 4 cows		1 Range Mean 2 Range Mean 3 Range Mean 4 Range Mean	0.2-0.6 <0.02-0.08 <0.02-0.05 <0.02 (0.3) (0.04) (0.04) (<0.02) 0.1-0.2 0.04-0.1 0.02-0.07 <0.02-0.04 (0.2) (0.07) (0.04) (0.02) 0.09-0.1 <0.02-0.08 <0.02-0.05 <0.02-0.02 (0.1) (0.05) (0.03) (0.02) 0.05-0.2 0.03-0.04 <0.02-0.05 <0.02 (0.14) (0.04) (0.03) (<0.02)
Australia Bull and Dougall, 1974	EC?	1	Milk & milk products Pre-treatment Milking Day 1st 1 2nd 1 3rd 2 4th 2 10th 5	Residues from 5 individual cows Whole milk Skim Range Mean milk[1] Butter[1] 0.03 0.02

Country, ref.	Formulation. Application rate, ai	No. of sprays	Sample	Residues, mg/kg, at withdrawal interval
				0.08 0.2-0.3 0.2 0.03 5.2 0.03-0.14 0.06 0.02 1.7 0.04-0.08 0.06 - - 0.8 0.02-0.05 0.03 - - 0.3 0.01-0.05 0.02 - - 0.05 [1]Composites Method 132, 0.01 mg/kg limit of detection reported. Recovery ≥97% from milk at 0.13 mg/kg
Bull and Dougall, 1974	500 mg/l (High vol. GAP) 10 l/animal (high vol.)		Milk & milk products	Residues from herd bulk storage Skim Pre-treatment Milk milk Milkings Cream Butter 1st 0.03 0.02 1st & 2nd 0.09 3rd 0.08 3rd & 4th 0.25 0.04 2.4 4.5 0.15 0.03 2.1 2.6 0.06 0.02 0.57 0.60 0.04 0.02 0.26 0.30 Herd size: 60. Method REM 20/71, autoanalyser.

Country, ref.	Formulation. Application rate, ai	No. of sprays	Sample	Residues, mg/kg, at withdrawal interval
				Recovery 76% at 0.05 mg/kg
Australia Bull *et al*., 1986a	EC 600 mg/l (1.2 X GAP)	1 10l/cow	Whole milk Pre-treatment MilkingHours 1 7 2 21 3 31 4 45 5 55 6 (GAP)7 7 80	# Range Mean (5 cows) <0.01 <0.01 0.08-0.4 0.2 0.05-0.1 0.08 0.04-0.07 0.06 0.01-0.03 0.02 0.01-0.03 0.02 <0.01-0.01 <0.01 <0.01 <0.01 GLC thermionic detection. Reported LOD 0.01 mg/kg, recovery 91% at 0.1 mg/kg. No corrections made to results.
Australia Strong *et al*., 1986b	EC 600 mg/l	1 10l/steer	 Muscle Liver Kidney Kidney fat Omental fat	1 7 14 21 days 0.06, 0.01, <0.01, NA[1] 0.06 0.01 <0.01 NA <0.01, <0.01, <0.01, NA 0.02 <0.01 <0.01 NA 0.07, 0.01, <0.01, NA 0.06 0.01 <0.01 NA 2.9, 0.4, 0.06, <0.01, 1.3 0.7 0.06 0.01 2.5, -- 0.2, <0.01, 1.4 -- 0.12 0.05

Country, ref.	Formulation. Application rate, ai	No. of sprays	Sample	Residues, mg/kg, at withdrawal interval
				GAP withdrawal interval is 3 days. [1]Not analysed. Two steer calves sampled at each time. -15°C storage. Uncorrected for recovery. Method 135. 0.01 mg/kg reported LOD. Recovery ≥88% at 0.1 mg/kg except omental fat 73%.
Egypt Kholif *et al*., 1994	EC 600 mg/l (GAP)	1? (Report not specific, but 1 application presumed) 2l/cow	Milk Hours 2 4 6 8 16 24 36 48	Cows / Buffaloes 0.05 / 0.11 0.2 / 0.2 0.2 / 0.3 0.1 / 0.2 0.05 / 0.02 0.03 / 0.01 ND[1] / 0.005 ND / ND GAP milk withdrawal interval is 3 days [1]Not detected. Method AOAC 1990, GLC/ECD. Total of 20 cows and buffaloes.
Australia, Rose, 1995; Queensland and New South Wales, 1996	EC 800 mg/l nominal 553 mg/l actual (800 is GAP)	1 Back spray 0.5 l/cow (GAP)	Loin Fat Renal fat	Days: 2 / 4 / 7 / 10 / 14 / 16 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 0.08 <0.05 <0.05

Country, ref.	Formulation. Application rate, ai	No. of sprays	Sample	Residues, mg/kg, at withdrawal interval
				Controls all <0.05 mg/kg 3 cows sampled at 2, 14, and 16 days; 6 cows at 4, 7 and 10 days. All samples except one 4-day loin fat contained <0.05 mg/kg. AOAC method, reported 0.02 mg/kg "limit of detection"

The 13 spray trials on cattle were from 1962 through 1996. One was not reviewed as it was available only in Russian (Leschchev *et al*., 1972). Hastie (1963a) reviewed three of the trials (Hastie and Cavey, 1962a,b, 1963). The first trial (Hastie and Cavey, 1962a) was not according to GAP. The second (Hastie and Cavey, 1962b), which might be interpreted to represent GAP, showed maximum residues of 1 mg/kg and 0.7 mg/kg in kidney fat and subcutaneous fat respectively. Hastie and Cavey (1963) reported residues of <0.01 mg/kg in milk from a GAP application rate after 3 days, the withholding period for milk in several countries. All of the trials were with single sprays whereas GAP allows multiple sprays in most countries.

Another old publication (Mathysse and Lisk, 1963) reports residues in milk up to 0.04 mg/kg with a mean of 0.03 mg/kg 3 days (the Australian withdrawal period) after a spray at 600 mg/l, slightly above the GAP rate for high-volume application, and up to 0.1 mg/kg with a mean of 0.04 mg/kg 4 days after a second spraying. Analysis was based on spectrophotometry.

Another dated publication (Claborn *et al.*, 1963) reported the residues of diazinon in omental fat resulting from 16 spray applications at weekly intervals. Residues 6 days after treatment at half the maximum GAP application rate increased with repeated applications from 0.06 mg/kg after the first application to 0.4 mg/kg after the 6th: they had decreased to <0.08 mg/kg 14 days after the last application. Residues 6 or 7 days after each application at the maximum GAP rate remained between 0.5 and 0.8 mg/kg. They had decreased to <0.05 mg/kg by 14 days after the 16th treatment. Analyses were again by spectrophotometry.

In a UK study Chilwell *et al*. (1967) reported diazinon residues in milk within 24 hours of spray treatments at 200 mg ai/l. The mean residues were 0.02 mg/kg after 2 hours, rose to 0.08 mg/kg after 9 hours and decreased to 0.02 mg/kg after 24 hours. No information on UK GAP for cattle sprays was provided, although the application rate is below most reported GAP rates.

In an old but reasonably well-described study in Switzerland (Blass, 1971) residues of diazinon in milk were determined at various intervals after applying 4 sprays 500 or 1000 mg/l at weekly intervals. The lower rate complies with GAP. After 4 days (the GAP interval for Switzerland) the residues from the lower rate did not exceed a maximum of 0.03 mg/kg or a mean of 0.02 mg/kg after the third spray (the maximum number allowed by Swiss GAP) nor above 0.04 mg/kg maximum (0.03 mg/kg mean) after 3 days, which is the withdrawal interval in other countries. Analyses (presumably colorimetric) were by an autoanalyser.

In another old, but fairly well-described, Australian study residues of diazinon in the milk of cattle were determined after various intervals (Bull and Dougall, 1974). Samples from five individual cows and bulk samples from the whole herd were analysed after one treatment at 500 mg ai/l, the GAP

rate. The cows were milked twice daily. The mean residues from the 5 cows were 0.2 mg/kg (0.3 mg/kg maximum) at the first milking, 0.06 mg/kg at the second, 0.03 at the 4th (day 2) and 0.02 mg/kg at the 10th (the 5th day after treatment). There is no specified Australian withdrawal period for milk but 3 or 4 days is common in other countries with similar GAP. Residues from the first milking were 0.03 mg/kg in skim milk and 5.2 mg/kg in butter, the residue in butter decreasing to 0.05 mg/kg after the 10th milking. Residue levels in the bulk herd samples were similar to the means of the 5 cows.

Two relatively recent studies from Australia were not especially well reported by current standards (e.g. they lacked details of the intervals from sampling to analysis and analytical confirmation of the active ingredient content of the sprayed solutions), but sample storage was at -15°C. In one of these studies diazinon residues were measured in the milk of 5 cows 7 to 80 hours after a single EC spray at 600 mg/l, 1.2 times the high-volume GAP rate (Bull, *et al*., 1986). The residues ranged from a mean of 0.2 mg/kg and a maximum of 0.4 mg/kg 7 hours after application to a mean of ≤0.01 mg/kg after about 3 days (70 hours).

In the other fairly recent Australian study (Strong *et al*., 1986b) a similar application was made to calves, from which tissues were analysed for diazinon. The residues were up to 2.9 mg/kg in fat, about 0.06 mg/kg in muscle and kidney and up to 0.02 mg/kg in liver 1 day after treatment. No results were available for the Australian withdrawal interval of three days, but the residues in muscle, liver and kidneys had decreased to ≤0.01 mg/kg after 7 days, when kidney fat contained up to 0.7 mg/kg. Residues up to 0.2 mg/kg were found in omental fat after 14 days.

In a more recent Egyptian study (Kholif *et al*., 1994) a total of 20 Friesian cows and buffaloes were spray-treated once after the morning milking with an EC formulation at 600 mg/l (the Egyptian GAP rate). The animals were milked twice daily by machine. The residues in the milk of the buffaloes and cows respectively decreased from a maximum of 0.3 and 0.2 mg/kg after 6 hours to 0.005 mg/kg and not detected after 36 hours. The Egyptian withdrawal interval for milk is three days. The trial was generally well described but some desirable details were not included (e.g. sample handling and storage conditions and period of storage.

In a recent Australian trial cattle were back-sprayed once with 0.5l at 553 mg diazinon/l: the nominal rate was 800 mg/l which is the maximum GAP concentration. Loin and renal fat were analysed for residues at intervals from 2 to 16 days after treatment (Rose, 1995; Queensland and New South Wales, 1996). Samples were taken from frozen export packs from the treated cattle. One loin fat sample of six taken four days after treatment contained 0.08 mg/kg; all other residues were below 0.05 mg/kg. Spray solutions were analysed for the active ingredient, but other important information such as sample handling and storage conditions and intervals from sampling to analysis was not reported.

Sheep, goats and pigs

Table 12. Residues of diazinon in the milk, fat or tissues of sheep, goats and pigs following spraying.

Country, ref.	Formulation. Application rate, ai	No. of sprays	Sample	Residues, mg/kg, at withdrawal interval
SHEEP				
Australia	EC	1		14 days (3 sheep)

Country, ref.	Formulation. Application rate, ai	No. of sprays	Sample	Residues, mg/kg, at withdrawal interval
Bull, 1971	800 mg/l	jetting	Muscle Fat	Sheep A B C Mean 0.05 0.05 0.05 0.05 0.15 0.08 0.06 0.13
	WP 800 mg/l	1 jetting	Muscle Fat	Sheep D E F Mean 0.03 0.06 0.09 0.06 Control 0.06 0.16 0.06 0.05 0.09 Control 0.03 Method 113 (sweep-codistillation; GLC thermionic). 0.02 mg/kg limit of detection reported. Recoveries 70% muscle, 92% fat at 0.2 and 0.5 mg/kg respectively. Results corrected.
Switzerland Morrison, 1994	EC 250 600 mg/l (GAP)	1 6 l/sheep (GAP = 3l)	fat (tail base biopsy)	Sheep no. 8 28 days 1 1.8 0.10 2 1.6 0.11 3 3.1 0.13 4 3.3 0.17 5 2.6 0.29 6 3.5 0.11

Country, ref.	Formulation. Application rate, ai	No. of sprays	Sample	Residues, mg/kg, at withdrawal interval
				Mean 2.7±0.8 0.15±0.07
	EC A-139 F 600 mg/l (GAP)	1 6 l/sheep	same	1 2.2 0.14 2 2.4 0.17 3 2.1 0.24 4 0.78 0.15 5 2.7 0.08 6 3.2 0.16 Mean 2.2± 0.16±0.05
	EC 600 600 mg/l (GAP)	1 6 l/sheep	same	Sheep no. 8 28 days 1 2.1 0.09 2 2.1 0.06 3 2.1 0.11 4 2.0 0.22 5 1.4 0.10 6 1.9 0.14 Mean 1.9± 0.12±0.12 Method 24480.O. Mean recoveries from fat 95-100% on different days at 0.025 mg/kg. Reported LOD 0.005 mg/kg; chromatograms suggest 0.01 mg/kg more practical.
GOATS				

Country, ref.	Formulation. Application rate, ai	No. of sprays	Sample	Residues, mg/kg, at withdrawal interval
Australia Bull *et al*., 1986b	EC 600 mg/l (GAP is 500 mg/l in Australia, 600 mg/l in other countries)	1 5 l/goat (GAP is 3 l)	Muscle Mean	1 3 7 14 21 days 0.14 0.03 0.01 <0.01 <0.01 0.06 0.04 0.02 <0.01 <0.01 0.1 0.04 0.02 <0.01 <0.01 Controls in all tissues <0.01 mg/kg GAP pre-slaughter interval is 14 days. Two goats sampled at each time.
			Liver Mean	0.04 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.03 <0.01 <0.01 <0.01 <0.01
			Kidney Mean	0.08 <0.01 <0.01 <0.01 <0.01 0.02 0.03 0.01 <0.01 <0.01 0.05 0.03 0.01 <0.01 <0.01
			Kidney fat Mean	3.4 1.0 0.04 <0.01 <0.01 1.1 1.4 0.22 0.02 <0.01 2.3 1.2 0.13 <0.02 <0.01
			Omental fat Mean	3.8 0.39 0.08 0.03 <0.01 0.91 1.2 0.2 0.01 <0.01 2.4 0.8 0.14 0.02 <0.01
Australia Strong *et al*., 1987	EC 600 mg/l	1 5 l/goat	Whole milk (5 goats)	Goat A B C

Country, ref.	Formulation. Application rate, ai	No. of sprays	Sample	Residues, mg/kg, at withdrawal interval		
						D
						E
						mean
			Pre-treatment	0.01	0.01	<0.01
			Hours			0.01
						0.01
						<0.01
			7			
				0.18	0.25	0.22
						0.18
						0.25
						0.22
				0.07	0.08	0.09
			24			0.06
						0.03
						0.07
				0.08	0.08	0.10
			30			0.08
						0.07
						0.08
				0.03	0.03	0.04
			48			0.02
						0.02
						0.03
				0.04	0.03	0.05
			54			0.03

Country, ref.	Formulation. Application rate, ai	No. of sprays	Sample	Residues, mg/kg, at withdrawal interval
				0.03
				0.04
				0.01 0.01
				0.02
			72	0.01
				0.01
				0.01
				0.02 0.02
				0.02
			78	0.01
				0.01
				0.02
				Method 132A. 0.01 mg/kg limit of "detection" reported. Recovery 81% at 0.1 mg/l.

PIGS						
Switzerland Formica, 1974b	EC 250 mg/l (GAP)	1 5 l/pig (GAP is 1 l)		1		3
			Muscle			—
			Liver			—
			Kidney			—
			Fat			7
			Skin			—
						—
						—
						14
						—
						—
						—
						28
						—
				0.04	0.02	
						0.02
						0.01
						0.01
				All <0.01		
				All <0.01		
				0.22	0.05	
						0.02
						<0.01
						<0.01
				All <0.01		
		2 (GAP is ≤3) 5 l/pig	Muscle	0.02	0.01	<0.01
			Liver			
			Kidney			
			Fat			
			Skin			<0.01
						<0.01
				All <0.01		
				All <0.01		
				0.15	0.06	<0.01
						<0.01
						<0.01
				0.05	0.02	<0.01
						<0.01
						<0.01

EC 500 mg/l	1 5 l/pig	Muscle Liver Kidney Fat Skin	0.08	0.04		0.01
						0.02
						0.04
			All <0.01			
			0.01	<0.01	<0.01	
						<0.01
						<0.01
			0.5		0.15	
						0.02
						<0.01
						<0.01
			0.13	0.02		
						<0.01
						<0.01
						<0.01
	2 5 l/pig	Muscle Liver Kidney Fat Skin	0.04	0.01		
						<0.01
						<0.01
						<0.01
			All <0.01			
			All <0.01			
			0.21	0.05		
						0.01
						<0.01
						<0.01
			0.01	0.01		<0.01
						<0.01
						<0.01
			Controls <0.01 mg/kg for all samples and times.			

				Method REM 4/74. Recoveries 125, 75 and 90% at 0.1 mg/kg from muscle, liver and fat respectively. 0.01 mg/kg limit of "detection" reported. Results not corrected for recovery
				<u>hydroxydiazinon</u>
			Fat	<0.02 mg/kg in all but two samples (0.02 and 0.03 mg/kg) at 250 and 500 mg/l respectively).
			Other tissues	<0.02 mg/kg in all samples.
				<u>pyrimidinol metabolite G 27 550</u>
			Kidney	<0.1 mg/kg in all samples at 250 mg/l. <0.1, 0.12, 0.12, 0.14, 0.14, 0.16 at 500 mg/l.
			Other tissues	<0.1 mg/kg in all samples.
				<u>diazoxon</u>
			All tissues	<0.01 mg/kg in all samples

Sheep. Two studies were provided. In an Australian study sheep were treated once with a single jet spray with either a WP or an EC formulation, at 800 mg/l (twice the reported GAP concentration for Australia). Muscle and fat samples were analysed after slaughter 14 days later, the GAP interval (Bull, 1971). Although fat was identified as omental, subcutaneous and kidney fat, the data did not specify which residues were in which fat. The residues did not exceed 0.16 mg/kg in fat or 0.09 mg/kg in muscle. Details such as sample handling and storage conditions, intervals from sampling to analysis and confirmation of spray concentrations were not recorded, nor were sample chromatograms supplied.

A more recent and well documented Swiss study (Morrison, 1994) was reported to be in accordance with OECD GLP. Three groups of six sheep were sprayed with different EC formulations, all nominally at the Swiss GAP rate of 600 mg/l, actually 576-592 mg/l, and 6l/animal (twice the GAP volume). Fat samples were taken by biopsy from the base of the tail 8 and 28 days later: the GAP slaughter interval is 21 days in Switzerland, 14 days in some other countries. Samples were stored at -20°C until analysis less than 6 months later. Blood samples were also taken.

Fat samples were extracted with acetonitrile, cleaned up on a solid-phase column and analysed by GLC with an NP detector. Recoveries were near 100% at 0.025 mg/kg. The reported limit of determination was 0.005 mg/kg, but 0.01 mg/kg appears to be more practical from the sample chromatograms.

Eight days after treatment diazinon residues in the fat ranged from 0.8 to 3.5 mg/kg with an overall mean of 2.3 mg/kg after 8 days and 0.06 to 0.3 mg/kg with a mean of 0.14 mg/kg after 28 days, with no significant difference between the three formulations.

Goats. Two Australian trials were reported. In one, each goat was treated once with 5l of an EC at 600 mg ai/l and 5l animal and the goats were slaughtered in pairs 1, 3, 7, 14 and 21 days after treatment (Bull *et al.*, 1986b). GAP in Australia requires 500 mg/l and in other countries up to 600 mg/l. The higher residues of each pair one day after treatment were 0.14 mg/kg in muscle, 0.04 mg/kg in liver, 0.08 mg/kg in kidney, 3.4 mg/kg in kidney fat and 3.8 mg/kg in omental fat. After the Australian GAP slaughter interval of 14 days the only residues above 0.01 mg/kg were 0.02 mg/kg in one sample of kidney fat and 0.03 mg/kg in one of omental fat.

Samples were stored at -15°C until analysis by method 135 for which a limit of determination of 0.01 mg/kg was reported, although no sample chromatograms were presented. Recoveries were ≥87% at the lowest fortification level of 0.1 mg/kg. The interval from sampling to analysis was not reported, nor was confirmation of the actual spray concentration.

In the second study 5 goats were each sprayed once with 5l of EC at 600 mg/l (Strong *et al.*, 1987). Diazinon residues were determined in the whole milk after 7 consecutive milkings by method 132A. Reported recoveries were 81% at 0.1 mg/kg but no sample chromatograms were presented. Samples were stored at -15°C until analysis, but the storage period was not reported nor was confirmation of the spray concentration. Residues decreased from a maximum of 0.25 and a mean of 0.22 mg/kg after 7 hours to 0.01-0.02 mg/kg after 72 and 78 hours. Withholding intervals for milk are 3 to 4 days in other countries.

Pigs. One old but relatively well documented Swiss study (Formica, 1974b) was reported. Pigs were each sprayed once or twice with 5l EC spray (1l is GAP) at either 250 or 500 mg ai/l, the GAP concentration in Switzerland and Egypt respectively. Samples of muscle, liver, kidney, skin and fat taken 1, 3, 7, 14 and 28 days after treatment were analysed for residues of diazinon, diazoxon, hydroxydiazinon and the pyrimidinol metabolite G27550. The Swiss GAP pre-slaughter interval is 21 days.

Although spray concentrations were not confirmed nor chromatograms provided, samples were stored at -20°C until analysis within 7 months of treatment. Residues of diazinon after 14 and 28 days were all ≤0.01 mg/kg except in two muscle samples with 0.02 and 0.04 mg/kg. Residues of hydroxydiazinon were <0.02 mg/kg in all samples except two of fat which contained 0.02 and 0.03 mg/kg. Residues of G 27550 were <0.1 mg/kg except in the kidneys of pigs treated at 500 mg ai/l, where they ranged from <0.1 to 0.16 mg/kg. No residues of diazoxon (<0.01 mg/kg) were detected in any sample.

Summary

For the convenience of a general picture, the residues resulting from treatments according to GAP shown in Tables 8 to 12 above are summarized in Table 13.

Table 13. Summary of diazinon residues found in milk and tissues of cattle, sheep, goats and pigs from treatments against ectoparasites according to GAP.

Sample Type of treatment	Diazinon, mg/kg, maximum and (median) residues in				Maximum residue, mg/kg, from any treatment
	Cattle	Sheep	Goats	Pigs	
Milk Ear tag	0.01 (0.01 mean) 0.3(0.2)fat				Milk 0.02 (mean)[1]
Dip		0.02(0.02)			
Spray	0.05 (0.02 mean)		0.02		
Muscle Ear tag	0.02 (tongue)				Muscle 0.03
Dip		0.03(0.02)			
Spray	0.03[2]		<0.01(<0.01)	0.01	
Liver Ear tag	<0.01				Liver 0.03[2]
Dip		0.01(<0.01)			
Spray	0.03[2]		<0.01(<0.01)	<0.01	

Kidney Ear tag	<0.01				Kidney 0.03[2]
Dip		0.02(0.02)			
Spray	0.03[2]		<0.01(<0.01)	<0.01	
Omental fat Dip		1.3(1.1)			Omental fat 1.3
Spray	0.2(0.2)		0.03(<0.02)		
Renal fat Backrub	0.3(0.2)				Renal fat 0.7
Ear tag	0.04				
Dip		0.7(0.7)			
Spray	0.7(0.6) (low side)		0.02(<0.02)		
Loin (= subcut.?) fat Backrub	0.7(0.2)				Loin fat (subcut.?) 0.7
Spray	0.08(0.05)				
Subcut.fat Ear tag	0.05 "back fat"				Subcutaneous fat 4.3
Dip		4.3(1.3)			
Spray		0.3(0.14) (tail base fat)		<0.01 ("fat")	
Max. mg/kg/ matrix/animal					
milk	0.01	0.02	0.02	--	
muscle	0.03	0.03	<0.01	0.01	
liver	0.03	0.01	<0.01	<0.01	
kidney	0.03	0.02	<0.01	<0.01	
fat -->	0.7	4.3	0.03	<0.01	

[1] Because milk is normally pooled, the highest mean, not the highest individual value, is recorded. This was 0.02 mg/kg from both spray and dip treatments.

[2] Estimated. There were no results at the GAP 3-day pre-slaughter interval, so an estimate was made on the basis of the residues found at 1 and 7 days.

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

Information on diazinon residues in commerce or at consumption from The Netherlands for the periods 1991-1993 and for 1994 is shown in Table 14.

Table. 14. Residues of diazinon in foods in commerce in The Netherlands 1991-1993 and 1994 (Netherlands, 1995).

Commodity	No. of samples	No. of samples without residues (<0.02 mg/kg)	No. of samples with residues ≥0.02 mg/kg)[1]
Citrus fruit			
1991-93			
tangerines	523	519	4
oranges	958	948	10
1994			
grapefruit	111	110	1
lemons	102	101	1
tangerines	215	214	1
oranges	348	335	13
Pome fruit			
1991-93			
apples	1698	1692	6
pears	501	498	3
1994			
apples	712	709	3
pears	162	161	1
Stone fruit			
1991-93			
plums	536	534	2
1994			
cherries	68	66	2
nectarines	103	102	1
Berries and small fruit			
1991-93			
grapes	999	995	4
strawberries	2976	2974	2
1994			
grapes	336	335	1
strawberries	913	912	1
other small fruit	103	101	2
Misc. fruit			
1991-93			
kiwifruit	309	294	15
pineapple	93	88	4
1994			
kiwifruit	96	91	5
Root and tuber veg.			
1991-93		561	
carrots	609	762	48
radishes	764		2
1994			
carrots	141	130	11 (mean 0.02 mg/kg)
Bulb veg.			

Commodity	No. of samples	No. of samples without residues (<0.02 mg/kg)	No. of samples with residues ≥0.02 mg/kg)[1]
1991-93			
bulb onions	106	104	2
Fruiting veg.			
1991-93	1129	1125	4
sweet peppers	344	339	5
melons			
1994			
peppers	571	568	3
cucumbers	341	340	1
melons	164	158	6
Brassica veg.			
1991-93			
broccoli	214	208	6
Brussels sprouts	132	128	4
Chinese cabbage	348	345	3
Leafy veg. & fresh herbs			
1991-93			
lettuce	2634	2621	13
endive	1724	1718	6
chives	33	31	32
parsley	563	560	3
1994			
lettuce	1277	1274	3
endive	511	508	3
parsley	158	154	4
Stem veg.			
1991-93			
celery	807	799	8 3 samples ≥0.5 mg/kg (mean 0.04 mg/kg)
1994			
celery	202	201	1
Pulses			
1991-93			
beans	855	853	2

[1] All residues were below 0.5 mg/kg except in the 3 noted samples of celery.

Monitoring data for 1994 were reported from Poland. No diazinon residues were found in 73 samples of apples, 17 of beetroot, 101 of carrots, 30 of cauliflowers, 23 of celery, 62 of greenhouse cucumbers, 20 of field cucumbers, 78 of black currants, 12 of leeks, 47 of bulb onions, 30 of parsley, 10 of sweet peppers (greenhouse), 10 of radishes, 29 of raspberries, 126 of strawberries, and 224 of tomatoes (greenhouse). Residues were found in 20 samples, distributed as shown below.

Commodity	No. of samples	No. positive	Residues, mg/kg	Mean, mg/kg
White cabbage	123	6	0.01-0.2	0.07
Red, white currants	20	13	0.03-0.5	0.15
Lettuce (greenhouse)	9	1	0.5	

NATIONAL MAXIMUM RESIDUE LIMITS

National maximum residue limits reported to the Meeting are listed below. In many cases national authorities have adopted Codex MRLs for animal commodities but several members of the European Union (underlined) will reportedly change to European Union MRLs in 1997.

Commodity	Country	MRL, mg/kg
Products of plant origin		
Cereals	Australia	0.1
	Netherlands, Poland	0.05
Citrus fruits	Australia	0.7
Fruits (except citrus, olives, peach)	Australia	0.5
Kiwifruit	Australia	0.5
Nuts	Australia (tree nuts)	0.1
	Netherlands	0.1
Oilseed	Netherlands	0.1
Olive oil, crude	Australia	2
Olives, unprocessed	Australia	2
"Other foods"	Netherlands	0[1](0.02)
"Other fruits"	Netherlands	0.5
Peach	Australia	0.7
Potato	Netherlands	0.02[1]
Sugar cane	Australia	0.5
Sweet corn (corn-on-the-cob)	Australia	0.7
	Netherlands	0.7
Vegetable oils (except olive oil, crude)	Australia	0.1
Vegetables	Australia	0.7
Products of animal origin		
Edible offal (mammalian)	Australia	0.7
Eggs	Australia	0.05*
Milk	Australia	0.5 (in the fat)
	Canada	0.1 (ear tag uses)

Commodity	Country	MRL, mg/kg
	Austria, Chile, China, Columbia, Egypt, Finland, France, Iraq, Jordan, Kenya, Kuwait, Lebanon, Libya, Morocco, Netherlands, Oman, Poland, Portugal, Saudi Arabia, Spain, Switzerland, Syria, Thailand, Turkey, UK, United Arab Emirates, Vietnam.	0.02
Meat (mammalian)	Canada	0.1 (ear tag uses)
	Switzerland	0.2
	Argentina, Australia, Austria, Chile, China, Columbia, Egypt, Finland, France, Iraq, Ireland, Jordan, Kenya, Kuwait, Lebanon, Libya, Morocco, Netherlands, New Zealand, Oman, Poland, Portugal, Saudi Arabia, Spain, Switzerland, Syria, Thailand, Turkey, UK, United Arab Emirates, USA, Vietnam.	0.7 (fat basis)
Poultry, edible offal of	Australia	0.05*
Poultry meat	Australia	0.05*

[1] Limit of detection
* At or about the limit of determination

APPRAISAL

Diazinon was first evaluated by the 1965 JMPR and has been reviewed several times since. In 1993 a periodic review was conducted and in 1994 a new MRL was recommended for hops. The 1993 JMPR recommended, among other items, an increase in the CXL for pome fruits from 0.5 to 2 mg/kg and the withdrawal of the CXLs for animal commodities in the absence of animal transfer studies and data from uses as an ectoparasiticide.

The CCPR in 1995 and 1996 endorsed most of the recommendations of the 1993 JMPR with the exception of the proposed MRL for pome fruits and the recommended withdrawal of the CXLs for milks and the meat of cattle, pigs and sheep.

Several countries were concerned that the proposed MRL for pome fruit, and to some extent the proposed limits for tomatoes and cabbages, might imply a high dietary intake. Insufficient new data were provided to review the recommendation, which was based on trials in the USA. It was the understanding of the Meeting that US GAP for pome fruits has been revised since 1993, and that additional trials which had been carried out according to the new GAP might support a lower limit. It is desirable that details of these trials be provided to a future JMPR. In order to provide a better estimate of dietary intake, the Meeting estimated STMR levels for pome fruit, tomatoes and cabbages from the data in the 1993 JMPR monograph on trials which complied with GAP at that time. These levels were 0.12, 0.12 and 0.16 mg/kg respectively. The respective proposed MRLs are 2, 0.5 and 2 mg/kg.

The main focus of the present evaluation was the review of submissions in support of MRLs for animal products. The 1993 JMPR considered animal transfer studies and information on veterinary uses to be desirable, and the 1995 CCPR retained the CXLs for animal products pending review by the JMPR of new data on animal feeding trials to be submitted by Australia and the manufacturer. The

Meeting reviewed new submissions on animal transfer studies and supervised trials involving approved uses for ectoparasite control together with reports of additional analytical methods (mainly for animal products) and animal metabolism studies, some of which were provided to the 1993 JMPR, but not to the FAO Panel.

Some studies of animal metabolism were resubmitted at the request of the Meeting, in particular on poultry metabolism which had not been reviewed in 1993. These confirmed that metabolism in hens is essentially similar to that in mammals and that diazinon, diazoxon and hydroxydiazinon generally constitute a small proportion of the total radioactive residue, which consists largely of the pyrimidinol metabolite and hydroxy derivatives of it, together with glucuronide and other conjugates.

The Meeting reviewed nine analytical methods submitted for the first time in addition to re-submissions of methods reviewed in 1993. The new submissions are mainly methods for animal products based on GLC with phosphorus- or NP-selective detectors. Most of them determine only diazinon *per se*, with limits of determination ranging from 0.01 to 0.05 mg/kg. Two methods determine hydroxydiazinon and diazoxon in addition to diazinon, although in most trials only residues of diazinon were reported.

A feeding trial on dairy cows at levels equivalent to 40, 120 and 400 ppm of diazinon in the diet was reported. The manufacturer considered 40 ppm to be a reasonable estimate of intake but the dietary burden based on Codex MRLs for those commodities with the greatest potential for residues in cattle feed, maize forage, sugar beet tops and apple pomace, is likely to be lower. If the maximum percentage of the feed dry weight for dairy and beef cattle is assumed to be 80 and 40% maize forage, 10 and 20% sugar beet tops, and 20 and 40% apple pomace, it can be estimated that the dry weight dietary burden would not exceed about 25 ppm for dairy or 15 ppm for beef cattle. This is based on adjusting the CXLs to a dry weight basis, assuming that they are not already so expressed. Since some countries include other feed items which might contain more diazinon, 40 ppm is a reasonable worst-case assumption, if not needed for estimates based on Codex MRLs.

The study at the 40 ppm level indicated that no residues (<0.01 mg/kg) of diazinon, diazoxon or hydroxydiazinon would be expected in the liver, kidney, muscle, or milk of cattle, but residues of diazinon up to 0.04 mg/kg occurred in omental fat. Intakes calculated from Codex MRLs would be expected to produce residues of ≤0.02 mg/kg in the fat of beef cattle. Diazinon residues in the fat were roughly proportional to the intake of diazinon. Diazinon was detectable in milk only at the tenfold dosing level: the pattern of residues in the milk of individual cows dosed at this rate suggests that residues of diazinon may peak after about 3-7 days and then decrease.

Information on the freezer storage stability of diazinon in milk and of diazoxon and hydroxydiazinon in meat and milk is desirable to confirm the above conclusions.

In a feeding study on poultry no residues (<0.01 mg/kg) of diazinon, diazoxon or hydroxydiazinon were found in any sample from hens fed for 28 consecutive days at rates equivalent to 0.5, 1.5 or 5 ppm in the diet. The basis for considering 0.5 ppm as a likely level in poultry feed was not explained. The only commodity with a Codex MRL which is a significant poultry feed item is maize, which might be up to 80% of a poultry diet. However the CXL for maize is at the limit of determination of 0.02 mg/kg, so a feeding level of 0.5 ppm is more than adequate. A maximum residue level of 0.02 mg/kg (limit of determination) could be supported for poultry meat, fat and eggs.

Eight trials of uses against ectoparasites were reviewed by the JMPR from 1967 to 1975 and 3 ear tag trials were reviewed in 1993. All of these were re-submitted for review by the present Meeting, together

with a number of studies not previously reviewed: 10 spray trials (6 on cows, 2 on goats, 1 on sheep, 1 on pigs), 4 ear tag trials on cattle, 3 sheep dip trials, 1 wound-dressing study on sheep and 1 backrub study on cattle.

Dust applications. Data from one very old study could not be related to GAP. The residues in milk increased from 0.02 mg/kg after two hours to 0.1 mg/kg after 9 hours.
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Wound dressings.In a 1974 Australian study residues of diazinon were determined in the fat, muscle and liver of fly-struck sheep (5 animals/treatment rate) 10 days after treatment with a 2% ai powder formulation applied as a wound dressing at 10 and 30 g/animal. It could not be confirmed with the label provided that 10 g/animal is the GAP rate. The maximum residues from the 10 g treatment after ten days compared to the 14-day Australian GAP slaughter interval were 0.01 mg/kg in liver, 0.03 mg/kg in muscle and 0.08 mg/kg in omental fat. The corresponding median residue levels were <0.01, 0.01 and 0.06 mg/kg.

Backrubber trials. The Meeting could not evaluate the results of a 1967 trial in the USA with both handrubbed and burlap bag applications for which no relevant GAP was available.

In a recent Australian trial on cattle in accordance with Australian GAP the maximum, median and mean diazinon residues were 0.66, 0.24 and 0.29 mg/kg in loin fat and 0.26, 0.16 and 0.16 mg/kg in renal fat 5 days after 19 days of exposure to a typical backrubber application. The GAP withdrawal period is 3 days and the recommended export slaughter interval 10 days. Residues had generally decreased substantially by 7 or more days after exposure ceased. The maximum, median and mean residues of diazinon were 0.3, 0.04 and 0.1 mg/kg in loin fat one day after 10 days of exposure. Residues in renal fat were on average about half those in loin fat. The residues in loin fat were higher after prolonged exposure. The Meeting concluded that residues would not be expected to exceed 0.7 mg/kg in the fat of cattle from applications of EC formulations in backrubbers according to Australian GAP if exposure does not exceed 19 days and a withdrawal interval of 5 days is observed, or 0.1 mg/kg after the 10-day export slaughter interval. The Meeting had no information about exposures greater than 19 days.

Ear tags. Three Canadian and 4 Australian trials were reported. The Canadian reports were rather abbreviated summaries: although most of the essential information was provided, analytical methods were only referenced and details were meagre on intervals from sampling to analysis, sample storage and handling conditions, and confirmation by analysis of the claimed levels of diazinon in the tags. No reference, or confirmation of adherence, to GLP was provided. The Canadian trials are probably according to GAP. The Canadian withdrawal interval is reported to be "Nil" and the label simply refers to removal before slaughter. The Meeting interpreted this to imply essentially a 0-day withholding period.

In the 1987 Canadian trials residues in whole milk were <0.5 ì g/kg 1 day after tag application and reached a maximum, median and mean of 1.7, 1.4 and 1.4 ì g/kg after 7 days. Residues in back fat and kidney fat were respectively 0.03 and 0.035 mg/kg after 14 days. No residues (<0.01 mg/kg) were reported in muscle, liver or tongue. Similar results were reported in the 1989 study, with a maximum of 0.05 mg/kg in back fat. The Meeting concluded that the Canadian trials were not reported in sufficient detail to draw firm conclusions.

Details were also lacking from the first two Australian trials (1989, 1990), although the storage conditions were indicated for the first and sample chromatograms were provided for the second. The

1989 study appears to reflect Australian GAP, but in 1990 only one ear of each animal was tagged whereas two tags conform to GAP. The maximum, median and mean residues in milk fat in 1989 were 0.02, 0.01 and 0.0125 mg/kg after 28 days, and were not significantly different from those after 1 day. The maximum residue would be equivalent to <0.01 mg/kg in whole milk assuming 4% fat. The Meeting placed less reliance on these trials than the better documented 1992/93 Australian trials.

The 1992 and 1993 Australian trials were similar in principle and appear to reflect Australian GAP. Both were reported to comply with OECD GLP. In the 1992 trial diazinon residues in the milk fat peaked after 7 days with maximum, median and mean residues of 0.26, 0.2 and 0.19 mg/kg, corresponding to a maximum of 0.01 mg/kg in whole milk assuming 4% fat. In the 1993 trial residues increased from a maximum, median and mean of 0.02, <0.01 and <0.0125 mg/kg milk fat after 1 day to 0.02, 0.01 and 0.02 mg/kg after 7 days and 0.03, 0.02 and 0.02 mg/kg after 42 days, then decreased to ≤0.01 mg/kg after 84 days of continuous exposure. Maximum and mean residues in whole milk would be ≤0.01 mg/kg assuming 4% milk fat.

Although the Meeting was more confident of the documentation for the 1992/93 Australian trials, there is an apparent inconsistency between the higher residue levels (up to 0.3 mg/kg) found in milk fat in 1992 compared with ≤0.03 mg/kg in 1993 and ≤0.02 mg/kg in the 1989 Australian trials. Before completion of the 1993 study this was attributed to the use of 15 g tags in 1992 compared with 10 g in 1989, but the 1993 tags were also 15 g and the residues were comparable to those in 1989.

After the 1993 study the authors speculated that the inconsistencies were due to differences in the extent of self-grooming by the cattle. This varies according to buffalo fly pressure, which was not recorded. The only other obvious differences between the trials were that in 1992 the cream was stored in a refrigerator for 5 days before the preparation of butter whereas preparation was immediate in 1993, and the ear tags were from different manufacturers. Unfortunately no reference was made to analysing the tags to confirm the diazinon content before the study.

Overall the Meeting gave greater weight to the more recent, better described Australian studies and concluded that diazinon residues from the use of ear tags on cattle according to GAP should not exceed 0.2 mg/kg (mean) in butter, 0.05 mg/kg in back fat or renal fat, 0.01 mg/kg (mean) in milk, or the limits of determination of 0.01 mg/kg in kidney, liver and muscle and 0.02 mg/kg in tongue.

Dipping. Of 8 trials, 3 on cattle and 1 on sheep were very old and insufficiently documented by current standards to provide a basis for estimating maximum residue levels. Only the sheep trial of these four (in 1962) appears to comply approximately with current GAP: it showed maximum and mean residues of 1.4 and 0.8 mg/kg in sheep kidney fat. Even in that study the sheep were dipped for 20 seconds, considered to be twice the recommended time. The Meeting placed little emphasis on these four studies. Since the other three very old trials were the only ones with cattle there are no useful cattle trials and any conclusions on likely residues in dipped cattle must be largely based on sheep dipping, which would normally produce higher residues.

The sheep trials in 1973 and 1974 were fairly well documented. In 1973 the residues in milk from treatments according to GAP were 0.01 and 0.02 mg/kg after the 4-day GAP milking interval. In 1974 the maximum residues were 0.4 mg/kg in muscle, <0.02 mg/kg in liver and 2.6 mg/kg in omental fat, but the dip concentration was 3 times the GAP level. In a reasonably well described Australian trial in 1986 sheep were slaughtered in pairs at intervals after a single dip. The maximum and mean residues after the 14-day GAP pre-slaughter interval were 0.03 and 0.02 mg/kg in muscle, 0.01 and <0.01 mg/kg in liver, 0.02 and 0.02 mg/kg in kidney, and 0.67 and 0.65 mg/kg in kidney fat. The mean residues were also the medians.

The best documented trial was in 1989 in the UK. The dip concentration was 1.6 times the GAP concentration in the UK, but complied with Irish and New Zealand GAP. Pre-slaughter intervals are 14 days in the UK and Ireland, and 21 days in New Zealand. The maximum, median and mean residues after 14 days were 1.3, 1.1 and 1.1 mg/kg in omental fat and 4.3, 1.3 and 2.3 mg/kg in subcutaneous fat, in which the three individual residues were 4.3, 1.4 and 1.3 mg/kg. After 21 days the maximum, median and mean residues were 1.2, 0.75 and 0.8 mg/kg in omental fat and 1.4, 1.0 and 1.2 mg/kg in subcutaneous fat, in which these were also the individual residues.

A comparison of the residues of 4.3, 1.3 and 1.4 mg/kg in subcutaneous fat at 14 days and consideration of the relation between 14- and 21-day residues in both omental and subcutaneous fat led the Meeting to conclude that 4.3 mg/kg was probably aberrant. It is not consistent with the other results, except to the extent that higher residues in subcutaneous fat than in other fats are not unexpected.

In summary, from the available data the maximum and median residues to be expected in sheep from single dipping according to GAP would be as follows.

	Residue, mg/kg	
	Maximum	Median
milk	0.02	0.02
muscle	0.03	0.02
liver	0.01	<0.01
kidney	0.02	0.02
kidney fat	0.7	0.7
omental fat	1.3	1.1
subcutaneous fat	1.4	1.4

From the available information on residues in individual sheep the Meeting concluded that a level of 2 mg/kg would be required to cover residues in sheep fat arising from single dipping according to GAP. Although this is greater than the current CXL of 0.7 mg/kg, several trials according to GAP show that residues above 0.7 mg/kg are likely to occur. A pre-slaughter interval of 35 days would appear to be required to reduce fat residues to the CXL. The Meeting noted that the most reliable sheep dipping trials which complied with GAP were with single dips, although GAP in most countries allows multiple dipping. This emphasises the need for a higher limit than 0.7 mg/kg. Additional trials meeting current standards and including multiple dips according to GAP are highly desirable, as are monitoring data on sheep fat, especially from sheep known to have received dip or spray applications at maximum GAP rates.

Cattle spraying. Thirteen trials were reported, but the Meeting did not review one from 1972 which was available only in Russian. Six of the remaining 12 were very old studies (1962-7), not meeting current reporting standards and often with outdated analytical methods. The Meeting placed little emphasis on these in estimating maximum residue levels in cattle, but considered 5 of the remaining 6 studies to be at least marginally acceptable to varying degrees. It was recognized that they were all with single applications, although GAP generally permits more than one spray. Four of them were concerned with residues in milk and two with residues in fat or tissues.

Residues in milk. A fairly well described 1971 Swiss trial was not fully acceptable to the Meeting, but

was of particular interest because it involved multiple applications in accordance with current GAP. However, because of the obsolete analytical method (autoanalyser) and inadequate reporting of certain details, some uncertainty remains on the validity of the results. The mean diazinon residues in the milk did not exceed 0.02 mg/kg (maximum 0.03 mg/kg) 4 days (the GAP withdrawal interval) after the 3rd spraying (the maximum GAP number). All residues were <0.02 mg/kg after 6 days and mean residues 0.03 mg/kg after 3 days, which is the GAP withdrawal period in other countries.

In a fairly well described Australian study in 1974 the mean diazinon residues in the milk from 5 cows from a herd treated according to GAP were 0.02 mg/kg (maximum 0.05 mg/kg) 5 days after treatment compared to 3- or 4-day GAP withdrawal intervals. This is consistent with the 1971 study. The residues were 0.05 mg/kg in a composite sample of butter and the mean residues in composited skim milk were about 1/3rd to 1/6th of the level in the whole milk, confirming the affinity of diazinon with fat. The study clearly demonstrates that the residues in milk from individual cows are significantly reduced when bulked with milk from other members of a treated herd.

In a 1986 Australian trial the mean diazinon residues in milk were <0.01 mg/kg 70 hours (2.9 days) after a single spray at 1.2 times the GAP concentration compared to the Australian pre-slaughter interval of 3 days and the milk withdrawal intervals of 3 or 4 days in other countries.

A 1994 Egyptian trial was at the concentration of Egyptian GAP, but with a single application whereas 3 are permitted. The highest diazinon residue in the milk was 0.3 mg/kg 6 hours after application, but decreased to <0.005 mg/kg after 36 hours, half the 3-day Egyptian GAP withdrawal interval. Some details were not reported.

Residues in tissues. In a 1986 Australian trial a single spray at 1.2 times the GAP concentration gave maximum and mean/median diazinon residues of 0.7 and 0.6 mg/kg in kidney fat after 7 days, and 0.2 and 0.2 mg/kg in omental fat after 14 days. There were no data at the 3-day GAP pre-slaughter interval. Subcutaneous fat, which would be expected to have higher residues than other fats, was not analysed. After 7 days residues were ≤0.01 mg/kg in muscle, kidney and liver. In a 1996 Australian back spray trial a single GAP application gave diazinon residues in subcutaneous fat of <0.05 mg/kg in 5 animals and 0.08 mg/kg in one after 4 days. The residues in renal fat were <0.05 in 6 animals.

In summary the cattle spraying trials suggest that single spray applications according to GAP might produce the following results.

Sample	Residue, mg/kg			Pre-slaughter interval, days	
	Max.	Median	Mean	In trial	GAP
Milk			0.02	3-5	3-4
Loin (subcutaneous) fat	0.08	<0.05		4	3
Renal fat	0.7	0.6		7	3
Omental fat	0.2	0.2		14	3
Muscle, liver, kidney	0.01			7	3
	0.07			1	3
	0.03[1]			3[1]	3

[1] Estimated residue at GAP pre-slaughter interval. See discussion below.

Some of these residues may be lower than likely maximum levels. In particular, especially in view of the results of the sheep dipping trials, some qualification must attach to the residue in subcutaneous cattle fat in particular because no residues in subcutaneous fat were reported in the GAP trial which gave 0.7 mg/kg in renal fat after 7 days. Residues would have been be expected to be higher in subcutaneous fat, probably above 1 mg/kg. Similarly, even the 0.7 mg/kg in renal fat may be too low as a maximum residue level since it occurred after 7 days whereas the GAP withdrawal interval is 3 days, and residues in renal fat in 2 separate animals of the study after a 1-day withdrawal period were 1.3 and 2.9 mg/kg.

Special attention should also be given to the residues in muscle, kidney and liver since no results were available at the 3-day GAP pre-slaughter interval. The maximum and mean residues after 1 day were 0.06 and 0.06 mg/kg in muscle, 0.02 and <0.02 mg/kg in liver and 0.07 and 0.07 mg/kg in kidney. The Meeting concluded that although the residues would be lower after 3 days they would still exceed the 0.01 mg/kg found after 7 days, and estimated a maximum residue level of 0.03 mg/kg in muscle, liver and kidney from single spray applications to cattle according to GAP.

No conclusion could be drawn with confidence about the results of multiple GAP applications as only very old, inadequately reported, studies were available. Although one of these suggested that residues in omental fat might increase with multiple applications, there were no results at a GAP withdrawal interval, and no analyses of other (e.g. subcutaneous) fat or tissues. The Meeting therefore considered modern spray trials on cattle under maximum GAP conditions (which include multiple sprays) to be highly desirable. Analyses should preferably be for diazinon, diazoxon and hydroxydiazinon in milk, muscle, edible offal and fat (including kidney, omental and especially subcutaneous fat).

Sheep spraying. In a rather poorly reported Australian trial in 1971 with EC and WP formulations applied at twice the GAP concentration, diazinon residues did not exceed 0.16 mg/kg in fat or 0.09 mg/kg in muscle after the GAP pre-slaughter interval of 14 days. In a well-documented Swiss trial in 1994 residues in fat (from the base of the tail) were determined 28 days after a single spray at the GAP concentration. The Swiss withdrawal interval is 21 days. The maximum and median residues from each of three different EC formulations were 0.29 and 0.12 mg/kg, 0.24 and 0.16 mg/kg, and 0.22 and 0.11 mg/kg with an overall maximum and median of 0.29 and 0.14 mg/kg. The Meeting concluded that diazinon residues in sheep fat are unlikely to exceed 0.3 mg/kg after 28 days from a single spray application according to Swiss GAP. There was no information on multiple applications, for which data on residues in milk and tissues are desirable, nor on residues at the GAP withdrawal interval of 21 days.

Goat spraying. In a 1986 Australian trial with a single application approximating the GAP concentration, the residues in two goats after the 14-day Australian GAP pre-slaughter interval were <0.01 mg/kg in the muscle, liver and kidney of both animals, 0.02 and <0.01 mg/kg in kidney fat and 0.03 and 0.01 mg/kg in omental fat. Subcutaneous fat was not analysed. In a 1987 Australian trial the mean residues in milk were 0.02 mg/kg after 78 hours. The GAP withdrawal interval for milk is 3 or 4 days in other countries.

Pig spraying. In a fairly well documented 1974 Swiss trial diazinon residues were ≤0.01 mg/kg in muscle and <0.01 mg/kg in liver, kidney, unspecified fat and skin 28 days after one or two sprays at the GAP concentration. The Swiss pre-slaughter interval is 21 days. Even at twice the GAP concentration the residues were all <0.01 mg/kg except one residue of 0.04 mg/kg in muscle. The samples were also analysed for hydroxydiazinon, the pyrimidinol G 27550 and diazoxon, but the only measurable residue was 0.02 mg/kg of hydroxydiazinon in a single fat sample at the GAP spray concentration. The Meeting concluded that diazinon residues would be unlikely to exceed 0.01 mg/kg in pig tissues from Swiss GAP. For risk assessment purposes the figure would be 0.03 mg/kg.

Estimates of STMR levels

Because the number of trials of ectoparasite treatments were limited and the residues would be from different populations depending on the animal and the type of treatment, the Meeting considered using recommended MRLs for the estimation of dietary intake. However, to conform to the general approach to such estimations, the Meeting estimated STMR levels for animal products.

Poultry. No diazinon (<0.01 mg/kg) was detected in any sample of skin, muscle, eggs, fat or liver after feeding diazinon at a level equivalent to 10 times the expected dietary intake. The Meeting therefore concluded that the effective STMR for poultry meat, poultry edible offal and eggs should be zero.

Milk. Because milk is normally bulked before distribution, the Meeting used mean values of the residues in milk from different animals in individual trials as the basis for STMR estimates. The mean residues in milk from GAP applications were 0.02 mg/kg in cattle and goats from spraying and in sheep from dipping, and 0.01 mg/kg in cattle from ear tags. The Meeting estimated 0.02 mg/kg as a maximum residue level and an STMR level for milk.

Meat (muscle). Although the maximum residue level for use as an MRL for meat is expressed on a fat basis, for dietary intake purposes the Meeting also estimated an STMR for whole muscle. The distribution of maximum residues (mg/kg) in the meat of the animals treated according to GAP, with the types of treatment, were goats <0.01 (spray), pigs 0.01 (spray), cattle 0.02 (ear tags), 0.03 (spray, extrapolated value), and sheep 0.03 (dip). The Meeting estimated an STMR level of 0.02 mg/kg for the meat (whole muscle) of cattle, pigs, sheep and goats.

Edible offal. The residues from applications according to GAP (mg/kg) were as follows. Liver: goats <0.01 (spray), pigs <0.01 (spray), cattle <0.01 (ear tag), sheep 0.01 (dip), cattle 0.03 (spray, extrapolated value). Kidney: goats <0.01 (spray), pigs <0.01 (spray), cattle <0.01 (ear tag), sheep 0.02 (dip), cattle 0.03 (spray, extrapolated value). Liver and kidney combined, in rank order: <0.01 (6), 0.01, 0.02, 0.03, 0.03.

The Meeting estimated an STMR of <0.01 mg/kg for the liver and kidney of cattle, goats, pigs and sheep.

Fat. An STMR was estimated on the basis of the residues in the fat of cattle, goats, pigs and sheep from different uses against ectoparasites according to GAP. Combining the data for these animals gave the following distribution of residues in rank order.

Omental fat: 0.03, 0.2, 1.3 mg/kg.
Renal fat: 0.02, 0.04, 0.3, 0.7, 0.7 mg/kg.
Loin (subcutaneous) fat: <0.01, 0.05 0.08, 0.3, 0.7, 1.4 mg/kg (omitting an aberrant value of 4.3 mg/kg).

All fat: <0.01, 0.02, 0.03, 0.04, 0.05, 0.08, 0.2, 0.3, 0.3, 0.7, 0.7, 0.7, 1.3, 1.4 mg/kg.

The Meeting estimated an STMR of 0.3 mg/kg.

General observations. As has been noted, the trials of diazinon for ectoparasite control range from very old studies, unusable by current standards, to a few relatively recent well-documented trials reported to be in accordance with GLP. The Meeting has tried to make the best use of the available studies that

prudence allows. In many cases even where GAP concentrations have been applied results are lacking at GAP withholding periods. Another common observation is that most of the acceptable studies are with single applications rather than the multiple applications permitted by GAP. In some cases very similar trials have inexplicably produced inconsistent results and in others no data are available on residues in subcutaneous fat, which has been shown to have higher residues than other fat after dermal applications.

These factors have required the Meeting to exercise judgement in estimating maximum residue levels and have lead to some uncertainty as to whether the recommended MRLs are sufficiently high to cover all uses of diazinon as an ectoparasiticide according to GAP. It is also at least possible that in practice some animals might be exposed to more than one type of treatment (e.g. spraying or dipping as well as ear tags or wound dressings). For these reasons the Meeting concluded that additional modern trials with diazinon used for ectoparasite control at maximum GAP concentrations and with multiple dip and spray applications, conducted in accordance with GLP, are desirable in order to confirm the estimated maximum residue levels and STMR levels.

RECOMMENDATIONS

On the basis of the data on residues and information on GAP provided the Meeting estimated the maximum residue levels listed below, which are recommended for use as MRLs. Corresponding STMRs are also listed for estimating dietary intake. For considering long-term dietary intake estimates the Meeting also recommended the use of median residues for pome fruit, tomatoes and cabbages of 0.12, 0.12 and 0.16 mg/kg respectively rather than the respective proposed MRLs of 2, 0.5 and 2 mg/kg.

Definition of the residue for compliance with MRLs and for estimation of dietary intake: diazinon.

The residue is fat-soluble.

CCN	Commodity	Recommended MRL, mg/kg		STMR, mg/kg	Withholding interval, days
		New	Previous		
PO 0840	Chicken, edible offal of	0.02*	--	0	
P 0840	Chicken eggs	0.02*	--	0	
PM 0840	Chicken meat	0.02*		0	
MO 0099	Liver of cattle, goats, pigs and sheep	0.03 V	--	0.01	3
MO 0098	Kidney of cattle, goats, pigs and sheep	0.03 V	--	0.01	3
MM 0097	Meat of cattle, pigs and sheep	2 (fat) V	W[1]	0.3 (fat) 0.02 (whole muscle)	3
MM 0814	Goat meat	2 (fat) V	--	0.3 (fat) 0.02 (whole muscle)	3
ML 0106	Milks	0.02 F V	W[1]	0.02	3
STMRs for fruits and vegetables					

CCN	Commodity	Recommended MRL, mg/kg		STMR, mg/kg	Withholding interval, days
		New	Previous		
FP 0009	Pome fruits			0.12	
VO 0448	Tomato			0.12	
VB 0041	Cabbages, Head			0.16	

* At or about the limit of determination.
[1] Withdrawal of existing CXL proposed by 1993 JMPR.

FURTHER WORK OR INFORMATION

Desirable

1. Studies of the stability of diazinon, diazoxon and hydroxydiazinon in stored analytical samples of meat, fat, edible offal, milk and eggs.

2. Modern dipping and spray trials on sheep and cattle at maximum GAP rates and including multiple dips and sprays. Analyses for diazinon residues in milk, muscle, edible offal and fat (kidney, omental and especially subcutaneous fat) would be desirable, as well as analyses for diazoxon and hydroxydiazinon in addition to diazinon.

3. Data from monitoring analyses of subcutaneous fat of sheep for diazinon, ideally sheep known to have received multiple dip or spray applications at maximum GAP rates.

4. Submission, when the new supervised trials of ectoparasite control are submitted in 1998, of information on current US GAP for pome fruits and cabbages and data from recently completed US supervised trials reflecting that GAP.

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DITHIOCARBAMATES (105)

APPRAISAL

Ferbam, thiram and ziram were evaluated at the present Meeting within the CCPR periodic review programme. The information on, and the STMR estimates for, the three compounds are discussed in their monographs.

Recommended MRLs for dithiocarbamates arising from the uses of thiram and ziram are consolidated here under the dithiocarbamate heading. The estimates of maximum residue levels for dithiocarbamates which rely primarily on ziram data are recommended as TMRLs until relevant data on environmental fate are evaluated. There are no recommendations for MRLs for dithiocarbamates arising from applications of ferbam.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting estimated the maximum residue levels listed below which are recommended for establishing MRLs or TMRLs. Estimates of STMR levels and definitions of the residues for dietary intake risk assessment are provided for thiram and ziram in their monographs.

Definition of the residue: The MRLs refer to total dithiocarbamates, determined as CS_2 evolved during acid digestion and expressed as mg CS_2/kg.

Commodity		Recommended MRL, mg/kg		Compounds on which estimates of maximum residue levels are based[1]
CCN	Name	New	Current	
AM 0660	Almond hulls	20[2]	20	maneb, ziram
TN 0660	Almonds	0.1*	0.1*	maneb ziram
TN 0672	Pecan	0.1* T		ziram
FP 0009	Pome fruits	5	5	mancozeb metiram propineb thiram ziram
FS 0012	Stone fruits	7 T[3]		thiram ziram
FB 0275	Strawberry	5		thiram

[1] The estimates are mainly based on data from uses of the underlined compounds.
[2] The estimated temporary maximum residue level arising from the use of ziram is 10 mg/kg, but the current draft MRL of 20 mg/kg should be maintained to accommodate uses of maneb.
[3] The estimated maximum residue level for dithiocarbamates arising from the use of thiram on plums and cherries is 1 mg/kg, but a TMRL of 7 mg/kg is recommended to accommodate uses of ziram on stone fruits.

FENARIMOL (192)

EXPLANATION

Fenarimol was reviewed for the first time by the 1995 JMPR and a number of maximum residue levels were estimated. Although data on the environmental fate of fenarimol in soil were submitted to the Environmental Core Assessment Group at that Meeting they were not, as would normally be expected, submitted for the consideration of the FAO Panel. The manufacturer agreed to submit the data to the FAO for consideration by the FAO Panel at the 1996 JMPR. The 1995 Meeting decided that in these circumstances the maximum residue levels should be recommended only as temporary MRLs with a requirement for the environmental fate studies. The manufacturer has now submitted data on the environmental fate of fenarimol in soil.

The 1995 Meeting concluded that a maximum residue level of 5 mg/kg for dry hops would be appropriate but, since samples in the relevant trials were stored for 13 months before analysis, decided not to recommend an MRL for hops in the absence of data confirming the stability of fenarimol in a leafy crop. A study of the stability of fenarimol residues in dried hops has now been submitted.

METABOLISM AND ENVIRONMENTAL FATE

Environmental fate in soil and water/sediment systems

The characteristics of the soils in some of the studies reviewed are given below (Table 1).

Table 1. Characteristics of soils in the studies reviewed.

Ref.	Soil description[1]	% oc	pH	% sand	% silt	% clay	Pre-study microbial activity (µg C/g soil)
Rainey, 1990	Greenfield (sandy loam)	1.5	7.1	66	21	13	
Althaus & Beaty, 1982	Coarse (sandy loam)	0.9	6.0	54	32	14	
Althaus & Beaty, 1982	Medium (silty loam)	1.3	6.1	28	57	15	
Althaus & Beaty, 1982	Fine (clay loam)	1.9	6.3	20	50	30	
Perkins, 1993	Ismaning (clay loam)	4.9	7.3	1	90	9	950
Perkins, 1993	Rohr (clay loam)	2.8	7.1	36	42	22	32
Perkins, 1993	Alsfeld (silt loam)[2]	1.5	6.5	42	32	26	
Perkins, 1993	Grebin (sandy silt loam)	1.2	5.6	47	36	17	25
Saunders & Powers, 1987	Neuces (sand)	0.3	7.7	89	6	5	
Saunders & Powers, 1987	Fox (sandy loam)	0.8	5.7	66	22	12	
Saunders & Powers, 1987	Crosby (loam)	1.0	6.5	28	48	24	
Saunders & Powers, 1987	Brookston (clay loam)	1.2	6.9	24	44	32	
Smith & Saunders, 1982	Hancock (silt loam)	1.6	6.2	24	60	16	
Sullivan & Saunders, 1976	Marion (sand)	0.6	8.1	91	5	4	
Sullivan & Saunders, 1976	Synthetic (sandy loam)	2.0	5.6	69	21	10	

Ref.	Soil description[1]	% oc	pH	% sand	% silt	% clay	Pre-study microbial activity (µg C/g soil)
Sullivan & Saunders, 1976	Hancock (loam)	1.2	7.7	40	34	26	
Sullivan & Saunders, 1976	Hancock (clay loam)	0.8	5.6	36	36	28	
Vonk & Hoven, 1981	Droevendaal (sand)[3]	2.5	5.0	86	6	4	
Vonk & Hoven, 1981	Lelystad (loam)[3]	1.7	7.5	32	36	21	

[1] UK or USA classification of sand, silt and clay used unless otherwise stated
[2] Sand >20-2000 µm, silt 2-20 µm, clay <2µm
[3] Sand 50-2000 µm, silt 2-50µm, clay <2µm

Degradation in soil - laboratory studies. In a study according to the German BBA guidelines (Jackson and Lewis, 1994) [*carbinol*-^{14}C]fenarimol (radiochemical purity 97.3%) was incubated with Marcham clay loam, Faringdon clay, Marcham sandy loam and Speyer 2.2 loamy sand at concentrations of 0.05 and 0.25 mg/kg, equivalent to 0.1 and 0.5 kg/ha, at 40% maximum water holding capacity and 20°C. Ethanolamine traps were used to collect gaseous compounds.

Single (duplicate for Speyer 2.2) samples were taken at six representative times up to 180 days after application at the low rate and at four times at the high rate. The soils were Soxhlet-extracted with 2-butanol/water and analysed by TLC. The extraction of fenarimol by this method was initially >95% and during the course of the experiment mass balances were generally 95-105%. Results are shown in Table 2.

Table 2. Degradation of fenarimol in soil; laboratory study.

Soil	Half-life of fenarimol, days	
	Low application rate	High application rate
Marcham clay loam	473	917
Marcham sandy loam	436	889
Faringdon clay	542	1204
Speyer 2.2 loamy sand	1360	1833

The radioactivity in the ethanolamine trap accounted for <5% of the applied ^{14}C and was assumed by the authors to be $^{14}CO_2$. Unextractable residues in the soil rose to 3.3-17.2% of the applied radioactivity (AR) at 180 days. Two unidentified products were extracted from the soil, each accounting for <3% of the AR at any time.

In a further study according to USA EPA guidelines (Rainey, 1990) [*carbinol*-^{14}C]fenarimol (radiochemical purity 97.6%) was evenly mixed with Greenfield sandy loam at 75% 33 kPa moisture content to produce a final concentration of 5 mg/kg (equivalent to 9.75 kg/ha). Soil was placed in a closed flow-through system with KOH and charcoal traps to collect volatile products and incubated in the dark at 24°C for one year. After ten representative intervals aliquots of soil (15 g) were removed, extracted with methanol/water under reflux (and later samples also with butanol/water under reflux) and the extracts analysed by TLC.

The mass balance for the radioactivity in combusted soil did not change over the year and was all accounted for in the various fractions after extraction. After one year the total radioactivity collected from the KOH trap had reached 0.6% of the AR and the unextractable residues in the soil

had risen steadily to 9.4%. A single product, identified by MS as Æ-(2-chlorophenyl)-Æ-(4-chlorophenyl)-1,2-dihydro-2-oxo-5-pyrimidinemethanol, reached a maximum level of 4.1% after 6 months. Fenarimol levels were reported to have decreased to 79% after one year and a half-life of 840 days was calculated.

Althaus and Beaty (1982) added [*carbinol*-^{14}C]fenarimol (radiochemical purity 98.9%) to coarse, medium and fine soils to produce a final concentration of 5 mg/kg (equivalent to 9.75 kg/ha). The soils were adjusted to 75% of 33 kPa moisture content and kept in the dark at 20-25°C for one year. Samples were removed at ten intervals, Soxhlet-extracted with 2-butanol/water and analysed by TLC. Anaerobic degradation was investigated in a further set of soils which were prepared in the same manner but flooded with water after four weeks of aerobic degradation and incubated for a further 4-8 weeks.

In the aerobic experiment the total radioactivity recovered throughout the study was 88-113% of the AR. Over the one-year period the unextractable radioactivity rose to 3.9-5.8% of the AR. Fenarimol accounted for 78-83% of the extractable radioactivity in the one-year samples and the remaining extractable radioactivity was not attributable to any individual compounds.

In the anaerobic system 94-96% of the total radioactivity remained as fenarimol after 8 weeks incubation and only 4% of the radioactivity was in the water phase.

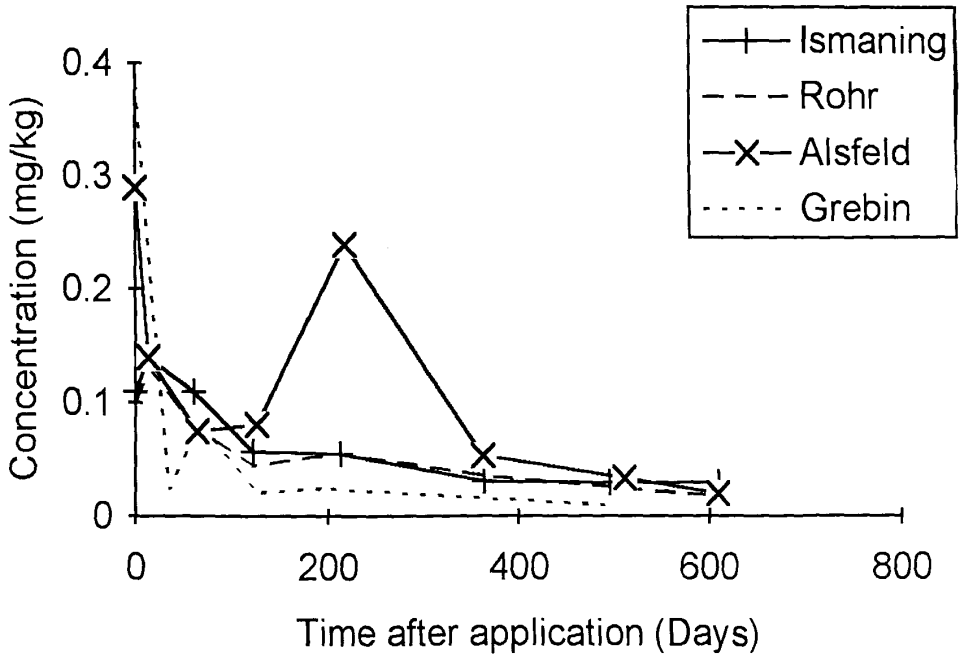
In a study by Althaus and Bewley (1978b) [*carbinol*-^{14}C]fenarimol (radiochemical purity >99%) was mixed with Crosby silt loam soil (3 kg, properties not given) to obtain a concentration of 1 mg/kg (equivalent to 1.95 kg/ha). The soil was then flooded with water and the atmosphere purged with nitrogen. The incubation temperature was not stated but after one year 98% of the AR was in the soil and 91% was extracted from soil/water samples as fenarimol.

Degradation in soil - field studies. An EC formulation of fenarimol was sprayed at 0.27 kg ai/ha on to bare soil at four German field sites (Ismaning, Rohr, Alsfeld and Grebin) in May 1990 (Perkins, 1993). Cores were taken at each site at 0, 14, 60, 120 and 210 days (100 cm depth) and at 12, 16 and 20-24 months (30 cm depth). The 100 cm cores were sectioned into depths of 0-10, 10-20, 20-40, 40-60 and 60-100 cm except in the Grebin trial where the samples from 0 to 60 days were inadvertently bulked. The 30 cores were divided into 0-10, 10-20 and 20-30 cm sections.

Each sub-section of the soil cores was mixed and sieved. Sub-samples were extracted with methanol/water, cleaned up and analysed by GLC. The LOD of the method was 0.05 mg/kg and the mean recovery was 94%.

0-10 and 10-20 cm soil cores were combined before analysis and no deeper samples were analysed. In the early Grebin cores the totals found in the 100 cm cores was assumed to be in the 0-20 cm range and concentrations were calculated accordingly. At Grebin and Alsfeld very high concentrations of fenarimol found at day 0 (three to four times those predicted from the application rate) resulted in short calculated half-life (DT50) values. The results are plotted in Figure 1 and the calculated DT50 and DT90 values are given in Table 3.

Figure 1. Concentrations of fenarimol in four German field soils.



The DT50 and DT90 values estimated from Figure 1 are shown in Table 3.

Table 3. DT50 and DT90 values for four German soils.

Soil	DT50, days	DT90, days
Ismaning	123	>610
Rohr	95	>610
Alsfeld	14	550
Grebin	21	120

In a further study according to BBA guidelines (Butcher and Rawle, 1994) an EC formulation of fenarimol was sprayed at 270 g ai/ha on to bare soil at two German field sites (Herford and Gornitz) in May 1992. Cores (20 cm long) were taken at each site and at day 0 they were sectioned into 0-10 and 10-20 cm and analysed separately. At days 14, 31-32, 90-92, 214, 361-365, 488-389 and 609 they were analysed as 0-20 cm.

The initial concentrations in the 10-20 cm sections were below the LOD at Gornitz and

0.006 mg/kg at Herford; the concentrations in the 0-10 cm sections were incorrectly calculated and should have been half those reported. The DT50 and DT90 values are given in Table 4 below.

Table 4. DT50 and DT90 values for two German soils.

Soil	DT50, days	DT90, days
Herford	60	489
Gornitz	130	>609

Crosby silt loam soil (properties not given) was used to fill steel cylinders to a height of 10 cm. [*Carbinol*-^{14}C]fenarimol (radiochemical purity >99%) was added to the surface at a nominal rate of 1.2 kg/ha and the cylinders were placed in the ground and subjected to normal weather conditions (not detailed) at Greenfield, Indiana, USA (Althaus and Bewley, 1978a). One cylinder was removed and the soil mixed at each sampling point at intervals during 511 days.

In another experiment at the same site (Althaus and Bewley, 1978b) was incorporated into the top 7.6 cm of a Crosby silt loam field plot at application rates of 1.1 and 5.6 kg/ha. Soil cores (depth at least 15 cm) were taken at each sampling point at intervals for 129 weeks.

In both experiments soil was Soxhlet-extracted with methanol and sometimes also butanol/water and analysed by TLC. In the experiment in which fenarimol was applied to the surface the half-life was 112 days and after 511 days 35% of the AR was extractable as fenarimol, 13% was unextractable, and unidentified extractable radioactivity accounted for 9% of the AR with no discrete region >5%. The rest of the radioactivity was reported as being dissipated but was not otherwise accounted for. Where fenarimol was incorporated into the soil no radioactivity was lost from the 0-15 cm core, but at times after 27 weeks 7-28% of the AR was found in the 7.6-15 cm section. No difference in the rate of degradation was observed between the two application rates. After 189 days extractable residues other than fenarimol individually accounted for <5% of the AR and after 903 days 65% of the AR was still fenarimol.

A long-term field dissipation study was carried out at locations in California, Florida, Indiana, and Maryland, using a WP formulation of fenarimol (Day, 1982). The soils were described as loam, sand, clay loam and silt loam although no detailed soil analysis was reported.

Two studies were conducted at each location. In the first, bare soil plots were divided to receive applications of fenarimol at 3.4 or 6.7 kg ai/ha in either the first year only, the first two years only, or for three years. In the second, bare soil plots were treated six to eight times with fenarimol at 0.56 kg ai/ha at biweekly intervals.

Soil samples were taken according to a defined schedule from all plots, in a few cases for as long as four years after application. Dissipation constants and DT50 values averaged from both investigations are shown in Table 5.

Table 5. Dissipation constant and DT50 values at four locations in the USA.

State	Average dissipation constant per day	DT50, days
California	0.00106	651
Florida	0.00722	98

Indiana	0.00217	322
Maryland	0.00167	413

The rate of dissipation was reported to be correlated with the moisture history of the area. Shorter half-lives were observed where the rainfall or supplemental irrigation was heavier.

Adsorption, desorption and mobility in soil

In a study according to US EPA guidelines, [*carbinol*-^{14}C]fenarimol (>99% radiochemical purity) dissolved in dilute $CaCl_2$ solution was added to four different soils in triplicate and equilibrated for 22 h at 25°C (Saunders and Powers, 1987). After centrifugation, supernatant (22 ml) was removed and desorption measured by equilibrating the soil with fresh dilute $CaCl_2$ solution for 22 h. This step was then repeated. The radioactivity present in the solutions was quantified by LSC. A further experiment showed that there was no adsorption to the glass centrifuge tubes. The results shown in Table 6 differ slightly from those given in the study which were calculated without converting the organic matter content to the organic carbon content. The slopes of the desorption isotherms were found to be less than those of the adsorption isotherms.

Table 6. Freundlich adsorption constants for fenarimol in four soils.

Soils	Adsorption			Desorption
	K_d	Slope 1/n	K_{oc}	K_d
Neuces (sand)	1.5	0.901	500	1.4-2.6
Fox (sandy loam)	5.1	0.858	634	5.1-11.5
Crosby (loam)	8.1	0.873	810	8.2-17.3
Brookston (clay loam)	11.9	0.861	992	12.7-28.7

Air-dried, sieved soils (Marion sand, synthetic sandy loam, Hancock loam, Hancock clay loam) were packed into 30 cm columns. [*Pyrimidine*-^{14}C]fenarimol (radiochemical purity 98%) was added (326 µg, equivalent to 1 kg/ha) to triplicate columns of each soil in a minimal amount of benzene which was allowed to evaporate overnight (Sullivan and Saunders, 1976). The columns were leached with water (2 litres, 64 cm) for 2-4 days. After extraction, the radioactivity in the soil and water was determined by LSC.

Depending on the soil, the first 250-400 ml water added was required to bring the soil to water capacity before leaching began. Recovery of the applied radioactivity was only 67-83%. In the four soils 0-0.4% of the recovered radioactivity was found in the leachate whilst 91-100% remained in the top 10 cm of the soil. By comparison, atrazine leached in the same soils showed 3.4-43% in the leachate and an even spread of the compound throughout the soil.

Columbus sandy loam soil (stored air-dried and water added one month before incubation to re-establish biological activity) maintained at 75% of 1/3 bar moisture content was incubated at 23-24°C in the dark for 30 days with a mixture of [*carbinol*-^{14}C]fenarimol (radiochemical purity 96%), [4-*chlorophenyl*-^{14}C]fenarimol (radiochemical purity >99%) and [2-*chlorophenyl*-^{14}C]fenarimol (radiochemical purity >99%) (Saunders *et al*., 1983).

Dry soil (Marion sand, Columbus sandy loam, Greenfield loam or Greenfield clay loam) was packed to a height of 25 cm in glass columns (1 cm diameter) and 5 cm of the aged soil containing

fenarimol was added. The columns were leached with water (40 ml, 51 cm) and the radioactivity in the soil and leachate determined by LSC.

After ageing the soil was found to contain 93% of the AR and after leaching recoveries of ^{14}C were 79.7-93.7% of the AR; some losses were considered to be due to volatilization during soil drying processes. Radioactivity in the leachate was 0.24-0.32% of the AR and almost all the radioactivity in the soil was in the top 12 cm. K_d values based on the distance moved by the ^{14}C were reported to be 2.7-7.3.

[*Carbinol*-^{14}C]fenarimol (radiochemical purity not stated) was photochemically degraded by exposure to natural sunlight for 50 h (Vonk and Hoven, 1981). This was then dissolved in methanol and found to contain 80 % of the AR of which 57.5% (46% of the AR) was fenarimol. Solutions of the degraded fenarimol were mixed with Droevendaal sand and Lelystad loam (10 g) at concentrations stated to be equivalent to 0.4 and 1.6 kg/ha. Columns 4.3 cm diameter were packed to a depth of 25 cm with air-dried, sieved soils and saturated with 25 mM $CaSO_4$ solution. The treated soil was added to the columns which were then leached with 25 mM $CaSO_4$ (30 cm) for 3 days. The levels of radioactivity in the leachates and soils were determined by LSC and its nature investigated by TLC.

The total recovery of the radioactivity applied to the columns was 88-100%. In the sand soil 1.7% was detected in the leachate whilst 80-92% remained in the top 5 cm of the columns and in the loam soil the corresponding figures were 5.5% and 89% (both soils). At the higher application rate slightly more radioactivity (4.5-9%) was found in the leachate and the major component was *o*-chlorobenzoic acid (34-50% of the radioactivity present). The remainder of the radioactivity was associated with a complex mixture of very polar compounds.

Photolysis in soil. [^{14}C]carbinol-, [^{14}C-]*p*-chlorophenyl- or [^{14}C]*o*-chlorophenyl-labelled fenarimol, or a mixture of the three, (radioactive purities >99%) were deposited in baking dishes by evaporation of a dichloromethane solution and exposed to natural sunlight in Indiana, USA, between December and February (the maximum temperature during this period was 18°C). After 100 days the dishes were thoroughly washed with methanol, and the extract purified by column chromatography and analysed by TLC (Althaus, 1984).

At the conclusion of the photolysis period 72-85% of the AR remained, suggesting that 15-28% had been lost by volatilization. Fenarimol accounted for 33-38% of the AR. Many products were observed but none individually accounted for >6%. The major product (3.1-5.7% of the AR) was *o*-chlorobenzoic acid and only one additional compound was seen by labelling in the phenyl rings (<1% of the AR).

In a further briefly summarized study (Althaus and Donoho, 1977) the examination of mixtures irradiated with natural or artificial light in which 40-50% of the [^{14}C]fenarimol had been degraded indicated the formation of more than 50 photodegradation products, but the most abundant products detected were less than 3% of the AR.

Comparisons of the TLC profiles and autoradiograms of the photolysis mixture and soil extracts indicated that the photodecomposition products were present in the soil, but the most abundant degradation product in soil accounted for less than 2% of the AR. The main compound in the soil extract, confirmed as fenarimol, accounted for 92% of the radioactivity.

Fenarimol formulated as either an EC or WP was added dropwise to 0.4 mm layers of silt

loam soil in petri dishes (Smith & Saunders, 1982a). Samples were exposed to natural sunlight in Indiana, USA, for periods up to 32 h. Soil was extracted by boiling with methanol/water and, after clean-up, analysed by GLC. There was no degradation of fenarimol after 32 h.

Photolysis in water. [*Carbinol*-^{14}C]fenarimol (radiochemical purity >99%) was dissolved in distilled water, sealed in ampoules and irradiated at 28°C under artificial light for 4 h (Smith and Saunders, 1982b). Analysis by GLC showed fenarimol to have decreased to 52% of its initial concentration and the tentatively identified 2′-chloro-2-(5-pyrimidinyl)-4-chlorobenzophenone to have increased to 17%. TLC and LSC showed ten other photodegradation products but none individually accounted for >3.3% of the AR.

A further study in which aqueous photolysis half-lives of fenarimol were calculated (Saunders, 1991) was submitted but not reviewed.

Stability of pesticide residues in stored analytical samples

Samples of two varieties of hops were fortified at 1 mg/kg and stored below -16°C for nearly two years. Samples were taken at intervals and analysed by GLC with an ECD. The procedural recoveries were in the range 73-96%. Residues of fenarimol (uncorrected for recovery) ranged from 0.66 to 0.91 mg/kg during the storage period as shown in Table 7.

Table 7. Concentrations of fenarimol in fortified hops (Target and Golding varieties) following storage at <-16°C.

Storage time, days	Residue, mg/kg	
	Target	Golding
0	0.79	0.76
161	0.78	0.86
276	0.66	0.84
371	0.75	0.70
463	0.83	0.91
666	0.72	0.66
706	-	0.83

APPRAISAL

Fenarimol was reviewed for the first time by the 1995 JMPR and a number of maximum residue levels were estimated. Although data on the environmental fate of fenarimol in soil were submitted to the Environmental Core Assessment Group at that Meeting they were not, as would normally be expected, submitted for the consideration of the FAO Panel. The manufacturer agreed to submit the data to the FAO for consideration by the FAO Panel at the 1996 JMPR. The 1995 Meeting decided that in these circumstances the maximum residue levels should be recommended only as temporary MRLs with a requirement for the environmental fate studies. The manufacturer has now submitted data on the environmental fate of fenarimol in soil.

The 1995 Meeting concluded that a maximum residue level of 5 mg/kg for dry hops would be appropriate but, since samples in the relevant trials were stored for 13 months before analysis, decided not to recommend an MRL for hops in the absence of data confirming the stability of

fenarimol in a leafy crop. A study of the stability of fenarimol residues in dried hops has now been submitted.

The rate of aerobic degradation in the laboratory was examined in five soils in the dark in two studies. These showed that at 20-24°C the degradation of fenarimol was very slow with a half-life of 436-1833 days. The production of CO_2 was low (<5%) over the 180- and 365-day studies, as were the levels of unextractable residues which amounted to 3.3-17.2% of the applied radioactivity (AR). The levels of degradation products were also low (up to 4.1%) and only one of the compounds was identified, Æ-(2-chlorophenyl)-Æ-(4-chlorophenyl)-1,2-dihydro-2-oxo-5-pyrimidinemethanol.

Dissipation under field conditions was investigated in two recent studies at six sites across Germany. Although samples were taken from several depths at some of the sites, only 0-20 cm cores were analysed in all the trials. At two sites (Grebin and Alsfeld) very high concentrations of fenarimol at day 0 (three to four times those predicted from the application rate) resulted in short DT50 values. Over all the trials the DT50 values ranged from 14 to 130 days (60-130 days excluding the Grebin and Alsfeld sites). Fenarimol persists in the soil and in five of the studies the DT90 value was more than one year. An earlier study in the USA gave a half-life of 112 days for fenarimol applied to the surface. When fenarimol was incorporated into the soil the half-life was >903 days with 35% of the AR as fenarimol, 13% unextractable and 9% extractable but unidentified after 511 days.

In further studies at four US sites fenarimol was lost slowly from bare soil with average DT50 values ranging from 98 to 651 days. The rate of dissipation appeared to increase with increasing soil moisture.

Four soils were examined to determine sorption, and K_{oc} values in the range 500-992 g/ml were calculated. In column leaching experiments with four soils the overall recovery of radioactivity was not high, but only 0-0.4% of the recovered radioactivity was found in the leachate after about 1.7 litres had been collected. Most of the of the radioactivity (91-100%) was recovered from the top 10 cm of the soil column. When fenarimol was incubated in soil for 30 days before leaching 0.28-0.32% of the AR was found in the leachates from columns of four different soil types. Slightly more radioactivity (1.7-9% of the AR) was leached when fenarimol-soil mixtures were aged in sunlight and the degraded fenarimol applied to a soil column. The majority of this radioactivity was found to be from *o*-chlorobenzoic acid.

In a photolysis experiment in Indiana, USA (maximum temperature 18°C), in which fenarimol was deposited in baking dishes and exposed to natural sunlight, 33-38% of the initial fenarimol was still present after 100 days. In a further study of irradiated mixtures in which 40-50% of the [^{14}C]fenarimol had been degraded, more than 50 low-level photodegradation products were separated but no major products were detected. In contrast a further experiment in Indiana showed no photolysis of fenarimol on a soil surface after 32 h exposure.

In a study of aqueous photolysis the major tentatively identified photodegradation product was 2′-chloro-2-(5-pyrimidinyl)-4-chlorobenzophenone, which accounted for 17% of the AR after 4 h incubation. Ten other unidentified products were observed, each accounting for <3.3% of the AR.

The Meeting considered these data on environmental fate to be satisfactory and recommended the use of the maximum residue levels estimated by the 1995 Meeting as MRLs, which should no longer be temporary.

Data on the stability of fenarimol in stored analytical samples of dry hops were reviewed. Residues were stable up to 2 years in dry hops fortified at 1 mg/kg and stored at <-16°C. The Meeting decided to recommend the maximum residue level of 5 mg/kg provisionally estimated by the 1995 JMPR for use as an MRL.

The Meeting noted the high persistence of fenarimol in soil, and recalled that the 1995 Meeting had listed as desirable a study to assess the likely residues in relevant succeeding or rotational crops. The 1995 JMPR report (Section 2.5.2) referred to the need to submit data on the uptake of compounds by crops from the soil.

The Meeting was informed that no data were available on the uptake from soil by crops, the bioavailability of fenarimol, or residues in rotational/succeeding crops, but a rotational crop study would be completed by 1997 and the data from it would be made available to a future Meeting.

RECOMMENDATIONS

The maximum residue levels shown below are recommended for use as MRLs.

Definition of the residue for compliance with MRLs and for estimation of dietary intake: fenarimol

Commodity		Recommended MRL, mg/kg		PHI on which based, days
CCN	Name	New	Previous	
AB 0266	Apple pomace, dry	5	5 T	-
VS 0620	Artichoke, Globe	0.1	0.1 T	7
FI 0327	Banana	0.2	0.2 T	0
MO 1280	Cattle, kidney	0.02*	0.02* T	-
MO 1281	Cattle, liver	0.05	0.05 T	-
MM 0812	Cattle meat	0.02*	0.02* T	-
FS 0013	Cherries	1	1 T	0
DF 0269	Dried grapes (= Currants, raisins and sultanas)	0.2	0.2 T	-
FB 0269	Grapes	0.3	0.3 T	14
DH 1100	Hops, dry	5	-	10
VC 0046	Melons, except Watermelon	0.05	0.05 T	1
FS 0247	Peach	0.5	0.5 T	7
TN 0672	Pecan	0.02*	0.02* T	30
VO 0445	Peppers, Sweet	0.5	0.5 T	7
FP 0009	Pome fruits	0.3	0.3 T	14-28
FB 0275	Strawberry	1	1 T	1

FURTHER WORK OR INFORMATION

Desirable

1. Full details of the methods of analysis used in all the residue studies where this information was not given. Validation of the methods of analysis for which validation data were not submitted (repeated from 1995 JMPR).

2. Information on the melting point, octanol/water partition coefficient, solubility and specific gravity of pure fenarimol (repeated from 1995 JMPR).

3. Submission of the study reports supporting the trials on apples, gooseberries, currants, gherkins and strawberries conducted in The Netherlands (repeated from 1995 JMPR).

4. Submission of the study on residues in rotational crops which the Meeting was informed would be completed in 1997.

5. An investigation into the uptake and translocation of fenarimol residues into crops from soil and their translocation. If the data indicate that measurable residues could occur in rotational crops, then a study to assess the nature of the residues in representative rotational crops.

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FERBAM (DITHIOCARBAMATES, 105)

EXPLANATION

Ferbam was originally evaluated in 1965 (toxicology) and 1967 (toxicology and residues) and is included in the dithiocarbamate group. Ferbam is a broad spectrum fungicide for the control of certain diseases in fruit trees, small fruits and berries, potatoes, ornamentals, conifers and tobacco.

The compound was evaluated at the present Meeting within the CCPR Periodic Review Programme.

IDENTITY

ISO common name: ferbam

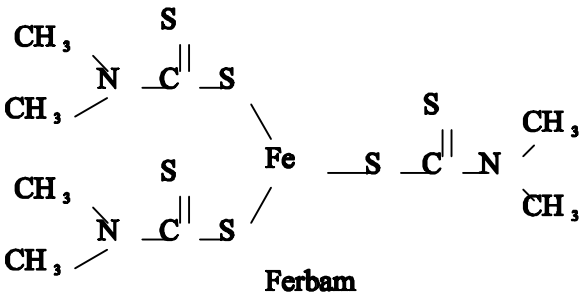
Chemical name

IUPAC: iron tris(dimethyldithiocarbamate)
iron(III) dimethyldithiocarbamate
ferric dimethyldithiocarbamate

CA: (*OC*-6-11)-tris(dimethylcarbamodithioato-*S*,*S*')iron

CAS registry no: 14484-64-1
CIPAC no: 57
EEC no: 238-484-2

Structural formula:



Ferbam

Molecular formula: $C_9H_{18}FeN_3S_6$

Molecular mass: 416.51

Physical and chemical properties

Pure active ingredient

Vapour pressure:	$<1.2\times10^{-4}$ Pa at 25°C (Lemal, 1986a).
Melting point:	>120°C
Octanol/buffer pH 8	
partition coefficient:	log P_{OW} = -1.6 (Lemal, 1986b)
Specific gravity:	0.21 g/cm^3

Lemal (1986a) measured the vapour pressure of ferbam by a gas saturation method. Nitrogen gas was passed through ferbam coated on a support material with a very high surface area and maintained at 25°C, then through a cotton wool dust filter followed by traps containing water. The contents of the absorption traps were analysed for iron by atomic absorption spectrometry. No iron was detected in the traps. The vapour pressure of ferbam at 25°C did not exceed 1.2×10^{-4} Pa.

Lemal (1986b) measured the octanol-water partition coefficient of ferbam (96%) according to OECD Guideline 107 (OECD 1981). Instead of water a pH 8 buffer was used. The octanol and aqueous phases were analysed for Fe using inductively coupled plasma emission spectrometry. The concentration of ferbam in the pH 8 buffer was about 40 times that in the octanol. In a series of tests the values for log P_{OW} ranged from -1.6857 to -1.4582, with a median value of -1.6292 at 20°C.

Technical material

The Meeting was informed that ferbam technical is not produced as such, but the synthesised ferbam is taken through the manufacturing process direct to the formulated product.

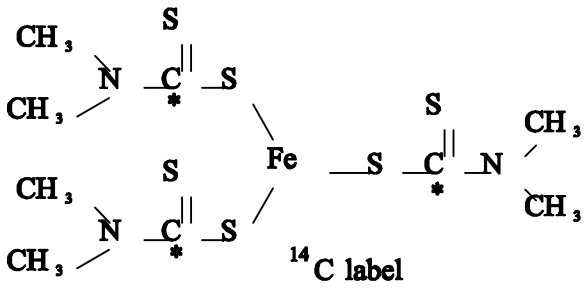
Formulations

Water dispersible granule, 76WG.

METABOLISM AND ENVIRONMENTAL FATE

Animal metabolism

Information was made available to the Meeting on studies of ferbam metabolism in lactating goats and sheep.



Tissue, milk and excreta residues were measured in lactating goats (2 goats, each weighing approximately 45 kg) dosed once daily after the morning milking for 5 consecutive days orally by capsule with 500 mg [*thiocarbonyl*-^{14}C]ferbam at 11.4 and 11.1 mg/kg bw/day, equivalent to 220-250 ppm ferbam in the feed (Daun, 1993). The feed consumption was 1 kg/animal/day of a grain

mixture (shelled maize, oats, mineral mix, wet molasses, dairy pellets) as well as alfalfa grass hay provided *ad libitum*; the mean total daily feed consumption was 2.0-2.3 kg. The animals were hand-milked twice daily; the milk production was 1.5-1.6 kg per day. Milk and excreta were collected throughout, and the goats were slaughtered 6 hours after the final dose for tissue collection.

The distribution of ^{14}C was as shown below.

	^{14}C % of dose	
	Goat 1	Goat 2
Milk	0.70	0.79
Urine	8.3	11.9
Faeces	32.1	25.0
Tissues	2.7	5.6
TOTAL	43.8	43.3

Levels of ^{14}C in the milk increased for the first 2 or 3 days of feeding and then reached a plateau (Table 1). Levels of ^{14}C were higher in the liver than in other tissues (Table 2) and higher in the kidneys than in fat and muscle. Less than 50% of the dose was accounted for by the ^{14}C found in the milk, urine and faeces. It is possible that ^{14}C was lost as $^{14}CS_2$ or $^{14}CO_2$ in expired air, which was not collected.

Most of the ^{14}C residues in the milk, liver, kidneys, muscle and fat were not extractable with a chloroform/methanol/water mixture until after protease treatment. The soluble ^{14}C components produced by protease were mostly polar water-soluble compounds. Lactose and casein containing the ^{14}C label were isolated from the milk. Urea containing ^{14}C was isolated from the urine. This evidence supported other strong indications that much of the ^{14}C in the milk and tissues had been incorporated into natural products.

Table 1. Levels of ^{14}C in milk produced by 2 goats dosed daily with [^{14}C]ferbam equivalent to 220-250 ppm in the feed (Daun, 1993).

Day	^{14}C as ferbam, mg/kg milk			
	Goat 1, am milking	Goat 1, pm milking	Goat 2, am milking	Goat 2, pm milking
1		0.96		0.94
2	1.8	2.3	2.0	2.9
3	2.4	2.8	2.9	3.3
4	2.8	4.0	3.4	3.9
5	2.9	3.4	3.5	3.7

Table 2. Levels of ^{14}C in tissues and fluids from 2 goats dosed daily with [^{14}C]ferbam equivalent to 220-250 ppm in the feed and slaughtered 6 hours after the final dose (Daun, 1993).

Sample	^{14}C as ferbam, mg/kg	
	Goat 1	Goat 2
Bile	6.2	13.2
Blood	2.5	3.2
Fat (omental)	0.47	0.66
Fat (renal)	0.54	0.89
Kidneys	8.3	9.4
Liver	63	75
Muscle	1.5	1.9

Hunt and Gilbert (1976) dosed sheep (ewes) orally by gelatin capsule with [^{3}H]ferbam and [^{35}S]ferbam and examined the excreta and tissues for the radiolabels. One sheep weighing 32 kg was dosed with [^{3}H]ferbam at 0.74 mg/kg bw and [^{35}S]ferbam at 0.45 mg/kg bw, a total dose of 1.19 mg ferbam/kg bw. The second sheep, also weighing 32 kg, was treated with 14.5 mg [^{35}S]ferbam equivalent to 0.45 mg/kg bw. The sheep were slaughtered 72 hours after dosing for tissue collection and analysis.

Over 80% of the dosed ^{3}H but only 23-24% of the ^{35}S was excreted in the urine and faeces (Table 3). This is consistent with studies of thiram in rats which showed that 40-60% of the CS_2 part of the molecule was eliminated as volatile compounds in exhaled air. The level of ^{3}H in the liver was much higher than that of ^{35}S, demonstrating that the dithiocarbamic acid moiety had largely been degraded (Table 4).

Table 3. Excretion of ^{35}S and ^{3}H from 2 sheep dosed with [^{35}S]ferbam + [^{3}H]ferbam (sheep A) and [^{35}S]ferbam (sheep B) and slaughtered 76 hours later (Hunt and Gilbert, 1976).

% of dose					
Sheep A				Sheep B	
Urine		Faeces		Urine	Faeces
^{35}S	^{3}H	^{35}S	^{3}H	^{35}S	^{35}S
12	62	11	20	14	10

Table 4. Residues of ^{35}S and ^{3}H (expressed as ferbam) in tissues of 2 sheep dosed with [^{35}S]ferbam + [^{3}H]ferbam (sheep A) and [^{35}S]ferbam (sheep B) and slaughtered 76 hours later (Hunt and Gilbert, 1976).

Sample	Residues of radiolabel, expressed as ferbam, mg/kg		
	Sheep A		Sheep B
	^{35}S	^{3}H	^{35}S
Fat, omental	0.05	0.09	0.04
Kidneys	0.72	0.81	0.77
Liver	0.66	3.4	1.0
Muscle	0.14	0.22	0.11

Plant metabolism

No information was available.

Environmental fate

No information was available on the fate of ferbam in soils or in water/sediment systems.

METHODS OF RESIDUE ANALYSIS

Analytical methods

The methods for ferbam are the same as those for other dithiocarbamates: acid hydrolysis to release CS_2, which is then measured by head-space gas chromatography.

In the procedure of the Dutch method manual (Ministry of Welfare, Health and Cultural Affairs, The Netherlands, 1988) dithiocarbamates are converted to CS_2 by treatment with hydrochloric acid in the presence of stannous chloride. The CS_2 in the head-space is determined by GLC with either an ECD or FPD in the sulphur mode.

Westberg and Tufts (1990) described the CS_2 evolution head-space GLC procedure used in the ferbam mango trials. The sample was reacted with stannous chloride/hydrochloric acid reagent at 100°C in a sealed reaction flask. An aliquot of the head-space gas was analysed by GLC and compared with ferbam standards similarly reacted and injected. Recoveries were satisfactory at 0.02, 0.3, 0.5 and 7.0 mg ferbam/kg. The LOD was 0.02 mg ferbam/kg. Koch (1996) used a similar method in a study of the frozen storage stability of ferbam and ziram in apples. Satisfactory recoveries were recorded for apples fortified at 0.2 and 6 mg ferbam/kg.

Stability of pesticide residues in stored analytical samples

Koch (1996) tested the stability of ferbam residues in macerated apples fortified at 1 mg/kg and stored in head-space bottles at -20°C for 22 weeks. Samples were analysed as indicated above. Ferbam residues were stable under these storage conditions for the duration of the experiment.

Table 5. Freezer storage stability of ferbam in macerated apples fortified at 1 mg/kg and stored at -20°C (Koch, 1996).

Storage period	Ferbam remaining in stored sample, mg/kg (as ferbam)	Method recovery, %, at time of stored sample analysis
0 day	0.93, 0.94	109, 104
2 weeks	0.79, 0.84	81, 74
4 weeks	0.83, 0.90	76, 84
12 weeks	0.96, 0.90	82, 94
22 weeks	0.78, 0.83	72, 89

Definition of the residue

Ferbam residues are measured as evolved CS_2 by the same methods as are used for the other dithiocarbamates. The samples from the supervised trials on ferbam have been analysed by these methods. The Meeting agreed that ferbam should be included in the definition of the dithiocarbamate residues (*The MRLs refer to total dithiocarbamates, determined as CS_2 evolved during acid digestion and expressed as mg CS_2/kg*) if adequate critical supporting studies become available.

USE PATTERN

Ferbam is a broad-spectrum fungicide for the control of certain diseases in fruit trees, small fruit and berry crops, potatoes, ornamentals, conifers and tobacco. The Meeting was provided with information on registered uses on these crops. The registered uses of ferbam on fruits and potatoes are summarized in Table 6.

Table 6. Registered uses of ferbam on fruits and potatoes. All foliar applications.

Crop	Country	Form	Application			PHI, days
			Max rate per applic., kg ai/ha	Spray conc., kg ai/hl	Number	
Apple	USA	WG	0.86-1.7	0.09-0.18	3-5	7
Cherry	USA	WG	1.3	0.14	2-3	0
Citrus fruit	USA	WG	1.3	0.14	2	0
Cranberry	USA	WG	1.7	0.18	5	stage M28
Grape	USA	WG	1.7	0.18	3	7
Nectarine	USA	WG	1.3	0.14	1-2	21
Peach	USA	WG	1.3	0.14	1-2	21
Pear	USA	WG	1.3	0.14	5	7
Potato	UK	WP	0.17	0.043-0.085	~10	7

M28: 28 days after mid-bloom

RESIDUES RESULTING FROM SUPERVISED TRIALS

Data from supervised trials on mangoes are summarized in Table 7.

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 are not corrected for recoveries.

The trials were fully reported and well documented. Ferbam was analysed by a CS_2 evolution method and residues had been expressed as ferbam. All the residues reported in this monograph are expressed as CS_2 irrespective of the original expression. The theoretical factor 0.547 was used to calculate CS_2 values from ferbam values.

Ferbam was applied to two test plots of mangoes by an airblast sprayer. Each test plot comprised 93 trees. Four replicate samples were taken from each plot at each sampling and analysed separately. Samples were stored at -18°C for 3-3½ months after harvest before analysis.

Table 7. Residues of ferbam (as CS_2) in mangoes (Tommy-Atkins variety) from foliar applications of ferbam WG in supervised trials in the USA. The 4 values at each PHI are from 4 replicate samples. Residues are expressed on a whole fruit basis.

Application			PHI, days	Ferbam residues as CS_2, mg/kg	Ref.
kg ai/ha	kg ai/hl	No.			
4.3	0.18	11	0 7 14 21	0.43 0.39 0.13 0.32 0.08 0.17 0.37 0.44 0.17 0.12 0.17 0.11 0.17 0.43 0.07 0.14	UCB 27-FER/91049-11
4.3	0.18	16	0 7 14 21	0.28 0.41 0.44 0.36 0.31 0.10 0.25 0.85 0.14 0.23 0.12 0.26 0.16 0.06 0.25 0.12	UCB 27-FER/91049-16

FATE OF RESIDUES IN STORAGE AND PROCESSING

No information was available.

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

Monitoring data for dithiocarbamates are included in the monograph on thiram.

NATIONAL MAXIMUM RESIDUE LIMITS

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

Country	Commodity and MRL, mg/kg
Netherlands	see dithiocarbamates
USA[1]	almonds 0.1; apples 7; apricots 7; asparagus 7; beans 7; beet greens 7; beets 7; blackberries 7; blueberries 7; boysenberries 7; broccoli 7; Brussels sprouts 7; cabbage 7; carrots 7; cauliflower 7; celery 7; cherries 7; citrus fruits 7; collards 7; corn 7; cranberries 7; cucumbers 7; currants 7; dates 7; dewberries 7; egg plants 7; gooseberries 7; grapes 7; guavas 7; kale 7; kohlrabi 7; lettuce 7; loganberries 7; mangos 7; melons 7; mustard greens 7; nectarines 7; onions 7; papayas 7; peaches 7; peanuts 7; pears 7; peas 7; peppers 7; plums 7; pumpkins 7; quinces 7; radish tops 7; radishes 7; raspberries 7; rutabaga tops 7; rutabagas 7; spinach 7; squash 7; strawberries 7; summer squash 7; tomatoes 7; turnip greens 7; turnips 7; youngberries 7.

[1] Residue definition: residues calculated as zinc ethylenebisdithiocarbamate

APPRAISAL

Ferbam was originally evaluated in 1965 (toxicology) and 1967 (toxicology and residues) and is included in the dithiocarbamate group of compounds. The compound was evaluated at the present Meeting within the CCPR Periodic Review Programme.

Ferbam is a broad-spectrum fungicide for the control of certain diseases in fruit trees, small fruits and berries, potatoes, ornamentals, conifers and tobacco.

The Meeting received information on the metabolism of ferbam in goats and sheep, methods of residue analysis, the stability of residues in stored analytical samples, approved use patterns, and supervised residue trials on mangoes.

When two lactating goats were dosed for 5 days with [*thiocarbonyl*-^{14}C]ferbam at a rate equivalent to 220-250 ppm ferbam in the feed, the levels of ^{14}C in the milk increased for the first 2 or 3 days of feeding and then reached a plateau. A large part of the administered ^{14}C was not accounted for (59% and 62%). By analogy with the animal metabolism of thiram losses as CS_2 and CO_2 in expired air would be expected, but ^{14}C in expired air was not measured. More of the ^{14}C dose was in the faeces (25% and 32%) than in the urine (8.3% and 11.9%), tissues (2.7% and 5.6%) or milk (0.70% and 0.79%).

Levels of ^{14}C were much higher in the liver (63 and 75 mg/kg ferbam equivalent) than in the kidneys (8.3 and 9.4 mg/kg), muscle (1.5 and 1.9 mg/kg), or fat (0.47-0.89 mg/kg). Most of the ^{14}C in the milk and tissues was not extractable without protease treatment, which produced polar water-soluble compounds incorporating the ^{14}C. Lactose and casein containing ^{14}C were isolated from the milk.

In a sheep given a single dose of [^{3}H]ferbam + [^{35}S]ferbam and slaughtered 72 hours later for tissue collection more than 80% of the dosed ^{3}H but only 23-24% of the ^{35}S was excreted in the urine and faeces. This is consistent with the animal metabolism of thiram where 40-60% of the CS_2 part of the molecule was eliminated as volatile compounds in exhaled air.

The level of ^{3}H in the liver was much higher than in the other tissues, but the levels of ^{35}S in the liver and kidneys were much the same. The level of ^{3}H in the liver was much higher than that for ^{35}S, demonstrating that the dithiocarbamic acid moiety had largely been degraded.

The analytical methods for ferbam residues are the same as those for other dithiocarbamates. They rely on acid hydrolysis to release CS_2, which may then be measured by head-space gas

chromatography or by spectrophotometry.

The head-space GLC methods used in the supervised trials on mangoes and the frozen storage stability studies on apples gave satisfactory recoveries of ferbam.

Ferbam residues were stable in macerated apples fortified at 1 mg/kg and stored at -20°C for 22 weeks.

Generally, the information on ferbam was quite limited. For a compound in the periodic review programme an adequate set of supporting studies is needed. Because of the absence of plant metabolism and environmental fate studies the Meeting would not have been able to recommend MRLs for dithiocarbamate residues from uses of ferbam even if adequate information on GAP and data from supervised trials had been available for some commodities.

Dithiocarbamate MRLs are derived from supervised trials on specific dithiocarbamate compounds according to the relevant GAP. The table of recommended MRLs for dithiocarbamates indicates the compound or compounds for which data have been evaluated and found to be adequate to support the recommended MRL. Because of the lack of critical supporting studies ferbam is not included in the list of dithiocarbamates with adequate data to support recommended MRLs for dithiocarbamates.

The Meeting recognised that for most dithiocarbamates there are no practical regulatory analytical methods to identify the compound producing dithiocarbamate residues in a food commodity. National governments, under approval and registration systems, may control which uses are permitted.

Ferbam residues found in the supervised trials were measured as evolved CS_2 by the same methods as are used for the other dithiocarbamates. The Meeting agreed that ferbam would be included in the residue definition of the dithiocarbamates if adequate critical supporting studies become available.

The Meeting received data on residues of ferbam from two supervised trials on mangoes in the USA, but the data could not be evaluated because information on GAP for the use of ferbam on mangoes was not available.

Monitoring data for dithiocarbamate residues in commodities in trade are included in the monograph on thiram.

FURTHER WORK OR INFORMATION

Desirable

1. An adequate set of critical supporting studies for ferbam is needed before it can be included in the list of compounds supporting recommended MRLs for dithiocarbamates (see report of 1995 JMPR, Section 2.5.2).

2. Information on attempts to develop specific methods of analysis for ferbam, whether successful or not.

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FLUMETHRIN (195)

IDENTITY

Flumethrin is pyrethroid acaracide composed of a mixture of two diasterioisomers (*trans-Z*-1 and *trans-Z*-2, with an approximate ratio 55:45) formed by the reaction of 4-fluoro-3-phenoxybenzaldehyde and *trans*-(*E*)-3-[2-chloro-2-(4-chlorophenyl)vinyl-2,2-dimethylcyclopropanecarboxylic acid chloride in the presence of cyanide.

ISO common name: flumethrin

Chemical name

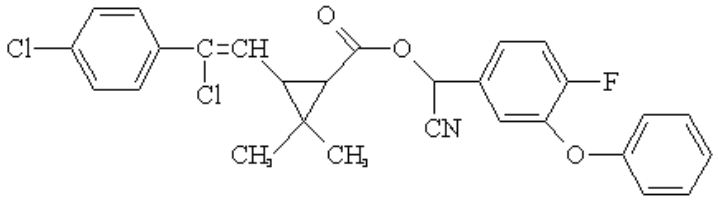
IUPAC: (*R,S*)-Æ-cyano-4-fluoro-3-phenoxybenzyl 3-(ß,4-dichlorostyryl)-2,2-dimethylcyclopropanecarboxylate

CA: cyano(4-fluoro-3-phenoxyphenyl)methyl 3-[2-chloro-2-(4-chlorophenyl)ethenyl]-2,2-dimethylcyclopropanecarboxylate

CAS No.: 69770-45-2

Synonyms: Bayticol; Bay VI 6045; FCR 1622; BAY Vq 1950; FCR 2769

Structural formula:



Molecular formula: $C_{28}H_{22}Cl_2FNO_3$

Molecular weight: 510.4

Physical and chemical properties (Krohn, 1995)

Pure active ingredient (98%, except determination of density)

Vapour pressure:

Sum of *Z*-1 and *Z*-2 partial v.p.	at 20°C	$<10^{-11}$ hPa
	at 25°C	$<10^{-10}$ hPa
Sum of *Z*-1 and *Z*-2 saturation v.p.	at 20°C	$<2 \times 10^{-4}$ ì g/m^3
	at 25°C	$<2 \times 10^{-3}$ ì g/m^3

Melting point: not provided
Octanol/water
partition coefficient: P_{ow} 1,600,000; log P_{ow} = 6.2

Solubility:	*Z*-1	*Z*-2	*Z*-1 + *Z*-2
(20°C, ì g/l)			
water (pure)	0.1	0.1	0.2
water (1% NaCl)	<0.03	<0.03	
water (pH 4 or 7 buffered)	<0.03	<0.03	
(Decreased solubility stated to be due to salinity, not to pH)			
water (pH 9 buffered)	Hydrolysed		
(20°C, g/l)			
n-heptane	11	8	19
xylene			>250
1,2-dichloroethane			>250
2-propanol	36	29	65
1-octanol	69	56	130
polyethylene glycol			100-200
acetone			>250
dimethylformamide			>250
acetonitrile			>250
ethyl acetate			>250
dimethyl sulfoxide			>250

Density (95% material): 1.28 g/cm^3 at 20°C
Hydrolysis: hydrolysed at pH 9
Photolysis: No information
Boiling point: >250°C, under decomposition
Thermal stability:

Differential thermal analysis (DTA) and thermogravimetric analyses (TGA) were employed, using OECD Guidelines. DTA indicated exothermic reaction above 270°C under nitrogen and 220°C in air. With TGA, weight loss started above 200°C in air and above 230°C in nitrogen.

Technical material

Purity Assay 90-100%
Sum of all by-products max. 10%

Formulations

6% EC solution (UK label for treatment of sheep provided).
75 g flumethrin/l liquid hydrocarbon solvent (solvent density 745 g/l, so 10% w/v ai) cattle dip and spray for cattle tick (Australian label provided).
10 g flumethrin/l pour-on solution for cattle tick and Buffalo fly control (Australian label provided).
Strip for pest control in bee hives (label provided).

METABOLISM AND ENVIRONMENTAL FATE

Information on the fate of flumethrin in rats and cattle was provided. Because flumethrin is used only for ectoparasite control on animals the manufacturer did not consider information on the fate of residues in plants, soil, or water/sediment systems to be applicable.

Animal metabolism

Oral, i.v. and duodenally administered flumethrin is hydrolysed to the substituted cyclopropanecarboxylic acid component (flumethrin acid) and (possibly through intermediate cyanohydrin and aldehyde oxidations) 4-fluoro-3-phenoxybenzoic acid. Flumethrin acid is conjugated to form the glucuronide and the benzoic acid component is oxidized to 4-fluoro-3-(4-hydroxyphenoxy)benzoic acid; both the hydroxylated and unhydroxylated acids are conjugated with glycine. A proposed metabolic pathway is shown in Figure 1, which is based on the following studies.

Rats. Because the fate of flumethrin in rats is considered in detail in the toxicological evaluation (Evaluations Part II - Toxicology), it will be described here only to the extent needed to view the metabolism in cattle in the context of the general metabolism in mammals. Five reports were available on the fate of flumethrin in rats, all from oral, i.v. or duodenal administration (none from topical application).

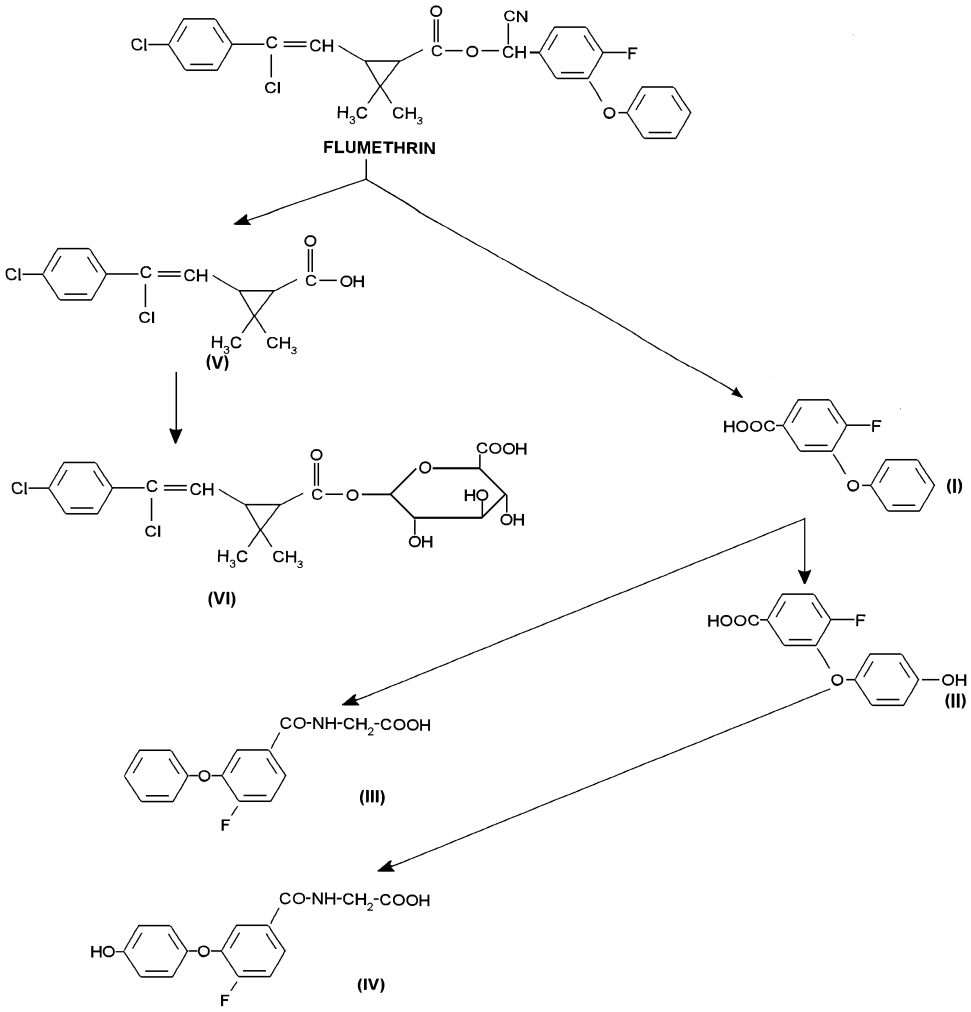
In a basic study on the elimination and metabolism of unlabelled flumethrin (Rauchschwalbe, 1980) rats were given a single oral dose. The author reported the presence of flumethrin and the metabolites I and V (Figure 1) in the faeces. The two metabolites were also eliminated in the urine, although the parent compound was not detected. Theoretical intermediates from the alcoholic portion of the molecule would also include the cyanohydrin (FCR 1271) and its oxidation product 4-fluoro-3-phenoxybenzaldehyde (FCR 1260), but their instability would make their detection unlikely and they were they reported.

The elimination of flumethrin and its metabolites in faeces peaked 3 or 4 days after administration, then dropped almost to zero. Residues of the two metabolites in urine dropped below the limit of detection within 5 days. Altogether 33% of the administered dose was eliminated in the urine and faeces.

A second study investigated the pharmacokinetics of the metabolism of fluorophenyl-labelled [^{14}C]flumethrin in rats after oral, i.v. and intraduodenal administration (Steinke *et al*., 1983). Approximately 50% of the ^{14}C was reported to be absorbed from oral administration, 45% of which was eliminated in the urine (the remainder in the faeces), compared with 75% renal elimination from i.v. administration. About 95% or more of the radioactive dose administered orally or intravenously was excreted within 48 hours. After 10 days only 1% of the administered ^{14}C was found in the animals. From duodenal administration, about 1/3 of the absorbed ^{14}C was eliminated via the bile.

In a study of the biotransformation of [U-*fluorophenyl*-^{14}C]flumethrin after oral administration (Ecker, 1983) the author reported the urinary elimination of two primary metabolites, 4-fluoro-3-(4-hydroxyphenoxy)benzoic acid (not reported by Rauchschwalbe) and 4-fluoro-3-phenoxybenzoic acid (found by Rauchschwalbe), 0-24 hours and 24-48 hours after administration. The hydroxyphenoxy metabolite accounted for 50 and 80% and the phenoxy metabolite for 35 and 10% of the radioactivity at these times. The glycine conjugates of the two primary metabolites were also reported, but accounted for at most 4 and 7.4% respectively of the urinary radioactivity.

Figure 1. Proposed metabolic pathways of flumethrin in rats and cattle.



(I) 4-fluoro-3-phenoxybenzoic acid
(II) 4-fluoro-3-(4-hydroxyphenoxy)benzoic acid
(III) 4-fluoro-3-phenoxybenzoylglycine (glycine conjugate of (I))
(IV) 4-fluoro-3-(4-hydroxyphenoxy)benzoylglycine
(V) 3-(ß,4-dichlorostyryl)-2,2-dimethylcyclopropanecarboxylic acid (flumethrin acid, BNF 5533A)
(VI) flumethrin acid glucuronide

In a fourth study whole-body autoradiography was used to study the distribution of total radioactivity during 48 hours after single oral doses of [U-*chlorophenyl*-^{14}C]flumethrin (Klein, 1993a). The author reported slow or delayed absorption and only slowly decreasing residues in the organs and tissues, with the highest concentration in the liver. Towards the end of the experiment the highest residues were in the organs of excretion.

A fifth study investigated the biokinetic behaviour and metabolism of flumethrin in rats after single oral doses or after oral dosing for 7 consecutive days with flumethrin labelled with ^{14}C in the chlorophenyl ring. In a separate experiment a single dose was also administered duodenally (Klein, 1993b). Again, radioactivity was reported to be only partially absorbed from the intestine with 77-88% slowly eliminated,(68% in the faeces, 2% in the urine). The faeces were the source of residues for identification. The highest concentrations of ^{14}C were found in the plasma and the lowest in the brain. Nine to 20% of the dose was found in non-intestinal tissues. Multiple doses demonstrated the accumulation of residues. The only two compounds identified in the faeces were flumethrin and BNF 5533A (flumethrin acid, V in Figure 1). The ratio of flumethrin to BNF 5533A was 53:16 in males and 24:30 in females.

Cattle. Three studies were reported. The first two were of the distribution of radioactivity, one from a topical application and the second from i.v. administration. The third was a continuation of the i.v. study for the identification and quantification of the residues.

In the first study (Cameron and Phillips, 1986), in accordance with GLP principles, a single dose of 938.5 mg (6.56 mCi) of formulated [U-*fluorophenyl*-^{14}C]flumethrin was applied by syringe to a 60 cm x 15 cm section along the spine of a 530 kg lactating Friesian dairy cow (1.8 mg/kg bw). The GAP rates for pour-on applications are 1.8-3.6 mg/kg bw (generally $\leq$2.5 mg/kg bw) and for spray applications approximately 1.5-2 mg ai/kg bw. Milking was by machine twice daily, and blood samples were taken at frequent intervals until slaughter 48 hours after treatment. Samples were taken of liver, kidney, perirenal fat, subcutaneous fat (beneath and away from the dose area) and muscle at different sites, as well as of bile and bladder urine. All samples were stored at -20°C until analysis for total radioactivity by liquid scintillation counting.

Residues in the plasma peaked at 6.3 ng flumethrin equivalents/ml after 23 hours, slowly decreasing thereafter. No radioactivity was detected in the milk on the first day after treatment but 1, 3 and 2 ng equivalents/ml were reported at the first and second milkings on the second day and the first milking on the third day respectively. The levels of radioactivity in other samples are shown in Table 1.

Table 1. Total radioactivity in tissues and fluids of a lactating cow 48 hours after topical administration of fluorophenyl-labelled [^{14}C]flumethrin at 1.8 mg/kg bw (Cameron and Phillips, 1986).

Sample	^{14}C, ng flumethrin equivalent/g or ml
Whole blood	2
Plasma	4
Liver	9
Kidney	10
Renal fat	2
Subcutaneous fat	
below dose area	0 (<7.7 ng equiv./g)
away from dose area	0 (<7.7 ng equiv./g)
Skeletal muscle	
fore leg	0(<3.9 ng equiv./g)
rump	0(<3.9 ng equiv./g)
dorsal	0(<3.9 ng equiv./g)
cheek	1(3.9 ng equiv./g limit of determination)
Bile	70
Bladder urine	281
Application area (surface wash and solubilized skin)	4.7 mCi = 71.5% of administered dose

In the second study, also in accordance with GLP, [U-*chlorophenyl*-^{14}C]flumethrin (98.2% pure by TLC, 96.3% by HPLC) was formulated as a solution (specific activity 9 Ì Ci/mg) and administered as a single i.v. dose into the jugular veins of both a lactating cow (545 kg) and a steer (340 kg) at a nominal rate of 1 mg ai/kg bw (Gifford and Dunsire, 1994). The integrity of the dose solution was confirmed by TLC after dosing. Urine, milk and faeces samples were taken until slaughter 8 hours after treatment and milk also just before dosing. Tissue samples were taken at slaughter and all

samples were transported under dry ice and stored at -20°C until analysed. Total ^{14}C was determined by liquid scintillation.

The cumulative recovery of ^{14}C is shown as a proportion of the administered dose is summarized in Table 2 and as flumethrin equivalents/kg in the milk, blood and tissues in Table 3.

Table 2. Recovery of radioactivity from [^{14}C]flumethrin 8 hours after administration of a single intravenous dose to cattle at 1 mg ai/kg bw (Gifford and Dunsire, 1994).

Sample	^{14}C, % of injected	
	Dairy cow	Steer
Urine	4	8
Faeces	0.03	0.35
Milk	0.32	NA
Tissues		
liver	21.13	4.4
kidney	0.22	0.28
muscle	7.13*	6*
fat	3.1*	2.5*
Total	31.6	13.2
Total	35.9	21.5

* Assumes muscle and fat account for 30% and 20% of body weight respectively.

Table 3. Residues of ^{14}C as flumethrin equivalents 8 hours after administration of a single intravenous dose of [^{14}C]flumethrin to cattle at 1 mg ai/kg bw (Gifford and Dunsire, 1994).

Sample	^{14}C as flumethrin, mg/kg	
	Dairy cow	Steer
Milk	0.3	NA
Liver	13	3.4
Kidney	0.9	1.4
Muscle - loin	0.19	0.18
flank	0.25	0.19
round	0.30	0.23
Fat - subcutaneous	0.17	0.24
omental	0.37	0.19
Whole blood	1.5	1.8
Plasma	2.2	2.8

The third study (Klein, 1995), also in accordance with GLP, was a continuation of the 1994 study of Gifford and Dunsire described above, with the objective of identifying and quantifying the residues in the edible tissues and milk. Identifications were based on the characterization and fractionation of urinary extracts by HPLC, followed by GC-MS and NMR spectrometry. One HPLC

fraction was shown by MS and NMR to contain the glucuronic acid conjugate of metabolite BNF 5533A. The identification was confirmed by the detection of BNF 5533A in glucuronidase/arylsulfatase hydrolysates of the conjugate. Another major urinary component was shown by GC-MS and NMR after methylation to be unconjugated BNF 5533A.

Samples of loin, flank and round muscle and of omental and subcutaneous fat were composited for each animal before extraction for analysis. Tissues were extracted with acetonitrile/water, and the extracts concentrated and partitioned with n-heptane. The heptane fractions were concentrated, taken up in acetonitrile and analysed by HPLC. The aqueous fractions were diluted with water, adjusted to pH 3 (except liver extracts) and partitioned with acetonitrile which was concentrated for HPLC. Milk was extracted with methanol and the residues were partitioned into heptane: only the heptane fraction was analysed as it contained 89% of the radioactivity. Residues were quantified by HPLC with integration of ^{14}C signals, and identified by comparison with reference standards in two HPLC systems. Table 4 shows the efficiencies of extraction of from the milk and tissues and the levels of ^{14}C found. Table 5 shows the levels and percentages of the identified compounds.

Table 4. Concentrations and extractable proportions of total radioactivity in milk and tissues of cattle 8 hours after i.v. administration of [^{14}C]flumethrin at 1 mg/kg bw (Klein, 1995).

Sample	^{14}C			
	Dairy Cow		Steer	
	Flumethrin equivalent, mg/kg	Extractable, %	Flumethrin equivalent, mg/kg	Extractable, %
Liver	13	96	3.4	92
Kidney	0.9	92	1.4	90
Muscle	0.25	87	0.2	87
Fat	0.27	78	0.22	88
Milk	0.34	89	-	-

Table 5. Distribution of ^{14}C in flumethrin and metabolites in milk and tissues of a lactating dairy cow and steer 8 hours after i.v. administration of [^{14}C]flumethrin at 1 mg/kg bw (Klein, 1995).

Sample	Component or fraction	Total % and mg/kg as flumethrin in sample

		Flumethrin	BNF 5533A	BNF 5533A glucuronide	Unknown	
Liver (cow)	%	87.1	7.0	1.0		95.1
	mg/kg	11.31	0.91	0.13		12.4
Liver (steer)	%	28.9	39.9	7.2		76
	mg/kg	0.97	1.34	0.24		2.6
Kidney (cow)	%	35.1	47.4	5.7		88.2
	mg/kg	0.31	0.42	0.05		0.8
Kidney (steer)	%	15.5	46.5	24.8		86.8
	mg/kg	0.22	0.66	0.35		1.2
Muscle (cow)	%	29	57.5			86.5
	mg/kg	0.07	0.14			0.2
Muscle (steer)	%	35.9	51.1			87
	mg/kg	0.07	0.1			0.2
Fat (cow)	%	23.8	54.5			78.3
	mg/kg	0.06	0.15			0.2

Fat (steer)	%	27.8	59.8			87.6
	mg/kg	0.06	0.13			0.2
Milk (cow)	%	67.9			11.5	67.9
	mg/kg	0.23			0.04	0.2

METHODS OF RESIDUE ANALYSIS

Analytical methods

A multi-residue analytical method used by Australian national authorities for the determination of pyrethroids including flumethrin in animal fat, and methods for the determination of flumethrin and in some cases also its metabolite BNF 5533A (flumethrin acid) in cattle tissues and milk, and for the determination of flumethrin in sheep tissues, honey and honey wax were reported.

Multi-residue methods

The Australian multi-residue Method 2A for the determination of pyrethroids in animal fat (Webster *et al*., 1996) was used in the supervised trials carried out by the Queensland Department of Primary Industries (Queensland and New South Wales, 1996). It is based on and very similar to published methods (Mills *et al*., 1963; EPA, 1980) for organochlorine pesticides. In Method 2A finely sliced fat, is rendered, dissolved in hexane, and partitioned with acetonitrile. The acetonitrile is diluted with water and the residues partitioned into hexane. The extract is concentrated, cleaned up on a Florisil column eluted with 10% ethyl ether in hexane, and the residue determined after concentration by GLC with an ECD. The method calls for immediate storage of rendered fat samples at -40°C until analysis, although the Australian residue reports did not specify how this was done or the period of storage.

The limit of "detection" of flumethrin was reported as 0.01 mg/kg, with a mean recovery of 87% (n=5, s.d. 6.9) at 0.02 mg/kg, the lowest validated fortification level. Recoveries were similar (92%) at 0.05 mg/kg. Sample chromatograms were not provided for an independent estimate of the limit of detection or determination.

Specific methods

Cattle. The earlier methods were for the determination of flumethrin in milk. Riegner (1986a) described a method for the determination of flumethrin in cows milk which involved extraction with water/acetonitrile (1:4), clean-up on a silica gel column, and determination by HPLC with a 254 nm UV detector. Recoveries of 66 and 77% and a limit of determination of 0.005 mg/kg were reported (sample chromatograms suggest that 0.1 mg/kg might be more realistic). The reported limit of detection was 0.002 mg/kg. Saito (1988) described a method for flumethrin in milk and plasma which consisted in extraction with hexane/water (2:1), concentration, partitioning between hexane and acetonitrile, clean-up on a Sep-Pak cartridge, and HPLC determination. Recoveries of 92.4% were reported for milk fortified at 0.5 mg/kg and the limit of "detection" was reported to be 0.03 mg/kg, but this could not be confirmed in the absence of sample chromatograms.

In one of the first methods reported for flumethrin in cattle tissues (Werthmann and Kaiser, 1980), an acetonitrile extract of minced tissues is cleaned up on a silica column and the dichloromethane eluate is concentrated and analysed by reversed-phase HPLC with UV detection at 266 nm. A "limit of detection" of 0.05 mg/kg was reported, with 80-90% recoveries at 0.08 mg/kg, but these figures could not be confirmed with the information provided.

Maasfeld (1989) described a method for the determination of flumethrin in cattle tissues and milk. Tissues are homogenized with acetonitrile, and the homogenate is partitioned successively with hexane (which is discarded) and dichloromethane before clean-up by silica gel chromatography. Milk is extracted with 1:4 water/acetonitrile (as in the Riegner method) and partitioned with dichloromethane. The extract is cleaned up on silica gel (elution with 55:45 hexane/dichloromethane). Determination is by HPLC with UV detection at 266 nm. Recoveries were generally about 80% or better from tissues at 0.01 mg/kg fortification levels and from milk at 0.005 mg/kg. The limit of detection (based on noise levels) was estimated to be approximately 0.004 mg/kg for tissues and 0.001 mg/kg for milk. The limit of determination was reported to be 0.01 mg/kg for tissues and 0.005 mg/kg for milk. Sample chromatograms support those estimates, at least for the author's laboratory. Permethrin, cypermethrin and cyfluthrin do not interfere.

Three more recent methods (Bohm and Paul, 1994a,b,c) for flumethrin in tissues and milk and for flumethrin acid (BNF 5533A) in tissues are based on the method of Maasfeld. Tissues are analysed in the same way, except that fat samples are ground and mixed with sea sand before extraction with acetonitrile. Milk solids are removed by the addition of acetone and centrifugation before extraction with dichloromethane, partitioning into acetonitrile and washing with hexane (which is discarded).

The determination of flumethrin acid in tissues is similar to that of flumethrin, except that extraction is with 8:1 acetonitrile/0.1% phosphoric acid instead of acetonitrile, and the silica gel column treatment is followed by further clean-up on a C-18 solid-phase extraction column.

The mean recoveries of flumethrin were 80 to 90% from tissues at 0.01 mg/kg fortification levels and 86% from milk at 0.005 mg/kg. A limit of determination of 0.01 mg/kg was reported for both flumethrin and flumethrin acid in tissues. Sample chromatograms suggest that this limit may be possible in the authors' laboratory for flumethrin and perhaps for flumethrin acid, except in kidney and liver where it is questionable. The reported limit of detection for flumethrin was 0.002 mg/kg and for flumethrin acid 0.002 mg/kg in kidney and muscle but 0.004 mg/kg in liver and fat. A limit of determination of 0.005 mg/kg was reported for flumethrin in milk and sample chromatograms suggest that this is possible in the authors' laboratory. The limit of detection was reported as 0.001 mg/k.

Two recent HPLC methods similar to those of Bohm and Paul have been reported for the determination of flumethrin and flumethrin acid in cattle tissues (Krebber, 1994a) and milk (Krebber, 1994b). In the tissue method flumethrin and flumethrin acid are extracted together from homogenates by the procedure used in the Bohm and Paul method for flumethrin acid (extraction with acetonitrile/phosphoric acid). The compounds are separated on a silica gel cartridge by eluting flumethrin with dichloromethane/hexane and flumethrin acid with dichloromethane/methanol. As in the Bohm and Paul method the flumethrin acid fraction is further cleaned up on a C-18 solid-phase cartridge and both fractions are analysed by HPLC.

Mean recoveries of flumethrin at 0.01 mg/kg were 92-104% from tissues except fat, and 68% from fat. Mean recoveries of flumethrin acid at 0.02 mg/kg from tissues were 87 to 110%. The limits of determination were reported to be 0.01 mg/kg and 0.02 mg/kg for flumethrin and flumethrin acid respectively. No response for flumethrin was seen in controls, but a limit of detection of 0.005 mg/kg was reported for flumethrin acid. Sample chromatograms were consistent with the reported limit of determination for flumethrin but were not as conclusive for flumethrin acid.

The Krebber (1994b) method for the determination of flumethrin and flumethrin acid in milk is essentially the same as that for tissues. At 0.005 and 0.2 mg/kg fortification levels the mean recoveries of flumethrin were 73 and 85% respectively and of flumethrin acid 102 and 90%. The limits of

determination were reported to be 0.005 and 0.01 mg/kg for flumethrin and flumethrin acid respectively. Again, sample chromatograms were consistent with the reported limit of determination for flumethrin, but were less conclusive for flumethrin acid.

Sheep. Separate methods have been reported for the determination of flumethrin, but not flumethrin acid, in sheep. The oldest of the methods provided to the Meeting was for the determination of flumethrin in sheep milk (Palermo, 1987). It involves extraction with a 1:1:2 solution of petroleum ether(PE)/acetone/acetonitrile, discarding the PE, extraction of the aqueous layer with chloroform, concentration, dissolution in PE and clean-up on a silica gel column before determination by HPLC with UV detection at 266 nm. The mean recovery was only 66% and a limit of "detection" of 0.01 mg/kg was reported. No sample chromatograms or details of recovery experiments were provided.

The method reported by Inveresk (1996) as "the method for flumethrin determination in sheep tissues" is a modification of method 00366 developed for the determination of flumethrin in rat serum (Krebber, 1994c) and later modified for serum analyses (Krebber, 1995).

In the original method serum was extracted with ethyl acetate, the extract was cleaned up on a silica gel column, eluted with n-hexane/dichloromethane (55:45), concentrated, taken up into acetonitrile and determined by HPLC with UV detection. The 1995 modification for serum consisted in acidification of serum in water with phosphoric acid and elution from an "Extrelut" cartridge with ethyl acetate before the silica column clean-up.

For the analysis of sheep tissues extraction with acetonitrile is followed by partitioning with hexane, silica gel column clean-up and HPLC determination (Inveresk, 1996). Only a summary of the modified method for sheep was provided. From the summary, the modified method seems similar to the method described by Maasfeld (1989) for cattle tissues, although the summary does not indicate whether tissues are ground before extraction. Limits of detection and determination of 0.01 mg/kg and 0.02 mg/kg were reported, with recoveries of 88, 82, 115 and 99% from liver, kidney, muscle and fat respectively at 0.02 mg/kg. The lack of details and sample chromatograms precluded independent confirmation of the reported limits.

Honey and wax. In the method of Riegner (1986b) for the determination of flumethrin in honey and beeswax honey is extracted with a mixture of toluene, dichloromethane and methanol (5:4:1), the solvent is evaporated and the residue taken up in 1:1 ethyl acetate/cyclohexane for clean-up by gel permeation followed by silica gel column chromatography. Wax is melted, dissolved in hot 2-propanol, and precipitated with methanol/water. The extract is further purified by partitioning between water and 1:1 ethyl acetate/cyclohexane, the solvent is evaporated and the residue taken up in acetonitrile, which is washed with hexane. The acetonitrile is evaporated and the residue taken up in toluene for silica gel chromatography. Determination is by HPLC with UV detection at 254 nm.

The mean recoveries were about 63% from honey at 0.003 to 0.004 mg/kg and from wax at 0.03 to 0.1 mg/kg. The "lower practical working range" was reported to be 0.002 mg/kg for honey and 0.025 mg/kg for wax. Sample chromatograms indicated that these levels were achievable in the author's laboratory.

Two more recent methods (Heukamp, 1993; Heukamp and Krebber, 1993) are very similar to and appear to be based on the Riegner (1986) method. Modifications include the use of an ultra sound bath for re-dissolving the residues from extracts which have been taken to dryness and of a variable wavelength detector, used at 266 nm, instead of the 254 nm detector. The reported mean recoveries from honey were 74% at 0.003 mg/kg, 87% at 0.013 mg/kg and 86% at 0.85 mg/kg, and from wax

60% at 0.026, 79% at 0.051, and 76% at 0.1 mg/kg. The reported limits of detection and determination were 0.001 and 0.003 mg/kg for honey and 0.02 and 0.026 mg/kg for wax. Sample chromatograms were consistent with these levels.

Stability of pesticide residues in stored analytical samples

No substantive studies of storage stability were provided. In one supervised trial milk fortified with 0.037 mg/kg flumethrin was stored for 40 days at -18°C and analysed after 10 and 40 days (Dorn and Maasfeld, 1989b). Since the recoveries, 74 and 77% respectively, were normal for the method the authors concluded that flumethrin was stable in milk under the conditions of storage.

Residue Definition

Although the metabolite BNF 5533A (flumethrin acid) was found in metabolism studies to occur at 1 to 1.5 times the level of flumethrin in cattle tissues, it was not reported in milk. If flumethrin is of significantly greater toxicological concern than the metabolite, if it is observed that it may occur in tissues at comparable levels to the metabolite, that only flumethrin was reported in milk and is the residue of concern in honey, flumethrin *per se* is a suitable indicator residue for regulatory purposes. Other issues relevant to expressing MRLs for meat are discussed in the appraisal.

USE PATTERN

Flumethrin is applied to cattle (including lactating cows), sheep, goats, horses and dogs as a spray, dip or pour-on treatment for the control of mange, mites, lice, biting lice and ticks. The only information provided on nationally approved uses (GAP) was on an Australian 75 g ai/l formulation for dips or sprays for cattle and horses, an Australian 1% ai pour-on for cattle and a UK 6% EC formulation for sheep dipping, all supported by labels. The submission made further general reference to a 6% EC for sprays or dips for sheep and dip for goats (Inveresk, 1996) but no labels, countries, withdrawal periods or treatment intervals were provided. The Inveresk reference to the 6% EC dip reported application rates of 44-66 mg ai/l to sheep (after milking if lactating) and 30-48 mg ai/l to goats. The application rate for sheep is consistent with the UK label. Flumethrin-impregnated plastic strips (3.6 mg/strip) are also available for the control of Varroa in bee colonies, 2 to 4 strips per chamber. This use is approved in the UK (Inveresk, 1996), but again labels and other details were not provided.

UK GAP for sheep. The 6% EC formulation for dipping sheep for scab and tick control is used at a rate of 1l product/900-1300 l water (46-67 mg ai/l). Sheep are dipped for at least one minute with total immersion (including the head and ears) at least twice. Re-dipping after 14 days is recommended if scabies is confirmed. A 3-month interval before shearing is recommended. No withdrawal period is required before consumption of tissues or milk, but lactating sheep must be dipped after milking.

Australian GAP for cattle and horses is summarized in Table 6.

Table 6. Australian uses of flumethrin on farm animals.

Application			Treatment interval, days	Withholding period, days	Notes
Method	Solution concn., g/l	Rate, mg/kg bw			
Dip and spray formulation, 75 g ai/l (cattle and horses)					
Plunge dip	75		10-21 (pest-dependent)	cattle 0 horses 1	Replenish at same concn.[1] before 1/4 volume loss; 20-25 animals used as stirrers (need re-dipping).
Recirculating spray	75		same	same	Replenish same concn. every 250 l vol. loss or after 1000 l, then after 500 l, then after every 250 l loss. Max. 750 adults before emptying and recharging.
Constant replenishment spray	75		same	same	Replenish at same concn. Max. 750 adults before emptying and recharging.
Hand spray or non-recirculating spray	75	1.2-1.5 (assuming 500 kg animal)	same	same	Minimum 8-10 l/animal
Pour-on Formulation 10 g/l (Cattle)					
Pour-on	10 g/l	2.3* 1.8-3.6** 1.5-2.5*** 1.5-2.2****	14-42[3]	"Nil"[2]	Applied along mid-line of back from front of shoulder to tail. * ≤ 150 kg; (33 ml product); ** 151-300 kg; (55 ml product); *** 301-500 kg; (75 ml product); **** 501-750 kg; (112.5 ml product

[1]It has been demonstrated over 40-70 week periods of practical cattle dipping that replenishments of flumethrin EC plunge dips at the initial rate maintain a concentration near the target without the need for replenishment at rates higher than the initial charge. For example an initial 50 mg/l will be stabilized between 40 and 50 mg/l (Terblanche, 1980c)

[2]A 56-day withholding period is "suggested" for exports and may be required by some meat processors, but is not a statutory requirement

[3]Implied treatment interval ("Control can be attained"). Pest-dependent for ticks. Controls Buffalo fly up to 10 days

RESIDUES RESULTING FROM SUPERVISED TRIALS

Supervised trials data were available on residues of flumethrin in the milk of cattle, sheep and goats and the tissues of cattle and sheep, and for residues of the metabolite flumethrin acid (BFN 553A) in the tissues of cattle. Data were also provided on residues in honey and beeswax from supervised trials in bee hives.

Cattle (Tables 7-10). Supervised trials were reported from Australia, South Africa, Germany and Japan, with applications by dipping, spraying and pour-on. The Meeting was informed of additional Australian studies to be completed in 1996.

Table 7. Flumethrin residues in cattle fat from 1994-95 Australian trials. Australian government submission.
Table 8. Flumethrin residues in cattle tissues (including older Australian trials). Submissions by the manufacturer.
Table 9. Flumethrin acid (metabolite BNF 5533A) residues in cattle tissues. Submission by the manufacturer (trials conducted in accordance with GLP).
Table 10. Flumethrin residues in cattle milk. Submission by the manufacturer.

Table 7. Flumethrin residues in loin (subcutaneous) and renal fat of cattle from 1994-95 supervised trials in Australia (Webster *et al*., 1996; Queensland and New South Wales, 1996)

Pre-slaughter interval, days	Sample	Residues, mg/kg, after indicated treatment			
Plunge Dips[1]		Treatment[2]			
		D1	D2	D3	D4
2	Loin fat	<0.005	--	<0.005	<0.005
		<0.005	--	<0.005	<0.005
		0.041	--	<0.005	<0.005
	Renal fat	<0.005	--	<0.005	<0.005
		<0.005	--	<0.005	<0.005
		0.047	--	<0.005	<0.005
4	Loin fat	<0.005 (3)[3]	<0.005 (3)	<0.005 (3)	<0.005 (3)
	Renal fat	<0.005 (3)	<0.005(2) 0.006	<0.005 (3)	<0.005 (3)
7	Loin fat	<0.005 (3)	<0.005 (3)	<0.005 (3)	<0.005 (3)
	Renal fat	<0.005 (3)	<0.005 (3)	<0.005 (3)	<0.005 (3)
15	Loin fat	<0.005 (3)	<0.005 (3)	<0.005 (3)	<0.005 (3)
	Renal fat	<0.005	<0.005	<0.005	<0.005
		0.008	<0.005	<0.005	<0.005
		<0.005	<0.005	<0.005	<0.005
21	Loin fat	All 12 residues <0.005			
	Renal fat	All 12 residues <0.005			
30	Loin fat	<0.005	<0.005	<0.005	0.009
		<0.005	<0.005	<0.005	<0.005
		<0.005	<0.005	<0.005	<0.005
	Renal fat	<0.005	<0.005	0.011	0.013
		<0.005	<0.005	<0.005	<0.005
		<0.005	<0.005	<0.005	<0.005

Pour-on applications[4]		Treatment	
		P1	P2
2	Loin fat	<0.005	0.015
		<0.005	0.014
		<0.005	0.013
	Renal fat	<0.005	0.04
		<0.005	0.034
		<0.005	0.023
4	Loin fat	0.023	0.028
		<0.005	0.025
		0.029 [0.04][5]	0.023
	Renal fat	0.032	0.058
		<0.005	0.022
		0.026 [0.04][5]	0.035
7	Loin fat	0.013 [0.015][5]	0.031
		0.011	0.018
		0.008	0.019
	Renal fat	0.015 [0.015][5]	0.037
		0.020	0.036
		0.015	0.022
10	Loin fat	<0.005	0.022
		0.014	0.022
		0.009	0.029
	Renal fat	<0.005	0.044
		0.019	0.038
		0.014	0.097
15	Loin fat	0.011	0.052
		0.007	0.020
		0.011	0.020
	Renal fat	0.012	0.14 (confirmed)
		0.014	0.038
		0.034	0.035
21	Loin fat	0.006	0.017
		0.008	0.011
		0.040 [0.06][5]	0.016
		0.017 [0.025][5]	0.015
		0.010	0.022
		0.029	0.019
	Renal fat	0.014	0.036
		0.021	0.026
		0.11 [0.06][5]	0.049
		0.024 [0.025][5]	0.026
		0.014	0.054
		0.042	0.033
30	Loin fat	0.020	0.014
		0.008	0.017
		0.008	0.020
	Renal fat	0.027	0.027
		0.029	0.030
		0.011	0.027
Pour-on applications[4]		Treatment	
		P1	P2
45	Loin fat	<0.005	0.023
		<0.005	0.012

		0.009	0.029
	Renal fat	0.018	0.051
		0.024	0.028
		0.020	0.044

[1]Generally 3 animals for each treatment and pre-slaughter interval, each sample from a different fat depth and the renal fat values corresponding successively to the loin fat values.

[2]D1: dipped once according to GAP.
D2: dipped twice, but the animals were also used to stir the dip. Complied with GAP.
D3: dipped twice. Not strictly GAP because retreatment interval was 3 days and GAP minimum is 10 days.
D4: dipped twice, but the animals were used as stirrers for both dips. Not strictly GAP because the retreatment interval
was 7 days instead of minimum of 14 days implied by label.
P1: one application according to GAP.
P2: two applicatons at 7-day interval. GAP interval is 14 days.

[3]Numbers in parentheses are the numbers of samples with the same residues.

[4]At GAP rate of 10 g ai/l and according to label instructions requiring 1.5 to 3.6 mg/kg bw, according to the weight of
the animals (see Table 6).

[5]Values in square brackets are means of replicate analyses of fat from core samples of cartons of frozen product (same
carcases) as distinct from loin and renal fat samples taken at slaughter.

Dip concentrations were checked by analysis before treatment. The multi-residue GLC method used is described in "Analytical methods". Although the reported limit of detection was 0.01 mg/kg, undetectable residues are recorded as <0.005 mg/kg. No information was provided on the length of time from slaughter to analysis, although the time from the start of the study until the final report was less than 14 months. The protocol called for sample storage at -40°C.

Figure 2 shows the variation of residues of flumethrin in loin and renal fat with time after one or two pour-on applications at GAP rates.

Figure 2. Average residues of flumethrin in loin (subcutaneous) fat and renal fat from one or two pour-on applications to cattle at GAP rates (Queensland and New South Wales, 1996).

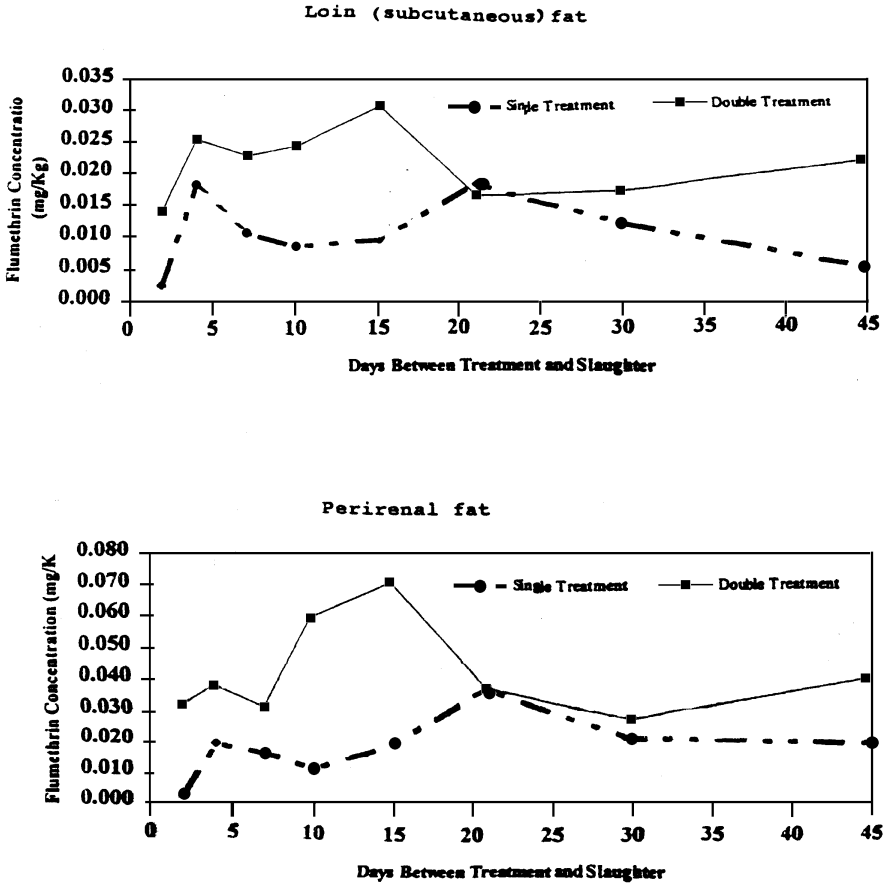


Table 8. Residues of flumethrin in cattle tissues from supervised trials.

Country, year, Formln.	Application			No. of cattle	Pre-slghtr. (days)	Flumethrin, mg/kg, in				Ref[1]
	Concn., ai	No. & (days intvl.)	mg/kg bw			fat[3]	liver	muscle	kidney	
Dip										
S. Africa 1980 7.5% EC (dip wash)	75 mg/l No GAP	4 (14)		3	3	<0.1(3)	<0.1(3)	<0.1(3)	<0.1(3)	40
				3	7	<0.1(3)	<0.1(3)	<0.1(3)	<0.1(3)	
				3	14	<0.1(3)	<0.1(3)	<0.1(3)	<0.1(3)	

Australia 1983 7.5% (plunge dips)	67 mg/l (within GAP)	1		3	1	0.007 <0.005(2)	<0.005(3)	0.01 <0.005(2)	<0.005(3)	36
				3	3	<0.005(3)	<0.005(3)	<0.005(3)	<0.005(3)	
Pour-on										
Germany 1989 1% pour-on	10 g/l No GAP	1	2	2[2]	1	0.05(2)	≤0.01, 0.03	nd, <0.01 nd = <0.01 = limit determ.	nd, <0.01	28
				2	4	0.07, 0.05	nd,0.013	nd,0.01	nd,0.01	
				2	7	0.07, 0.08	nd(2)	nd(2)	nd(2)	
				2	14	0.06(2)	nd(2)	nd(2)	nd(2)	
				2	21	0.03, 0.08	nd(2)	nd(2)	nd(2)	
				2	28	0.07, 0.06	nd(2)	nd(2)	nd(2)	
S. Africa 1984 0.5% pour-on No GAP	5 g/l	6 (7)	1.2	3	0.5	<0.05(2), 0.07	<0.05(3)	<0.05(3)	<0.05(3)	25
S. Africa 1984 1% pour-on No GAP	10 g/l	6 (7)	1.2	2	0.5	<0.05(2)	<0.05(2)	<0.05(2)	<0.05(2)	26
Australia 1984 0.5% pour-on	5 g/l (within GAP)	1	1 GAP?	3 3	1 3	max./av. 0.01/ 0.007 0.005/ <0.005	max./av. <0.005(3) 0.01/ <0.007	max./av. 0.005/ <0.005 <0.005(3)	max./av. <0.005(3) <0.005(3)	34
	5 g/l	1	2 (GAP)	3 3	1 3	0.005/ <0.005 0.005/ <0.005	<0.005(3) <0.005(3)	<0.005(3) <0.005(3)	<0.005(3) <0.005(3)	
	5 g/l	1	4	3 3	1 3	0.13/ <0.09 0.005/ <0.005	0.01/ <0.007 <0.005(3)	0.02/ <0.01 <0.005(3)	0.005/ <0.005 <0.005(3)	
1% pour-on	10 g/l (GAP)	1	1 (within GAP)	3 3	1 3	0.01/ <0.007 0.025/ <0.01	<0.005(3) <0.005(3)	0.005/ <0.005 <0.005(3)	0.01/ <0.007 <0.005(3)	34
	10 g/l	1	2 (GAP)	3 3	1 3	<0.005(3) 0.005/ <0.005	<0.005(3) <0.005(3)	<0.005(3) <0.005(3)	<0.005(3) <0.005(3)	
	10 g/l	1	4	3 3	1 3	0.015/ <0.01 0.055/ <0.02	0.005(3) 0.005/ <0.005	0.005/ <0.005 <0.005(3)	<0.005(3) 0.01/ <0.007	
	Median for ref. 34 (N=36):				GAP treatments All treatments	<0.005 0.005	<0.005	<0.005	<0.005	

Spray										
S. Africa 1984 6%EC	30 mg/l No GAP	4 14		2	3	<0.05(3)	<0.05(3)	<0.05(3)	<0.05(3)	24
				2	7	<0.05(3)	<0.05(3)	<0.05(3)	<0.05(3)	
				2	14	<0.05(3)	<0.05(3)	<0.05(3)	<0.05(3)	
Australia 1981 7.5% EC	50 mg/l (2/3 max. GAP)	1		3	1	<0.05	<0.05	<0.05	<0.05	32
	50	1		3	3	<0.05	<0.05	<0.05	<0.05	
	100 (1.3X GAP)	1		3	1	<0.05	<0.05	<0.05	<0.05	
	100	1		3	3	<0.05	<0.05	<0.05	<0.05	
	200	1		3	1	<0.05	<0.05	<0.05	<0.05	
	200	1		3	3	<0.05	<0.05	<0.05	<0.05	
Australia 1981 7.5% EC + 16% coumaphos	100	1		3 3	1 3	<0.05 <0.05	<0.05 <0.05	<0.05 <0.05	<0.05 <0.05	32

[1]Numbers correspond to tab numbers in 1996 Bayer submission, Vol. III:
24. Amelsfoort, 1984a; 25. Amelsfoort, 1984b; 26.Amelsfoort, 1984c; 28. Dorn and Maasfeld, 1989a; 32. Hopkins and Lindsay, 1981; 34. Lindsay and Hopkins, 1984; 36. Lindsay, 1983b; 40. Terblanche, 1980a.
[2]Duplicate analyses on each cow. Results are means of duplicates.
[3]Reference 40 fat was described as perirenal fat, references 28 and 36 as "fat" and the rest "minced fat".

Table 9. Residues of flumethrin acid (BNF 5533A) in cattle tissues from supervised trials with a 1% pour-on flumethrin formulation, Germany, 1994 (Tesch and Doberschütz, 1994).

Treatment	No. of cattle	Pre-slaughter (days)	Flumethrin acid (BNF 5533A), mg/kg[1]			
			fat (suet)[2]	liver	muscle	kidney
2 x 10 g ai/l, 10 days apart (2 mg/kg bw)	6	1	0.03 0.02 (2) <0.01 (2) <0.004	0.05 0.04 0.03 0.02 <0.01	0.01 <0.01(4) <0.002	0.03(2) 0.02(2) <0.01 (2)
	6	2	0.04 0.02 (3) 0.013 0.01	0.06 0.05 0.04 0.03(2) 0.02	0.01 <0.01(3) <0.002(2)	0.05 0.03 0.02 0.01(3)
	6	4	<0.004(6)	0.02 0.01(2) <0.01	<0.01(3) <0.002(3)	0.03 <0.01(5)

Treatment	No. of cattle	Pre-slaughter (days)	Flumethrin acid (BNF 5533A), mg/kg[1]			
			fat (suet)[2]	liver	muscle	kidney
				<0.002(2)		
	6	7	0.02 <0.01 <0.004(4)	0.01 (2) <0.004 (4)	<0.01 <0.002(5)	0.02 <0.01 <0.002(4)
	6	21	<0.004(6)	<0.004(6)	<0.002(6)	<0.002(6)
	6	35	<0.004(6)	<0.004(6)	<0.002(6)	<0.002(6)

[1]Limit of detection 0.002 mg/kg in kidney and muscle and 0.004 mg/kg in liver and fat; limit of determination 0.01 mg/kg in all tissues.

[2]Suet = loin or kidney fat

Table 10. Residues of flumethrin in cattle milk from supervised trials with pour-on and spray applications.

Country, year formln.	Application			No. of cattle	Flumethrin, mg/kg, at intervals after last application[3]						Ref[1]
	Concn., ai	No. & (days intvl.)	mg/kg bw		Hours	Days					
					4	2	4	7	10	10	
Pour-on											
Germany 1989 1% Pour-on	10 g/l (withers to tail base)	2 (14)	2	5	<0.005 (4h) 0.03 M[2] 0.07 E	0.06	0.04	0.01	0.006		29
Germany 1989 1% Pour-on	10 g/l (hip pt. to tail base)	2 (14)	2	5	0.005 (4 h) 0.02 M 0.04 E	0.04	0.02	0.01	0.006		30
					Hours						
					6	8-9	18-19	22-25	30	≥42	
Australia 1984 1% pour-on	10 g/l	2 (3)	2[3]	3		9 h 0.01 <0.01 (2)[4] *	* From bulked milk: skim milk <0.01 mg/kg milk fat 0.14 mg/kg				31
Australia 1984 1% pour-on	10 g/l	1	1 (GAP)	3		9 h 0.01 <0.01 (2)		24 h 0.01 <0.01 (2)		72 h 0.01 <0.01 (2)	37

Country, year formln.	Application			No. of cattle	Flumethrin, mg/kg, at intervals after last application[3]						Ref[1]
	Concn., ai	No. & (days intvl.)	mg/kg bw		Hours	Days					
					4	2	4	7	10	10	
	10 g/l	1	2 (GAP)	3		0.04 0.01 <0.01		0.01 0.02 <0.01		0.01 0.01 <0.01	
	10 g/l	1	4	3		0.1 0.05 0.04		0.01 0.01 <0.01		0.01 0.03 0.01	
Australia 1984 0.5% pour-on	5 g/l	1	1 (GAP)	3		0.04 0.03 <0.01		0.03 0.04 0.01		0.02 0.01 <0.01	38
	5 g/l w/in GAP	1	2 (GAP)	3		0.03 0.01 0.01		0.03 <0.01 0.01		0.01 <0.01 <0.01	
	5 g/l	1	4	3		0.04 0.02 <0.01		0.01 0.01 0.02		<0.01 <0.01 0.01	
Japan 1987 1% pour-on	10 g/l	1	1	3		8 h <0.03 (3)		25 h <0.03 (3)			39
	10 g/l	3 (7)	1	3		1 h <0.03 (3) 8 h <0.03 (3)		25 h <0.03 (3)			
S. Africa 1984 0.5% Pour-on	5 g/l (withers to loins) [7]	6 (7)	1.2	3		8 h <0.05 (3)	19 h <0.05 (3)		30 h <0.05 (3)	42 h <0.05 (3) 66 h <0.05 (3)	27
Spray											
Australia 1984 7.5% EC	75 g/l (≤8 l/cow)	2 (3)		3		0.01 (2) <0.01					31
Australia 1981 7.5% EC (bulked samples)	75 g/l (10 l/cow) (GAP)	1		10		9 h <0.1		22 h <0.1			33

Country, year formln.	Application			No. of cattle	Flumethrin, mg/kg, at intervals after last application[3]						Ref[1]
	Concn., ai	No. & (days intvl.)	mg/kg bw		Hours	Days					
					4	2	4	7	10	10	
	150 (10 l/cow)	1		10		0.6		0.2			
Australia 1983 7.5% EC	100 (10 l/cow) (1.3 x GAP)	1		6		<0.01 (6)		<0.01 (6)			35
S. Africa 1980 7.5%[5]	75 g/l	4 (14)		9	<0.01 (9)		18 h <0.01 (9)	24 h <0.01 (9)		48 h <0.01 (9)	41

[1]Numbers correspond to tab numbers in 1996 Bayer submission, Vol. III:
27. Amelsfoort, 1984d; 29. Dorn and Maasfeld, 1989b; 30. Dorn and Maasfeld, 1989c; 31. Gyr, 1984; 33. Lindsay and Gyr, 1981; 35. Lindsay, 1983a; 37. Lindsay, 1984a; 38. Lindsay, 1984b; 39. Ohta, 1988; 41. Terblanche, 1980b.

[2]For refs. 29 and 30 the residues are the means of the means of the morning and evening milkings of the 5 cows, except on day 1 where morning (M) and evening (E) means are recorded separately because they differed significantly. After day 2 there were no significant differences between the morning and evening milkings.

[3]Calculated by authors from surface areas. Calculation from actual weights gives 1.4-1.6 mg/kg bw.

[4]Numbers in parentheses following the residues are the numbers of samples with those values.

[5]This trial is listed here as a spray. The submitted working paper summary lists it as a dip. The original report states that the cattle were "sprayed" with Bay L 6045 "dipwash" using a power spraypump.

Sheep and goats (Tables 11 and 12). Data were available from trials in Australia, South Africa, Italy and the UK, although information on national GAP was available only for Australia and the UK. Residues in sheep tissues and in sheep and goat milk are shown in Tables 11 and 12 respectively.

Table 11. Residues of flumethrin in sheep tissues from supervised trials with one application of pour-on or dip formulations.

Country, year formulation	Concn., ai	No. of sheep	Pre-slaughter (days)	Flumethrin, mg/kg, in				Ref.[5]
				fat[1]	liver	muscle	kidney	
Pour-on								
Australia 1986 Bay 1950 1% pour-on	10 g/l (2 mg/kg bw) mid back (wool parted)	3	0.5	<0.05 (3)[2]	<0.05 (3)	<0.05 (3)	<0.05 (3)	58

Country, year formulation	Concn., ai	No. of sheep	Pre-slaughter (days)	Flumethrin, mg/kg, in				Ref.[5]
				fat[1]	liver	muscle	kidney	
		3	14 hours	<0.05 (2) 0.06	<0.05 (3)	<0.05 (3)	<0.05 (3)	
		3	1	<0.05 (3)	<0.05 (3)	<0.05 (3)	<0.05 (3)	
		3	2	<0.05 (3)	<0.05 (3)	<0.05 (3)	<0.05 (3)	
		3	3	<0.05 (3)	<0.05 (3)	<0.05 (3)	<0.05 (3)	
South Africa 1990 1% pour-on[3]	10 (1 mg/kg bw)	2	1	0.007, 0.003	0.003, 0.002	<0.002 (2)	<0.002 (2)	60
		2	3	<0.002 (2)	<0.002, 0.004	<0.002 (2)	<0.002 (2)	
		2	5	0.06, 0.01	0.008, <0.002	0.009, 0.004	<0.002 (2)	
		2	7	0.02, 0.003	0.01, 0.002	0.002, 0.007	<0.002 92)	
		2	10	0.004, 0.008	<0.002, 0.005	<0.002, 0.004	<0.002 (2)	
Dip (Australia plunge dip)								
Australia 1983 Bay 6045 7.5% EC 1 min. dip	60 mg/l	2	1	0.005, 0.02	<0.005 (2)	<0.005 (2)	<0.005 (2)	59
		2	3	<0.005 (2)	<0.005 (2)	<0.005 (2)	<0.005 (2)	
	90 mg/l	2	1	<0.005, 0.04	<0.005 (2)	<0.005 (2)	<0.005 (2)	
		2	3	<0.005 (2)	<0.005 (2)	<0.005 (2)	<0.005 (2)	
Australia 1983 Bay 6045 6% SLC 1 min. dip	60 g/l	2	1	0.02, 0.03	<0.005 (2)	<0.005 (2)	<0.005 (2)	
		2	3	<0.005 (2)	<0.005 (2)	<0.005 (2)	<0.005 (2)	
	90 g/l	2	1	<0.005, 0.01	<0.005 (2)	<0.005 (2)	<0.005 (2)	
		2	3	<0.005 (2)	<0.005 (2)	<0.005 (2)	<0.005 (2)	
U.K. 1992 5.9% EC dip 1 min. dip	70 g/l	4	0.5	<0.01 (4) (omental) <0.01(3), 0.02/0.03 (subcut.)[4]	<0.01 (4)	<0.01 (4)	<0.01 (4)	61

Country, year formulation	Concn., ai	No. of sheep	Pre-slaughter (days)	Flumethrin, mg/kg, in				Ref.[5]
				fat[1]	liver	muscle	kidney	
		4	1	<0.01 (4) (omental) <0.01(3), 0.01/0.01 (subcut.)[4]	<0.01(3), 0.02/ <0.01[4]	<0.01 (4)	<0.01 (4)	

U.K. 1992 5.9% EC dip 1 min. dip		4	2	<0.01 (4) (omental) <0.01 (4) (subcut.)	<0.01 (4)	<0.01 (4)	<0.01 (4)	61
		4	4	<0.01 (4) (omental) <0.01(3), 0.01/0.02 (subcut.)[4]	<0.01(3), 0.01/0.01[4]	<0.01 (4)	<0.01 (4)	

[1]Unspecified except in UK
[2]Numbers in parentheses following the residues are the numbers of samples with those values.
[3]The report only states that applications were dermal. It was a pour-on formulation as indicated.
[4]Positive samples were re-analysed (the analyses separated by /).
[5]Numbers correspond to tab numbers in 1996 Bayer submission, Vol. IV:
58. Hopkins and Gyr, 1986; 59. Lindsay, 1983c. 60. Nieuwenhuis, 1990. 61. Redgrave, 1992.

Table 12. Residues of flumethrin in sheep and goat milk from supervised trials with 1 application of pour-on or spray formulation.

Species Country, year Formln.	Concn., ai & area treated	No. of Ani-mals	Flumethrin, mg/kg, at interval, hours, after last application								Ref.[3]
			8	12	18	24	36	48	60	72	
Pour-on											
Goats Australia 1984 1950 0.5% Pour-on	5 g/l (6 mg/kg bw) back mid-line	3		0.01 (2)[1] 0.02		0.01, 0.02, 0.04					IV,56
1950 1% Pour-on	10 g/l (4.6-6 mg/kg bw) back mid-line	3		<0.01 (3)		<0.01 (3)					
Sheep Australia 1986 1740 1% pour-on	10 g/l (2 mg/kg bw) back, neck to tail	6	<0.01 (3)		<0.01 (3)[2]	<0.01 (3)				<0.01 (3)	IV,57
1950 1% Pour-on	10 g/l (2 mg/kg	6	<0.01 (3)		<0.01 (3)[2]	<0.01 (3)				<0.02 (3)	

	bw) back, neck to tail										
Italy 1987 1% Pour-on	10 g/l (2 mg/kg bw)	5		<0.01 (5)		<0.01 (5)	<0.01 (5)	<0.01 (5)	<0.01 (5)	<0.01 (5)	III,21
Spray											
Italy 1987 6% EC	60 mg/l (2 mg/kg bw)	5		<0.01 (5)		<0.01 (5)	<0.01 (5)	<0.01 (5)	<0.01 (5)	<0.01 (5)	III,21

[1]Numbers in parentheses following the residues are the numbers of samples with those values.
[2]A different group of 3 animals was used for the 18 hour samples.
[3]Numbers correspond to tab numbers in 1996 Bayer submission, Vols. III & IV:
III,21. Palermo, 1987 IV,56. Griffin, 1984 IV,57. Griffin, 1986

Residues in honey and beeswax

Eleven supervised trials have been conducted in Germany, Switzerland and the UK with flumethrin used for mite control in honey-bee colonies by means of impregnated strips (0.5 mg flumethrin per cm^3, equivalent to 3.6 mg/strip, 4 strips/frame of 8-10 combs). Several seasonal periods were represented. The residues found in honey and beeswax are shown in Tables 13 and 14 respectively.

Table 13. Residues of flumethrin in honey from bee colonies treated with 4 strips/frame at 3.6 mg flumethrin/strip according to UK GAP.

Country/year	No. Colonies/ No. Samples	Treatment		Time of sample collection	Residues, mg/kg	Ref.[3]
		Duration (weeks)	Period			
Germany 1987-88	6/3[1]	6	early Sept. to mid-Oct. (pre-winter storage period)	June 1988 (after early nectar flow)	nd (<0.002) (3)[2]	49
Germany 1987-88	6/6	18	late Oct. 1987 to mid-March 1988	June 1988 (after early nectar flow)	nd (<0.002) (6)	50
Germany 1986	7/15	not given	early May (1985?)to mid- April 1986	not given	nd (<0.002) (15)	51
Germany 1988	4/4	20	May to Sept. 1988 (during nectar flow)	August 1988 (last wk.)	nd (<0.002) (4)	52
Germany (Lindlar) 1992-93	24/1	23	Oct. 12, 1992 to March 8, 1993	June 15, 1993	nd (<0.001)	47
Germany (Leverkusen) 1991-92	12/4	56	early Sept. 1991 to mid-Oct. 1992	1993 (end fruit at dandelion flowering)	nd (<0.001) (4)	47

Country/year	No. Colonies/ No. Samples	Treatment		Time of sample collection	Residues, mg/kg	Ref.[3]
		Duration (weeks)	Period			
UK	not given/1	not given	not given	1993 (spring)	nd (<0.001)	48

[1]Three 2-colony samples
[2]Numbers in parentheses following the residues are the numbers of samples with those values
[3]Numbers correspond to tab numbers in 1996 Bayer submission, Vol. IV:
47. Krebber, 1994f; 48. Krebber, 1994g; 49. Krieger and Riegner, 1990a; 50. Krieger and Riegner, 1990b; 51. Krieger and Riegner, 1990c; 52. Krieger and Riegner, 1990d.

Table 14. Residues of flumethrin in beeswax from bee colonies treated with 4 strips/frame at 3.6 mg flumethrin/strip[1]

Country, year	No. Colonies/ No. Samples	Treatment		Time of sample collection	Residues, mg/kg[1]	Ref.[3]
		Duration (weeks)	Period			
Germany 1986	2/4	6	early March to mid-April 1986 (before nectar flow)	April 1986	<0.015, 0.017, 0.015, 0.04	53
Germany 1987	6/3	6	early Sept. to mid-Oct. 1987	June 1988 (after early nectar flow)	<0.02, 0.04, 0.05	54
Germany 1988	4/4	20	May to Sept. 1988	Sept. 1988	0.03, 0.1 (2), 0.13	55
Switzerland	not given/13 (3 regions)	not given	not given	1993	<0.03 (4), 0.03 (2), 0.04, 0.05 (2), 0.06, 0.07(2), 0.2	45
Germany 1991	4/4	4	June 28 to July 23, 1991	August 1991	0.07, 0.1 (2), 0.15[2]	46

[1]Numbers in parentheses following the residues are the numbers of samples with those values.
[2]The treatment for these samples was with 40 strips/hive: 10 times the recommended rate.
[3]Numbers correspond to tab numbers in 1996 Bayer submission, Vol. IV:
45. Krebber, 1994d; 46 Krebber, 1994e; 53. Krieger and Riegner, 1990e; 54. Krieger and Riegner, 1990f; 55. Krieger and Riegner, 1990g.

FATE OF RESIDUES IN STORAGE AND PROCESSING

No information was provided.

Residues in the edible portion of food commodities

All the residues from supervised trials were in edible items.

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

Information was provided on residues of flumethrin in the perirenal fat of animals from an Australian random survey from July 1993 to December 1995. No residues (<0.02 mg/kg) were found in the fat of 2545 pigs, 801 horses, 642 goats, 158 deer, 154 buffalo 94 game pigs, 93 kangaroos, or 27 game goats. Flumethrin is not registered in Australia for use on these animals, but data were available because multi-residue methods were used. Although flumethrin is not registered for use on sheep in Australia, one of 4675 samples of sheep fat contained a residue in the range >0.1-0.2 mg/kg. Residues were also detected in perirenal beef fat in 59,6657 samples (0.9%), distributed as shown below.

	Residue range, mg/kg					
	0.02	>0.02-0.04	>0.04-0.1	>0.1-0.2	>0.2	Total
No. of samples in range	1	16	36	5	1	59
% of 6657 samples analysed	0.02	0.24	0.54	0.08	0.02	0.9

As a follow-up, treatment histories were obtained in 26 cases. Although details of the treatments were not recorded (except that they were pour-on treatments) they were reported to have complied with GAP. Residues were detected up to 1.1 mg/kg (Table 15).

Table 15. Flumethrin residues in perirenal fat[1] of Australian cattle treated on the farm with a flumethrin pour-on formulation (Webster *et al*., 1996).

Residue range, mg/kg	Number of samples within residue range at indicated interval between treatment and slaughter							
	<2 weeks	3-4 weeks	1-2 months	3-6 months	6-9 months	9-12 months	>12 months	?
0.05-0.1	2		6		1		1	
0.11-0.2			3	3	1	1	1	1[2]
0.21-0.5		3						
>0.5		1 (0.56)	2 (0.61, 1.1)					

[1]The nature of the samples was not specified but they were presumably perirenal fat because the study was to follow up positive results in random monitoring of perirenal fat and because it is Australian regulatory practice to analyse perirenal fat (Webster *et al*., 1996).

[2]This sample was from a cow which was not known to have been treated.

NATIONAL MAXIMUM RESIDUE LIMITS

The following national MRLs for flumethrin in animal products were reported for Australia (Webster *et al*., 1996).

	mg/kg[1]	
Cattle meat	0.05	Temporary
Cattle, edible offal of	0.05	Temporary
Milks	0.05	Temporary
Horse, edible offal	0.1	
Horse meat	0.1	

[1]Assumed to be whole-product basis (not indicated otherwise).

APPRAISAL

Flumethrin, (*R,S*)-Æ-cyano-4-fluoro-3-phenoxybenzyl 3-(ß,4-dichlorostyryl)-2,2-dimethylcyclopropanecarboxylate, is a pyrethroid acaracide composed of a mixture of two diasterioisomers (*trans*-*Z*-1 and *trans*-*Z*-2, with an approximate ratio 55:45) formed by the reaction of 4-fluoro-3-phenoxybenzaldehyde and *trans*-(*E*)-3-[2-chloro-2-(4-chlorophenyl)vinyl]-2,2-dimethylcyclopropanecarboxylic acid chloride in the presence of cyanide. It is widely used as a topical pesticide for the control of ectoparasites such as ticks and buffalo flies on farm animals by spraying, dipping or other treatments. It was reviewed by the present Meeting for the first time. The focus was on the uses against animal ectoparasites, although flumethrin residues in honey and beeswax from supervised trials on honey bee colonies were also provided and reviewed.

The Meeting agreed that data on environmental fate were not required in relation to potential flumethrin residues in animal products from uses as an ectoparasiticide, but considered such information to be desirable for assessing the potential for undesirable environmental effects.

The mammalian metabolism of flumethrin was reported for rats and cattle. Flumethrin administered orally, i.v. and duodenally showed ester hydrolysis to 3-(ß,4-dichlorostyryl)-2,2-dimethylcyclopropanecarboxylic acid (flumethrin acid, BFN 5533A) and (through the probable cyanohydrin (FCR 1271) and 4-fluoro-3-phenoxybenzaldehyde (FCR 1260) intermediates) to the other main identified metabolite 4-fluoro-3-phenoxybenzoic acid. Flumethrin acid is conjugated to form the glucuronide and the fluorophenoxybenzoic acid component is further oxidized to 4-fluoro-3-(4-hydroxyphenoxy)benzoic acid, the last two compounds being conjugated to glycine. The studies indicate substantially greater ^{14}C elimination in the faeces than in the urine from chlorophenyl-labelled flumethrin and roughly equal elimination in faeces and urine from the fluorophenyl-labelled compound, with faster elimination of the fluorophenyl label.

Although the rat metabolism studies with labelled or unlabelled flumethrin are useful for identifying metabolites and provide useful information on the mammalian metabolism of orally, i.v. or

duodenally administered flumethrin, they do not fully reflect exposure from topical application which is relevant to the approved uses on cattle, horses, goats or sheep.

A 1986 material balance and distribution study on cattle was based on the administration of fluorophenyl-labelled ^{14}C-flumethrin as a back treatment, approximately at approved rates. After 48 hours 71% of the administered dose remained in or on the skin in the application area with $\leq$10 ng flumethrin equivalents/g in the tissues and $\leq$3 ng equivalents/ml in the milk through 31 hours, demonstrating slow absorption within this short test period.

In a similar study on cattle in 1994 chlorophenyl-labelled flumethrin was administrated at approximately an approved rate intravenously as opposed to topically and samples were analysed to study metabolism. Relatively little radioactivity was eliminated within the short period of 8 hours before slaughter, but significant amounts of the administered dose (32% in a lactating cow, 13% in a steer) were detected in edible tissues in the decreasing order liver (in the cow) or muscle (in the steer), fat and kidney. Residues as mg/kg flumethrin equivalents in the tissues were highest in liver (13.4 mg/kg cow, 3.4 mg/kg steer), with 0.2-0.3 mg/kg in muscle, 0.2-0.4 mg/kg in fat and 0.3 mg/kg in milk. The liver residues suggest greater metabolic activity in the steer (lower radioactivity in the liver) than in the cow. This is reinforced by the ratios of flumethrin to BNF 5533A of 87:7 and 29:40 in cow and steer livers respectively and by the higher levels of glucuronide found in the liver and kidney of the steer. The opposite may be seen in male and female rats if flumethrin to metabolite ratios in faeces are compared: the proportion of the metabolite is higher in female faeces.

Of the measured radioactivity, 76 to 95% of the residues were identified in tissues and 68% in milk. Only flumethrin was identified in the milk but an unidentified metabolite constituted almost 12% of the milk radioactivity. BNF 5533A glucuronide was identified only in the liver and kidney. Residue levels of BNF 5533A were 1-1.5 times those of flumethrin in muscle and fat with no pronounced difference between the cow and steer. While the log P_{ow} of 6 for flumethrin indicates fat-solubility, residue levels of flumethrin *per se* from the i.v. injections in this study are comparable in the muscle and fat of both steers and cows, actually slightly higher in the muscle. However, as will be discussed later, residues from topical applications in the supervised trials were higher in the fat.

Analytical methods are available for the determination of flumethrin and flumethrin acid (BNF 5533A) in the tissues of cattle and sheep and of flumethrin in milk. Only flumethrin and, at lower levels, an unidentified metabolite were reported in milk in cattle metabolism studies. In recent analytical methods homogenized tissues are generally extracted with acetonitrile or acetonitrile/phosphoric acid solution, partitioned into dichloromethane and/or hexane, cleaned up on silica gel columns and determined by HPLC using UV detectors. In some cases the flumethrin acid metabolite is further cleaned up on C-18 solid phase cartridges after separation from flumethrin on silica gel before HPLC determination. Analysis of milk is similar, although in some cases milk solids are removed by the addition of acetone before partitioning into acetonitrile.

Multi-residue methods for organochlorine compounds have also been modified for the determination of pyrethroids, including flumethrin, in animal fat. The modified method involves the partition of rendered finely sliced fat between acetonitrile and hexane, dilution of the acetonitrile, Florisil column clean-up and determination by GLC with EC detection. Information was not sufficient for an independent estimate of a limit of determination for this method, although satisfactory recoveries were achieved at fortification levels of 0.02 mg/kg.

Generally, analytical recoveries are 80% or better by the more recent methods in tissues and milk with fortification at or near the reported limits of determination. Limits of determination of 0.005 mg/kg for flumethrin in milk, 0.01 mg/kg for flumethrin acid in milk, 0.01 mg/kg for flumethrin in

tissues, and 0.01 or 0.02 mg/kg for flumethrin acid in tissues are generally reported, depending on the method. For the most part these limits are supported by sample chromatograms from the authors' laboratories, although in some cases sample chromatograms do not convincingly support a 0.01 mg/kg limit of determination in liver and kidney.

While the reported limits of determination may be achievable in the authors' laboratories, the Meeting concluded that limits of determination of 0.01 mg/kg for flumethrin in milk and 0.02 mg.kg for flumethrin and flumethrin acid in tissues are more realistic for routine enforcement among different laboratories. However, for dietary intake estimates the use of half these levels would be appropriate where no residues are detected.

Analytical methods have also been reported for residues of flumethrin in honey and beeswax, with recoveries generally about 75% or better. The reported limits of detection and determination were 0.001 and 0.003 mg/kg for honey and 0.02 and 0.026 mg/kg for wax, and the authors' sample chromatograms were consistent with these levels.

The manufacturer's working paper considered information on the stability of residues in stored analytical samples not to be necessary and no such studies were submitted, except relevant information incidentally included in one supervised trial report which showed flumethrin residues in milk to be stable for 40 days at -18°C. On the basis of this report and the persistence of flumethrin residues in fat even in live cows, the Meeting considered that the information provided was adequate to support estimates of maximum residue levels in cattle meat (fat) and milk. The Meeting further concluded that information on the stability of flumethrin in stored samples of other tissues (liver, kidney) was needed before maximum residue levels estimated for these tissues could be recommended for use as MRLs.

Data were available on supervised trials in a number of countries of ectoparasite control in cattle, sheep or goats using a variety of flumethrin formulations, as well as on mite control in beehives.

Data on supervised trials of ectoparasite control on cattle were available from Australia, Germany, South Africa and Japan. Approved uses (including labels) were provided only for Australia (on cattle and horses). The most recent, comprehensive and best described studies of flumethrin residues in cattle from plunge dipping or pour-on applications are 1994-1995 Australian trials submitted by the Australian government, but there was no information on whether GLP was followed in them. For example, no information was provided on the interval from slaughter to analysis, and actual storage conditions were not reported although the protocol called for storage at -40°C. An exception was a GLP study to determine the potential for residues of flumethrin acid from the treatment of cattle with a flumethrin pour-on formulation.

The 1994-95 Australian studies did not include data from spray applications, which are approved in Australia, although older Australian (and other) studies submitted to the Meeting included data from some types of approved spray applications. The older studies for the most part were also not reported to have been conducted under GLP, although in many cases essential information was available to give a reasonable degree of confidence in the data. Because only Australian approved uses for ectoparasite control in cattle were available, the Meeting based its analysis of the cattle data primarily on the Australian trials. That situation was not ideal since in the most recent and best documented studies residues were determined only in fat whereas some of the older trials included analyses of fat, liver, muscle, kidney and milk.

In the 1994-95 Australian trials low residues ($\underline{\leq}$0.008 mg/kg from one dip, $\underline{\leq}$0.013 mg/kg from two dips) were reported for plunge dip treatments, except in one of 84 test animals which showed 0.04 and 0.05 mg/kg flumethrin in loin and renal fat respectively. The treatments were in accordance with

approved uses, except that the interval between the two treatments was 3 days compared to the recommended minimum of 10 days. Higher residues were reported from approved pour-on applications to a total of 56 animals, with maximum residues of 0.04 mg/kg in loin fat from one application and 0.05 mg/kg from two applications 7 days apart, as compared with an implied minimum approved interval of 14 days. The maximum residues in renal fat were 0.11 mg/kg from one treatment and 0.14 mg/kg from two applications at the 7-day interval. The combined data from dip and pour-on trials at approved rates in the 1994-95 and 1981-84 Australian trials are shown below. The numbers of samples with the same residue or within the same ranges are shown in parentheses.

Single dips

Fat <0.005 (67), 0.006, 0.007, 0.008, 0.041, 0.047 mg/kg.
Liver <0.005 (6) mg/kg.
Muscle <0.005 (5), 0.01 mg/kg.
Kidney <0.005 (6) mg/kg.

2-dips

Fat <0.005 (69), 0.009, <0.011, <0.013 mg/kg (3-day interval as compared with the approved 10-day).

Single pour-on

Fat <0.005 (24), 0.005 (9), 0.006-0.01 (11), 0.011-0.015 (13), 0.017-0.020 (6), 0.023-0.029 (9), 0.032, 0.034, 0.04, 0.042, 0.11 mg/kg. Total number = 77.
Liver <0.005 (23), 0.01 mg/kg.
Muscle <0.005 (22), 0.005, 0.01 mg/kg.
Kidney <0.005 (23), 0.01 mg/kg.

2 pour-ons

Fat 0.011-0.015 (8), 0.016-0.020 (9), 0.022-0.025 (9), 0.026-0.03 (9), 0.031-0.051 (15), 0.052-0.058 (3), 0.097, 0.14 mg/kg (7-day interval as compared with the approved 14-day). Total number = 55.

"Fat" includes renal and subcutaneous fat.
The double-underlined ranges within which the median residues fall.

In the Australian spray trials in 1981 at 0.7-2.6 times GAP rates, the residues in fat, liver, muscle and kidney (24 samples of each) were all <0.05 mg/kg.

On the basis of the single pour-on applications according to GAP the Meeting estimated an STMR of 0.01 mg/kg for the fat of meat and <0.005 mg/kg for whole meat.

As noted above, information on approved uses on cattle was provided only for Australia, but it is useful and of interest to relate the results of trials in other countries to Australian approved uses. In such trials the maximum residues from applications approximating Australian approved uses were 0.08 mg/kg (or <0.1 mg/kg depending on the study) in fat, and <0.01 to <0.1 mg/kg, again depending on the study, in liver, muscle and kidney. In one German study residues in liver were 0.03 mg/kg. While such a comparison may be questionable, it suggests that the maximum flumethrin residues in cattle are likely to be similar if approved uses in those countries are similar to those in Australia. The German studies also show that flumethrin residues in fat from pour-on applications reach their highest level after about

4 days and stay at or near that level for up to 28 days. This confirms the finding in the Australian trials.

It is clear that the potential for residues in cattle tissues is greater from approved pour-on uses than from spray or dip applications and, in contrast to metabolism studies with i.v. administration, field trials indicate that flumethrin residues from topical applications are likely to be significantly greater in fat than in other tissues. It is also of interest to note that the maximum residues of 0.11 to 0.14 mg/kg found in cattle fat in the pour-on trials are consistent with residues up to 0.2 mg/kg found in random Australian monitoring and less than some residues (as high as 1.1 mg/kg) found in follow-up investigations prompted by the finding of residues in random monitoring.

The supervised trials data are consistent with MRLs of 0.2 mg/kg in the carcase fat of cattle and 0.01 mg/kg in cattle muscle and kidney. The Meeting noted maximum flumethrin residues of 0.01 mg/kg in liver in the Australian trials, took into account residues up to 0.03 mg/kg in German trials approximating approved Australian uses and <0.05 or <0.1 mg/kg in other non-Australian trials, and concluded that prudence required a 0.05 mg/kg level for liver.

In the absence of studies of the storage stability of residues in tissues other than fat and in view of differences between the ratios of flumethrin residues in fat to those in non-fatty tissues found in metabolism studies and supervised trials, the Meeting was unwilling to recommend the use of the maximum residue levels estimated for liver and kidney as MRLs. This could be reconsidered at a future JMPR if relevant studies of storage stability with tissues other than fat become available.

The monitoring data suggest that residues in fat may occasionally exceed 0.2 mg/kg, especially from pour-on applications. For dietary intake purposes a level of 0.005 mg/kg (generally the lowest reported limit of determination) would be reasonable for flumethrin in the muscle, liver, fat and kidney of cattle.

A ratio of 1.9 (0.84 correlation coefficient) was reported for the residues in perirenal to those in subcutaneous fat arising from pour-on applications. Residues were also reported to be up to 32% lower in the fat of animals with greater fat deposits, presumably indicating fat dilution of the residues. In selected samples, analysis of extracted fat from core samples from cartons of frozen carcases correlate well with renal and loin fat samples taken at slaughter from the same animals.

Flumethrin residues in loin and renal fat from single approved pour-on applications increased rapidly from 2 days after treatment through the fourth day, then declined slowly until a second increase after 21 days, then declined gradually to 45 days. A similar pattern of two peaks was noted for two applications, although residues were higher and the second peak later owing to the second application. The pattern confirms the persistence of flumethrin in animal fat.

Supervised trials of ectoparasite control in sheep were available from Australia, South Africa and the U.K. Because information on approved uses was available only from the UK, the Meeting based its conclusions on sheep primarily on the single UK study. Sheep were dipped once (re-dipping is permitted after 14 days) approximately according to approved uses and samples of fat, liver, muscle and kidney were taken for analysis at intervals from 0.5 to 4 days after treatment. Although residues were low, they tended to be higher in subcutaneous than in omental fat. The maximum residues were 0.03 and <0.01 mg/kg in subcutaneous and omental fat respectively, 0.02 mg/kg in liver and <0.01 mg/kg in muscle and kidney.

Maximum residues in the relatively old Australian dip trials at 0.9 to 1.3 times UK approved use rates were 0.04 mg/kg in fat and <0.005 mg/kg in liver, kidney and muscle. Relatively old data were also available from Australia and South Africa from pour-on applications, but no relevant

approved uses were provided. The maximum residues found in the Australian sheep dipping trials were comparable to those found in the dip treatments of Australian cattle, and reasonably consistent with the UK dipping results when the use rates were similar. However, because only one well-documented sheep study was available which reflected approved uses, because only one dip was represented and because sheep are generally expected to have higher residues than cattle, the Meeting concluded that the data were insufficient to estimate maximum residue levels for sheep.

Data on residues in milk were available from supervised trials on cattle, sheep and goats. Data on residues in cattle milk were available from Australia, Germany, Japan and South Africa. As with cattle tissues information on approved uses was available only for Australia and the Meeting placed most emphasis on the Australian trials. Although the Australian studies were relatively old, they were for the most part acceptably documented, included pour-on and spray applications and covered a range of intervals after treatment. The maximum residues approximately reflecting Australian approved uses after various intervals were as follows.

Single Pour-on	GAP rate	Ratio to 9 h	24 h	72 h
		0.5	0.01	0.01
	0.01			
		1.0	0.04	0.02
	0.01			
		0.5	0.04	0.04
	0.02			
		1.0	0.03	0.03
	0.01			
Single Spray				
		1.0	<0.1	<0.1
		1.0	0.01 (2 applications)	
		1.3	<0.01	<0.01

The combined results from the pour-on treatments at 0.5 and 1 times the GAP rate gave the following residues at 9-72 hours: <0.01 (14), 0.01 (13), 0.02, 0.02, 0.03 (4), 0.04 (3).

The Meeting estimated an STMR for flumethrin in milk of 0.01 mg/kg.

Although no information on German approved uses was provided, two pour-on applications at approved Australian use rates resulted in maximum residues in 2 trials of 0.04 and 0.06 mg/kg after 2 days, decreasing to 0.02 and 0.04 mg/kg after 4 days and then continuing to decrease slowly.

As in the case of tissues it is clear that higher residues result from pour-on applications than from other types of application. The results suggest that multiple applications produce higher residues and point to the need for additional trials with multiple applications at approved use rates.

Supervised trials data on residues of flumethrin in sheep and goat milk were also available, but without information on relevant approved uses. At rates approved for pour-on applications to cattle in Australia, the maximum residues were 0.04 mg/kg and <0.01 mg/kg in goat and sheep milk respectively.

On the basis of the available information a maximum residue level of 0.05 mg/kg for cattle milk is reasonable, although additional data from trials with multiple treatments at approved use rates

are needed to confirm that estimate.

In addition to residues of flumethrin *per se* in cattle tissues, data were also available from one trial on residues of flumethrin acid in tissues at intervals of 1 to 35 days after the second of two pour-on applications of flumethrin at approved application rates. The maximum flumethrin acid residues, found after 2 days were 0.04 mg/kg in fat, 0.06 mg/kg in liver, 0.01 mg/kg in muscle, and 0.05 mg/kg in kidney. These indicate that the acid metabolite is less soluble than the parent compound in fat.

Honey and beeswax. Eleven supervised trials were conducted in Switzerland, the UK, and Germany (mostly Germany) to determine the potential for flumethrin residues in honey and beeswax from the treatment of bee colonies for mite control. Applications were in the form of flumethrin-impregnated strips. The trials varied in duration from 4 to 56 weeks and covered a variety of periods of honey production, including pre-winter storage periods and before or during nectar flow. At the recommended rate of 4 strips/frame (3.6 mg ai/strip) approved in the UK, no residues (<0.001 or <0.002 mg/kg, depending on the analytical method) were measured in any of the 34 honey samples analysed. The Meeting concluded that a maximum residue level of 0.005 mg/kg (limit of determination) would be suitable for use as an MRL for honey.

Residues in beeswax were <0.02 (2), 0.02 (2), <0.03 (4), 0.03, 0.04 (3), 0.05 (3), 0.06, 0.07 (2), 0.1 (2), 0.13 and 0.2 mg/kg. The maximum residues in each of the four trials were 0.02, 0.05, 0.13 and 0.2 mg/kg with a mean of 0.1 mg/kg and an estimated median of 0.09 mg/kg. In one trial with treatment at ten times the recommended rate the maximum residue was 0.15 mg/kg. Information was lacking on many aspects of the Swiss trial which gave the maximum residue of 0.2 mg/kg.

Residue Definition. The flumethrin acid metabolite BNF 5533A was found in cow metabolism studies (8 hours after the i.v. injection of flumethrin) to occur at 1 to 1.5 times the level of flumethrin in animal tissues, but was not reported in milk. The Meeting assumed that flumethrin would be of significantly greater toxicological concern than the metabolite, noted that only flumethrin *per se* was reported in milk and was the main residue in tissues (especially in fat) found in supervised trials, and concluded that flumethrin was the preferred indicator residue for regulatory purposes.

For the estimation of dietary intake, it is useful to note that metabolism studies suggest that the total residues (or flumethrin *per se*) in meat (muscle) could be similar to or slightly higher than in fat, although that did not occur in the supervised trials where flumethrin residues were higher in fat than in muscle, essentially in all instances. The total residues of flumethrin and flumethrin acid in tissues could be expected to be at most about three times those of flumethrin.

Fat-solubility and expression of residues in meat. The log P_{ow} of 6 for flumethrin indicates high fat-solubility. This is supported by a metabolism study with back treatments of a lactating cow, where measurable residues were found in renal fat but not in muscle or subcutaneous fat. Metabolism studies with i.v. dosing, however, indicate that once flumethrin residues enter the blood stream, levels of flumethrin *per se* or of total radioactivity are similar in the muscle and fat of both steers and cows. If there is a difference residues in muscle under these conditions appear to be slightly higher. The same is true for BNF 5533A, the flumethrin acid metabolite. Analytical methods are available for the determination of flumethrin residues in carcase meat or fat. Residues in edible offal can conveniently be on a whole-commodity basis.

The manufacturer expects to propose limits to the European Union for liver (0.04 mg/kg), milk (0.12 mg/kg) and fat (0.1 mg/kg), but none for muscle or kidney, since no residues were reported in these tissues in cattle or sheep. However, the procedures for sampling and analysis in the field trials and for the regulation of residues in meat (muscle) are key factors in determining how residues in meat

should be expressed.

The most relevant, recent and comprehensive supervised trials (Australian 1994-95) involved the determination of residues in fat rendered from finely sliced loin (subcutaneous) and renal fat. In the older studies, even though mean residues were at or below the limit of detection or determination, residues in the meat (muscle) of individual cows were measurable in some cases (up to 0.02 mg/kg). In older trials on cattle and sheep the type of fat was not defined, except in a 1980 South African trial where it was renal fat. No information on approved uses was provided for the older (1980-1989) trials, except those in Australia.

Since meat is often regulated at the international level on the basis of residues in subcutaneous fat, as a practical matter it is convenient to propose limits for meat on a fat basis (in the carcase fat) derived from residues in loin and subcutaneous fat found in supervised trials, noting that residues can be higher in renal than in subcutaneous fat. For these reasons the Meeting recommended that limits for flumethrin in meat be expressed on the carcase fat.

RECOMMENDATIONS

The residue levels recorded below are recommended for use as MRLs or for the estimation of dietary intakes.

Definition of the residue for compliance with MRLs and for estimation of dietary intake: flumethrin

The residue is fat-soluble.

Commodity		Recommendations, mg/kg	
CCN	Name	MRL	STMR
MM 0812	Cattle meat	0.2 (fat) V	0.01 (fat) 0.005 (whole muscle)
ML 0812	Cattle milk	0.05 F V	0.01
	Honey	0.005*	0.005

* At or about the limit of determination

FURTHER WORK OR INFORMATION

Desirable

1. Information on the stability of flumethrin residues in stored analytical samples of liver and kidney in relation to the periods and conditions of storage of the samples from supervised trials.

2. Submission of data from new supervised trials on animals expected to be available in June 1996 (Webster, *et al*., 1996).

3. Results of analyses of tissues and milk from additional supervised trials on cattle in which

multiple, especially pour-on, applications have been made in accordance with approved uses.

4. Studies on the fate of flumethrin in the environment, especially its persistence and mobility in soil.

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HALOXYFOP (193)

EXPLANATION

Haloxyfop was evaluated for the first time by the 1995 JMPR. That Meeting received data on residues in beans and peas but the exact Codex commodities to which the data referred were not clear. The 1995 Meeting agreed not to estimate maximum residue levels until the commodity descriptions were clarified. Supplementary information on the commodity described as "peas" has now been made available.

The 1995 Meeting could not complete the evaluation of the ruminant and poultry metabolism studies in the time available and reviewed the residue data on ruminants and poultry on the assumption that metabolism in these species is essentially the same as indicated in the metabolism studies on rats, mice, dogs, monkeys and humans. The present Meeting completed the evaluation of the studies of ruminant and poultry metabolism.

The 1995 JMPR estimated a number of maximum residue levels, but agreed not to recommend their use as MRLs because of the lack of critical supporting data on the uptake of soil degradation products by crops. Studies on the uptake of haloxyfop or its degradation products from soil treated with haloxyfop have been made available to the present Meeting.

METABOLISM AND ENVIRONMENTAL FATE

Animal metabolism

Ruminants. Two lactating goats were dosed with [*phenyl*-U-^{14}C]haloxyfop in gelatin capsules twice daily for ten consecutive days at a rate equivalent to 16 ppm in the diet (Yackovich and Miller, 1983a). Urine and faeces were collected each morning during the test period. Blood samples were taken daily and expired air was collected from one goat for a ten-hour period. Milk was collected each morning and evening. Twelve hours after the final dose the animals were slaughtered and tissue samples and gut contents taken.

The recovery of the administered radioactivity was 90-96%. Most was excreted in the urine (84-92% of total administered dose) as unchanged haloxyfop; milk contained 1.9-3.2% and faeces 1.5-1.9%. No radioactivity was found in the expired air. Radioactivity as haloxyfop equivalents was 0.02 mg/kg in muscle, 0.1 mg/kg in fat, 0.4 mg/kg in liver and 1.3 mg/kg in kidney.

The liver and kidneys contained only haloxyfop, while the fat contained a non-polar conjugate which was easily hydrolysed to yield haloxyfop under alkaline conditions. The residue in muscle was not identified because the radioactivity was too low.

The radioactivity in the milk reached a plateau within two days after the first dose at 0.2 to 0.3 mg/kg haloxyfop equivalent, representing about 2 to 3% of the daily dose. The residue was

primarily in the milk fat fraction in the form of non-polar conjugate(s) which were easily hydrolysed to yield haloxyfop under alkaline conditions. Enzymatic hydrolysis with lipase (triacylglycerol-acyl-hydrolase) indicated that the conjugates were triacylglycerols.

The recovery of radioactivity and its distribution are shown in Table 1.

Table 1. Recovery and distribution of radioactivity following the feeding of [^{14}C]haloxyfop to lactating goats.

Sample	Recovery of radioactivity, % of total dose	Radioactivity as mg/kg haloxyfop equivalent	% of haloxyfop in residual radioactivity
Fat	0.04-0.06	0.06-0.11[1]	>90[3]
Heart	<0.01	0.02-0.07[1]	
Kidneys	0.09-0.11	1.07-1.45[1]	>90[4]
Liver	0.14-0.16	0.31-0.45[1]	>90[4]
Muscle	0.09-0.11	<0.01-0.02[1]	
Gut contents	0.25-0.40	-	
Milk (whole)	1.92-3.18	0.23-0.33[2]	
Milk (fat)	-	1.53-3.41[2]	>93[3]
Urine	84.4-91.7	-	99[5]
Faeces	1.48-1.88	-	
Expired air	<0.01	-	
Total	89.8-96.2	-	

[1] At 12 hours after the final dose
[2] Average concentration from third day
[3] Non-polar conjugate
[4] Free haloxyfop or polar conjugate
[5] Free haloxyfop

Poultry. Four laying hens were dosed with [*phenyl*-U-^{14}C]haloxyfop in gelatin capsules once a day for eleven consecutive days at a rate equivalent to 12 ppm in the diet (Yackovich and Miller, 1983b). Eggs and faeces were collected each morning during the test period. The hens were killed 24 hours after the final dose and tissue samples and gut contents taken.

The recovery of the administered radioactivity was 93-102%, mostly from the excreta with small amounts in eggs (82-90% and 1.1-2.6% of total administered dose respectively). In the tissues the radioactivity as haloxyfop equivalent was 0.12 mg/kg in muscle, 0.99 mg/kg in fat, 1.8 mg/kg in liver and 4.2 mg/kg in the kidneys.

The excreta, liver and kidneys contained mainly haloxyfop with some polar and/or non-polar conjugates, while most of the radioactivity in the fat was due to a non-polar conjugate of haloxyfop. Both the polar and non-polar conjugates were easily hydrolysed to yield haloxyfop under alkaline conditions. The residue in muscle was not identified because its radioactivity was too low.

The residue in the eggs was mainly in the yolk, where its radioactivity reached a plateau within seven days after the first dose at about 3 mg/kg haloxyfop equivalent, representing about 2 to 3% of the daily dose. As in goat milk fat, the residue in the egg yolk was identified as non-polar conjugate(s) of haloxyfop, easily hydrolysed by alkali to yield haloxyfop and shown by enzymatic hydrolysis with lipase to be triacylglycerol(s). The recovery and distribution of radioactivity are shown in Table 2.

Table 2. Recovery and distribution of radioactivity following the feeding of [^{14}C]haloxyfop to laying hens.

Sample	Recovery of radioactivity, % of total dose	Radioactivity as mg/kg haloxyfop equivalent	% of haloxyfop in residual radioactivity
Fat	0.15	0.99[1]	99[(3)]
Heart	-	0.28[1]	
Kidneys	0.23	4.22[1]	90[(4)]
Liver	0.37	1.82[1]	91[(4)]
Muscle	0.25	0.12[1]	
Gut contents	7.5	-	
Egg (whole)	1.58	-	
Egg (white)	-	0.25[2]	
Egg (yolk)	-	2.94[2]	92[(3)]
Developing yolk	0.49	-	
Faeces	86.54	-	97[(5)]
Total	97.95	-	

[1] At 24 hours after final dose
[2] Average concentration from 7th day
[3] Non-polar conjugate
[4] 60-70% as free haloxyfop, non-polar and polar conjugates
[5] Free haloxyfop or polar conjugates

Uptake by crops from treated soil

Because haloxyfop is often applied to soil the 1995 Meeting concluded that information was needed on the uptake by crops of haloxyfop and its degradation products from soil. Appropriate studies were reported to the present Meeting.

An emulsifiable formulation of [*phenyl*-U-^{14}C]haloxyfop-butyl was applied to bare soil at a rate equivalent to 0.56 kg ai/ha (Yackovich and Miller, 1983c). Thirty days later the plot was tilled and planted with spring wheat, soya beans, carrots, turnips and lettuce. The crops were grown to maturity and the uptake of ^{14}C determined by combustion and LSC. The highest residue, 0.07 mg/kg haloxyfop equivalent, was found in immature soya bean foliage sampled 56 days after planting. Residues in the edible portions of the crops were ≤0.01 mg/kg haloxyfop equivalent. Owing to the

low levels of radioactivity, attempts to isolate and identify the residues were unsuccessful. The results are given in Table 3.

Table 3. Uptake of ^{14}C by crops planted 30 days after treatment of soil with [^{14}C]haloxyfop at 0.56 kg ai/ha.

Sample	Days from planting to harvest	^{14}C as mg/kg haloxyfop equivalent
Lettuce	49	0.01
Turnip foliage	64	<0.01
Turnip root	64	<0.01
Wheat grain	110	0.01
Wheat straw	110	0.02
Soya bean	113	<0.01
Soya bean forage	56	0.07
Soya bean straw	113	0.01
Carrot forage	115	<0.01
Carrot root	115	<0.01

An EC formulation of [*phenyl*-U-^{14}C]haloxyfop-butyl was applied to a crop of 37.5 cm soya bean plants at a rate equivalent to 0.56 kg ai/ha (Yackovich and Miller, 1983d). After harvesting the crop 130 days after treatment, the top 5 cm of soil was removed, brought into the laboratory, and sown with spring wheat, soya beans, sugar beet and lettuce. The crops were grown to maturity and the uptake of ^{14}C determined by combustion and LSC. Residues in the edible portions of the commodities were ≤0.04 mg/kg haloxyfop equivalent. Owing to the low levels of radioactivity, the residues could not be identified. The results are shown in Table 4.

Table 4. Uptake of ^{14}C by rotational crops from soil after treatment of original crop with [^{14}C]haloxyfop at 0.56 kg ai/ha. Rotational crops planted 130 days after treatment of primary crop.

Rotational crop sample	Days from planting to harvest	^{14}C as mg/kg haloxyfop equivalent
Lettuce	37	0.01
Lettuce	50	0.01
Lettuce	67	0.01
Lettuce	95	0.01
Soya bean	117	0.04
Soya bean forage	50	0.02
Soya bean straw	117	0.05
Wheat grain	104	0.02
Wheat straw	104	0.02
Sugar beet	132	0.01
Sugar beet forage	132	0.02

An emulsifiable formulation of [*phenyl*-U-^{14}C]haloxyfop-butyl was applied to a crop of soya bean plants at a rate equivalent to 0.28 or 0.56 kg ai/ha. Carrots, sugar beet, lettuce, wheat and soya beans were planted in the same plot the following year, grown to maturity and analyzed to determine the uptake of ^{14}C (Yackovich and Miller, 1984). The ^{14}C was determined by combustion and LSC. The highest residues, 0.007 and 0.01 mg/kg haloxyfop equivalent, were seen in soya bean grain and straw after treatment of the original crop at the higher application rate. Residues in the edible portions of the other crops were <0.005 mg/kg haloxyfop equivalent. The residues were too low for isolation and identification. The results are given in Table 5.

Table 5. Uptake of ^{14}C by rotational crops from soil after treatment of original crop with [^{14}C]haloxyfop. All rotational crops planted 240 days after original application.

Rotational crop sample	Application to original crop, kg ai/ha	Days from planting to harvest	^{14}C as mg/kg haloxyfop equivalent
Wheat grain	0.28	95	<0.005
Wheat straw	0.28	95	0.005
Soya bean	0.28	161	<0.005
Soya bean forage	0.28	75	<0.005
Soya bean straw	0.28	161	<0.005
Soya bean	0.56	161	0.007
Soya bean forage	0.56	75	<0.005
Soya bean straw	0.56	161	0.01
Lettuce	0.56	39	<0.005
Lettuce	0.56	47	<0.005
Lettuce	0.56	69	<0.005
Carrot root	0.56	153	<0.005
Carrot forage	0.56	153	<0.005
Sugar beet	0.56	139	<0.005
Sugar beet forage	0.56	139	<0.005

Unlabelled haloxyfop-methyl was applied to soya beans at a rate of 0.28 kg ai/ha or to cotton at a rate of 0.28 or 0.56 kg ai/ha (Bjerke *et al*., 1985). At 25-34 or 92-148 days after application, the primary crop was harvested and wheat, lettuce or sugar beet planted as rotational crops in the same plots. The crops were grown to maturity and analyzed by gas chromatography to determine the uptake of haloxyfop. The highest residues were found in immature wheat forage grown on plots where the primary crop had been harvested 25 days after treatment, at a level of 0.01 mg/kg. Residues in the edible portions of the rotational crops were <0.01 mg/kg haloxyfop. The validated LOD was 0.01 mg/kg for all substrates except wheat straw, for which it was 0.02 mg/kg.

USE PATTERN

Information on use patterns was submitted to the 1995 JMPR and reported in the 1995 monograph. The use pattern on beans and peas was recorded in Table 8 (p. 422).

RESIDUES RESULTING FROM SUPERVISED TRIALS

Supervised trials data on peas were submitted to the 1995 JMPR and reported in the 1995 monograph (Table 18, p. 432). The present Meeting was informed that all commodities referred to as "peas" were *Pisum sativum*. Because it is now possible to determine which residues resulted from treatments according to GAP, the Table is repeated below as Table 6.

Peas (pods and succulent seeds). Four supervised trials in France with racemic haloxyfop-etotyl were at 0.1 or 0.21 kg ai/ha with PHIs of 36-68 days. Three others in France and four in Germany were with haloxyfop-R-methyl at 0.052 or 0.1 kg ai/ha with PHIs of 36-60 days (Table 6).

Table 6. Residues of haloxyfop in peas (pods and succulent seeds). All single EC applications.

Country Year	Application			PHI, days	Growth stage at last treatment	Residues, mg/kg	Reference
	Compound/Form.	Kg ai/ha	Kg ai/hl				
France 1984	SR-EE EC	0.1	0.016	68	15-20 cm height	0.07	GHE-P-1671 (N66)
France 1988	SR-EE EC	0.1 0.21	N.S.[1] N.S.	39 39	8-9 leaves	<0.05 0.11	GHE-P-1956 (N30(R))
France 1989	SR-EE EC	0.1 0.21	0.021 0.042	36 36	5-6 leaves	0.03 0.06	GHE-P-2057 (N31(R))
France 1989	SR-EE EC	0.1 0.21	0.042 0.084	36 36	flower buds hidden by top leaves	0.04 0.07	GHE-P-2057 (N31(R))
France 1988	R-Me EC	0.052 0.1	N.S. N.S.	39 39	8-9 leaves	<0.05 0.06	GHE-P-1956 (N30(R))
France 1989	R-Me EC	0.052 0.1	0.01 0.021	36 36	5-6 leaves	0.03 0.04	GHE-P-2057 (N31(R))
France 1989	R-Me EC	0.052 0.1	0.021 0.042	36 36	flower buds hidden by top leaves	0.03 0.05	GHE-P-2057 (N31(R))
Germany 1989	R-Me EC	0.1	0.035	43 56	3 leaves	<0.02[2] ≤0.02	GHE-P-2154 (N36(R))
Germany 1989	R-Me EC	0.1	0.035	38 53	4 leaves	<0.02[2] ≤0.02	GHE-P-2154 (N36(R))
Germany 1989	R-Me	0.1	0.026	42	10 leaves	0.07[2]	GHE-P-2154 (N36(R))
Germany 1989	R-Me EC	0.1	0.026	42 60	6-7 leaves	0.03[2] ≤0.02	GHE-P-2154 (N36(R))

[1] Not specified in report
[2] Pod

APPRAISAL

Haloxyfop was evaluated for the first time by the 1995 JMPR. That Meeting received data on residues in beans and peas but the exact Codex commodities to which the data referred were not

clear. The 1995 Meeting agreed not to estimate maximum residue levels until the commodity descriptions were clarified. Supplementary information on the commodity described as "peas" has now been made available.

The 1995 Meeting could not complete the evaluation of the ruminant and poultry metabolism studies in the time available and reviewed the residue data on ruminants and poultry on the assumption that metabolism in these species is essentially the same as indicated in the metabolism studies on rats, mice, dogs, monkeys and humans. The present Meeting completed the evaluation of the studies of ruminant and poultry metabolism.

The 1995 JMPR estimated a number of maximum residue levels, but agreed not to recommend their use as MRLs because of the lack of critical supporting data on the uptake of soil degradation products by crops. Studies on the uptake of haloxyfop or its degradation products from soil treated with haloxyfop have been made available to the present Meeting.

Metabolism in lactating goats and laying hens

Metabolic studies on lactating goats and laying hens with 10 or 11 days consecutive oral dosing at rates equivalent to 12-16 ppm in the feed were reported.

In goats, the administered haloxyfop was rapidly absorbed from the gastrointestinal tract and mainly excreted in the urine unchanged (84-92%). The radioactivity remaining in the body was relatively low, at levels of 0.02, 0.1, 0.4 and 1.3 mg/kg haloxyfop equivalent in the muscle, body fat, liver and kidneys respectively 12 hours after the last dose. The liver and kidneys contained only haloxyfop or its polar conjugates; the radioactivity in muscle was too low for identification of the residues. The predominant metabolites in the body fat and milk fat were identified as non-polar conjugates of haloxyfop and were evidently triacylglycerols because they were hydrolysed by lipase to produce haloxyfop.

Laying hens eliminated 82-90% of the dose as intact haloxyfop in their excreta. Radioactive residues in tissues were 0.12, 0.99, 1.8 and 4.2 mg/kg haloxyfop equivalent in the muscle, body fat, liver and kidneys respectively 24 hours after the final administration. Polar and non-polar conjugates were found as major metabolites in the tissues. The nature of metabolites in the body fat and egg yolk was same as in goat body and milk fat. They were easily hydrolysed by lipase to yield haloxyfop.

In conclusion, the metabolism of haloxyfop in poultry is similar to that in ruminants, which is essentially the same as in the other mammalian species studied: rats, mice, dogs, monkeys and humans.

These studies show that the definition of the residue for products of animal origin should be the same as for plant products.

Residue evaluations

Peas (pods and succulent seeds). Four supervised trials were carried out with racemic haloxyfop-etotyl in France at application rates of 0.1 or 0.21 kg ai/ha. There was no information on French GAP but conditions in three of the trials were according to Spanish Gap for legumes (0.1-0.21 kg ai/ha applied after weed emergence at 2-4-leaf stage). In the other trial application was at the early budding stage, which is not recommended practice. The residues in the trials according to GAP were ≤0.05-0.11 mg/kg.

Three supervised trials were carried out in France and four in Germany with applications of haloxyfop-R-methyl at 0.052-0.1 kg ai/ha. No information on GAP was available from these countries, but the trial conditions complied with the GAP of some East European countries (0.052-0.13 kg ai/ha, 60-day PHI, or 0.052-0.16 kg ai/ha, up to closing of canopy). The residues were <0.02-0.07 mg/kg. The trials in which the application rates were more than 30% below the maximum registered rate were not used in estimating the STMR.

Two French trials with racemic haloxyfop were according to maximum Spanish GAP; the residues were 0.06 and 0.11 mg/kg. Three German trials with 0.1 kg ai/ha of haloxyfop-R and 53-60 days PHI were comparable with maximum GAP in Poland (0.13 kg ai/ha, 60-day PHI). The residues in all three were <0.02 mg/kg.

The residues from the 5 trials in rank order were <0.02 (3), 0.06 and 0.11 mg/kg.

The Meeting estimated a maximum residue level 0.2 mg/kg and an STMR of 0.02 mg/kg for haloxyfop in peas (pods and succulent seeds).

Pea hay or fodder (dry). Six supervised trials on field peas in France and four in Germany complied with French GAP for fodder peas (0.052-0.1 kg ai/ha of haloxyfop-R applied up to early tillering); the residues were <0.02-0.1 mg/kg. The same data were evaluated by the 1995 Meeting in estimating a maximum residue level for dry pulses. The residues in the peas under the use pattern for fodder peas were below the maximum residue level estimated for dry pulses (0.2 mg/kg). Data were submitted to the 1995 Meeting on residues in pea haulms from four supervised trials in Germany and in whole plants of pigeon peas from two trials in Australia. However as the moisture content of the samples was not known the residues could not be referred to a dry weight basis, as required for residues in animal feeds (see report of 1980 JMPR, Section 2.8).

The Meeting could not estimate a maximum residue level for pea hay or fodder (dry).

Estimation of STMR and STMR-P levels for commodities for which maximum residue levels were estimated at the 1995 JMPR

The residue definition for the estimation of STMR or STMR-P levels should be the same as for the estimation of maximum residue levels (haloxyfop esters, haloxyfop and its conjugates expressed as haloxyfop), because no other metabolites were found in plant or animal metabolic studies.

Orchard crops. Haloxyfop is used in orchards to control grass weeds. Since the application is directed at the weeds growing at the base of the trees, residues in fruits will be caused only by drift contamination or translocation after the uptake of residues from soil by the roots. The Meeting therefore concluded that orchard crops should be evaluated as a single group.

Although bananas are not an orchard crop, they can be regarded as orchard crops for evaluation since the purpose and method of application is the same.

Eight supervised trials on citrus fruits, three on apples, nine on grapes and two on bananas were carried out in Australia, Brazil, France, Italy and New Zealand. The residues were below the limit of determination (<0.01- <0.1 mg/kg) in all the trials, except one in Australia on grapes which showed 0.03 mg/kg, even in trials carried out at excessive application rates.

The Meeting estimated a nil residue for orchard crops and bananas taking into consideration the use pattern and the fact that practically no uptake of residues from soil was observed.

The Meeting estimated an STMR of 0 mg/kg for haloxyfop in citrus fruits, apples, grapes and bananas.

Pulses (dry). Haloxyfop is registered for use on several pulses. The Meeting concluded that the supervised trials on pulses could be evaluated together because of the similarities in the use patterns and residue behaviour.

Broad bean (dry). Conditions in two supervised trials (0.1 and 0.16 kg ai/ha, 103 and 171-day PHIs respectively) were comparable with maximum Australian GAP (0.1 kg ai/ha, 147-day PHI); the residues in both were <0.05 mg/kg. One trial in France was according to maximum Spanish GAP (0.21 kg ai/ha, with weeds at 2-4-leaf stage) and the residue was 0.03 mg/kg.

Chick-pea (dry). Three supervised trials in Australia (0.1 kg ai/ha, 78-99 days PHI) approximated maximum Australian GAP (0.1 kg ai/ha, 98-day PHI). The residues were <0.03, 0.03 and 0.04 mg/kg.

Common bean (dry). One Australian trial (0.16 kg ai/ha, 75-day PHI, 6-leaf stage) was comparable with maximum Australian GAP (0.16 kg ai/ha, 91-day PHI). The residue was 0.03 mg/kg. The Meeting considered that conditions in another Australian trial with applications at early budding were unpractical.

Field pea (dry). Two Australian supervised trials (0.21 kg ai/ha, 93-94-day PHI) could be used in the estimation of an STMR although the doses were higher than the maximum Australian rate (0.16 kg ai/ha, 91-day PHI) because the residues were below the limit of determination of 0.01 mg/kg.

Six supervised trials were carried out with racemic haloxyfop in France according to maximum Spanish GAP (0.21 kg ai/ha, with weeds at 2-4-leaf stage). The residues were <0.02 (3), <0.05, 0.06 and 0.14 mg/kg.

Two supervised trials in Australia with haloxyfop-R (0.1 kg ai/ha, 93-94-day PHI) were comparable with maximum Australian GAP (0.078 kg ai/ha, 91-day PHI). The residues in both were <0.01 mg/kg.

One trial in France (0.1 kg ai/ha, 68-day PHI) and one in Germany (0.1 kg ai/ha, 60-day PHI) were comparable with maximum GAP in East European countries (0.13 kg ai/ha, 60-day PHI or 0.16 kg ai/ha, up to closing of canopy). The residues were 0.06 and 0.03 mg/kg.

Lupin (dry). Four Australian supervised trials with racemic haloxyfop-etotyl at 0.078-0.12 kg ai/ha, with 92-115 days PHI were comparable with maximum Australian GAP (0.1 kg ai/ha, 119-day PHI). The residues were <0.05 (2), 0.03 and 0.11 mg/kg. One supervised trial with haloxyfop-R-methyl in Australia at 0.052 kg ai/ha, 92-day PHI, approximated maximum Australian GAP (0.052 kg ai/ha, 119-day PHI). The residue was 0.05 mg/kg.

Soya bean (dry). Three supervised trials (0.16 kg ai/ha, 102-122-day PHI) with racemic haloxyfop in Australia complied with maximum GAP (0.16 kg ai/ha, 119-day PHI). The residues were all <0.03 mg/kg.

Five supervised trials (0.12 kg ai/ha, 97-110-day PHI) in Brazil with racemic haloxyfop were according to maximum GAP conditions (0.12 kg ai/ha, 98-day PHI). The residues were <0.05 (4) and 0.06 mg/kg.

Four French and three Italian trials with haloxyfop-R at 0.1 kg ai/ha with PHIs of 76-133 days were according to maximum French GAP (0.1 kg ai/ha, up to early tillering). The residues were <0.02 (5), 0.07 and 0.09 mg/kg.

The haloxyfop residues in pulses from the 39 trials in rank order were <0.01 (4), <0.02 (8), <0.03 (4), 0.03 (5), 0.04, <0.05 (9), 0.05, 0.06 (3), 0.07, 0.09, 0.11 and 0.14 mg/kg. The Meeting estimated an STMR of 0.03 mg/kg for haloxyfop in pulses (dry).

Potatoes. Nineteen supervised trials were carried out with racemic haloxyfop in Belgium, Germany, The Netherlands, Norway, Sweden and the UK according to maximum Irish GAP (0.21 kg ai/ha, application up to 60 cm height of crop).

The residues from the 19 trials in rank order were <0.01 (3), 0.01, 0.02, 0.03 (3), 0.04 (5), 0.05, 0.06, 0.07 (3) and 0.1 mg/kg. The Meeting estimated an STMR of 0.04 mg/kg for haloxyfop in potatoes.

Sugar beet, fodder beet and sugar beet leaves or tops

The Meeting concluded that supervised trials on sugar beet and fodder beet could be evaluated together because the use pattern of haloxyfop on these crops is the same and the residue behaviour is expected to be similar.

Sugar beet. Twelve supervised trials were carried out in France and 13 in the UK with racemic haloxyfop according to maximum French GAP (0.21 kg ai/ha, up to early weed tillering). In the French trials the residues in the roots were <0.01, <0.02 (3), 0.02, <0.03 (3), 0.03 (2), 0.05 and 0.1 mg/kg. In the UK trials the residues in the roots were <0.01(3), 0.01 (2), 0.02 (2), <0.03, 0.03 (2), 0.05 (2) and 0.23 mg/kg and in the leaves or tops <0.02 (3), 0.02, <0.03 (3), 0.03, 0.04 (2), 0.09, 0.11 and 0.28 mg/kg.

Eight supervised trials were carried out with racemic haloxyfop in Germany according to maximum GAP (0.21 kg ai/ha, 90-day PHI). The residues in the roots were <0.005, 0.01 (3), 0.02, 0.04, 0.14 and 0.16 mg/kg and in the leaves or tops <0.01, <0.02 (2), 0.03, 0.04, 0.08, 0.28 and 0.3 mg/kg.

In seven supervised trials with haloxyfop-R in France, Germany and Italy according to maximum French GAP (0.1 kg ai/ha, up to early weed tillering) the residues in the roots were 0.01, <0.02 (3), 0.02, 0.03 and 0.06 mg/kg. Residues in the leaves or tops in four of the trials were <0.02, 0.09 (2) and 0.14 mg/kg.

Fodder beet. In five supervised trials with racemic haloxyfop in Germany according to maximum GAP (0.21 kg ai/ha, 90-day PHI) the residues in the roots were <0.01 (2), 0.01, 0.03 and 0.04 mg/kg and in the leaves or tops <0.02 (3), 0.03 and 0.05 mg/kg.

The residues in the roots from the 45 trials in rank order were <0.005, <0.01 (6), 0.01 (6), <0.02 (6), 0.02 (5), <0.03 (4), 0.03 (6), 0.04 (2), 0.05 (3), 0.06, 0.1, 0.14, 0.16 and 0.23 mg/kg.

The residues in the leaves or tops from 30 of the trials in rank order were <0.01, <0.02 (9),

0.02, <0.03 (3), 0.03 (3), 0.04 (3), 0.05, 0.08, 0.09 (3), 0.11, 0.14, 0.28 (2) and 0.3 mg/kg. However, because the Meeting did not estimate a maximum residue level for the leaves or tops, no STMR was estimated.

The Meeting estimated an STMR of 0.02 mg/kg for haloxyfop in sugar beet and fodder beet.

Rice. In nine supervised trials on rice with racemic haloxyfop in Brazil, Colombia, Mexico and Costa Rica the application rates were comparable with the maximum rates in some South American countries of 0.11 kg ai/ha. All the residues were <0.01 mg/kg.

The Meeting estimated an STMR of 0 mg/kg for haloxyfop in husked and polished rice taking into consideration that no residue was found even in trials carried out at an excessive dose rate.

Cotton seed. Four supervised trials with racemic haloxyfop in Australia according to maximum Australian GAP (0.16 kg ai/ha, 119-day PHI). The residues were <0.05 (2), 0.06 and 0.08 mg/kg. Conditions in four supervised trials carried out with racemic haloxyfop in Brazil (0.24 kg ai/ha applied 22-40 days after planting, 93-112-day PHI) were comparable with maximum GAP in Paraguay (0.18 kg ai/ha, applied with weeds at 2-4 leaf stage). The residues were <0.1 (2), 0.1 and 0.2 mg/kg.

The residues from the 8 trials in rank order were <0.05 (2), 0.06, 0.08, <0.1 (2), 0.1 and 0.2 mg/kg. The Meeting estimated an STMR of 0.09 mg/kg for haloxyfop in cotton seed.

Peanuts. Two trials with racemic haloxyfop in Argentina were at dose rates of 0.24 and 0.4 kg ai/ha, comparable to maximum GAP (0.3 kg ai/ha, weeds at 2-4-leaf stage). The residues were 0.03 and <0.05 mg/kg.

Three supervised trials with racemic haloxyfop in Australia were at 0.12-0.16 kg ai/ha, 98-117 days PHI, similar to maximum GAP (0.16 kg ai/ha, 119 days PHI). The residues were <0.03 and 0.03 (2) mg/kg.

The residues from the 5 trials were <0.03, 0.03 (3) and <0.05 mg/kg.

The Meeting estimated an STMR of 0.03 mg/kg for haloxyfop in peanuts.

Rape seed and rape fodder. Two supervised trials with racemic haloxyfop in Australia at a rate of 0.16 kg ai/ha, slightly higher than maximum GAP (0.1 kg ai/ha, 119-day PHI). In this case the influence of the dose rate on the residue of haloxyfop is assumed to be little, since the application was made at an early growth stage (2-6 leaves) causing less direct exposure of the crops to haloxyfop. The Meeting therefore concluded that the trials were comparable with GAP. The residues in the rape seed were <0.03 and 0.07 mg/kg.

Thirteen supervised trials were carried out with racemic haloxyfop in France according to maximum French GAP (0.21 kg ai/ha, up to early tillering). The residues in the rape seed were <0.05 (7), 0.05, 0.09, 0.14, 0.145, 0.37 and 0.66 mg/kg. Seven of the French trials also included treatments comparable with maximum Spanish GAP (0.42 kg ai/ha, applied with weeds at 2-4 leaf stage). The residues in the seed were <0.05, 0.05 (2), 0.17, 0.315, 0.32 and 1.68 mg/kg.

Six supervised trials with racemic haloxyfop in Germany were according to maximum GAP (0.21 kg ai/ha, post weed emergence). The residues were <0.05, 0.1, 0.13 (2), 0.15 and 0.77 mg/kg in

the seed and <0.05 (2), 0.09 and 0.12 mg/kg in four samples of fodder.

Eighteen supervised trials (three trials in 1984 were counted as two trials each, because the applications were made in the year before harvest or the year of harvest) with racemic haloxyfop in the UK were according to maximum French or German GAP (0.21 kg ai/ha, up to early tillering or post weed emergence). The residues were <0.05 (11), 0.05, 0.06, 0.09, 0.1, 0.11, 0.44 and 0.64 mg/kg in the seed and <0.05 (9), 0.05, 0.07 and 0.08 mg/kg in twelve samples of fodder.

Three supervised trials with haloxyfop-R in France and two in Germany which complied with maximum French GAP (0.1 kg ai/ha, up to early tillering) showed residues in the seed of <0.05 (4) and 0.07 mg/kg. The residues in rape fodder in the German trials were both <0.05 mg/kg.

The residues in the rape seed from the 51 trials in rank order were <0.03, <0.05 (24), 0.05 (4), 0.06, 0.07 (2), 0.09 (2), 0.1 (2), 0.11, 0.13 (2), 0.14, 0.145, 0.15, 0.17, 0.315, 0.32, 0.37, 0.44, 0.64, 0.66, 0.77 and 1.68 mg/kg. However the residues in the seven French trials in which application was at the maximum Spanish GAP rate (0.42 kg ai/ha) seem to be from a different population from the others. The Meeting concluded that an STMR for haloxyfop in rape seed should be estimated form this higher population. The residues in rape seed from these 7 trials in rank order were <0.05, 0.05 (2), 0.17, 0.315, 0.32 and 1.68 mg/kg.

The residues in rape fodder from 18 trials in rank order were <0.05 (13), 0.05, 0.07, 0.08, 0.09 and 0.12 mg/kg. The Meeting estimated an STMR of 0.17 mg/kg for haloxyfop in rape seed.

Sunflower seed. Eight supervised trials were carried out with racemic haloxyfop in Argentina, Australia and France at the relevant maximum GAP (rates of 0.3, 0.16 and 0.21 kg ai/ha respectively). The residues were <0.03 (2), 0.03, 0.04, <0.05 (2), 0.143 and 0.16 mg/kg.

One supervised trial with haloxyfop-R in France at the maximum GAP rate of 0.1 kg ai/ha gave a residue of 0.07 mg/kg.

The residues from the 9 trials in rank order were <0.03 (2), 0.03, 0.04, <0.05 (2), 0.07, 0.143 and 0.16 mg/kg. The Meeting estimated an STMR of 0.05 mg/kg for haloxyfop in sunflower seed.

Alfalfa. In two supervised trials with racemic haloxyfop in Australia the conditions (0.21 kg ai/ha, 21-22 days PHI) were comparable with maximum GAP (0.16 kg ai/ha, 21-day PHI). The residues were 2.45 and 3.11 mg/kg. In two further trials with haloxyfop-R in Australia the conditions (0.1 kg ai/ha, 22-day PHI) were again comparable with maximum GAP (0.078 kg ai/ha, 21-day PHI) and the residues were 1.8 and 2.21 mg/kg.

The residues from the 4 trials in rank order were 1.8, 2.21, 2.45 and 3.11 mg/kg.

Pasture. Four supervised trials with racemic haloxyfop and two with haloxyfop-R in Australia were according to maximum GAP (0.1 kg ai/ha racemic haloxyfop, 0.052 kg ai/ha haloxyfop-R, 7-day PHI in both cases). The residues from the 6 trials in rank order were 0.49, 0.99, 1.47, 1.71, 2.04 and 3.35 mg/kg.

Processing

Sugar beet. Two processing studies were carried out and no residues of haloxyfop (<0.01 mg/kg)

were found in sugar derived from sugar beet containing 0.07 and 0.11 mg/kg.

The Meeting estimated an STMR-P of 0.002 mg/kg for haloxyfop in sugar.

The concentration factors for pressed pulp were 0.36 and 0.43. The Meeting estimated an STMR-P of 0.008 mg/kg for haloxyfop in pressed pulp by applying the mean concentration factor (0.4) to the sugar beet STMR of 0.02 mg/kg.

Soya beans. Concentration factors from 4 trials were 0.75, 1.19, 1.25 and 1.31 for meal, 0.375, 0.41, 0.79 and 1.25 for crude oil and 0.33, 0.375, 0.75 and 1.22 for refined oil, giving mean factors of 1.13, 0.71 and 0.67 respectively. The Meeting estimated STMR-P levels of 0.03, 0.02 and 0.02 mg/kg for meal, crude oil and refined oil respectively by calculation from the STMR for pulses (0.03 mg/kg).

Rice. The residues in rice bran from normally treated rice were <0.02 mg/kg, and the Meeting estimated an STMR-P of 0.02 mg/kg for rice bran, unprocessed.

Cotton seed. Concentration factors for crude oil from 3 trials were 0.88, 1.0 and 1.6, giving a mean of 1.16. The Meeting estimated an STMR-P of 0.10 mg/kg for crude oil from the STMR for cotton seed of 0.09 mg/kg.

Rape seed. Concentration factors from 4 trials were 0.72, 0.89, 0.92 and 0.93 for meal or cake and 1.43, 1.97, 2.34 and 2.79 for crude oil (residues in pressed oil were not used for calculation of the concentration factors because the process is not current commercial practice). The factors for refined oil were 1.07 and 2.19. The Meeting estimated STMR-P levels of 0.15, 0.36 and 0.28 mg/kg for meal, crude oil and refined oil from the mean concentration factors of 0.87, 2.13 and 1.63 respectively and the STMR for rape seed of 0.17 mg/kg.

Note - Correction to report of 1995 JMPR

The concentration factors of 1.7 for crude oil and 2.1 for refined oil should be replaced by 2.13 for crude and 1.63 for refined oil.

Products of animal origin

Cattle. The Meeting was aware that the dosing levels in the feeding studies evaluated by the 1995 JMPR were expressed on a dry-weight basis, whereas the provisional maximum residue levels for the feed items were estimated on a wet-weight basis. The Meeting therefore reconsidered the conclusions of the 1995 Meeting with respect to residues in cattle products.

Fodder beet, alfalfa, pasture, sugar beet tops, pulses, rape fodder and processed fractions of oil seed and sugar beet can be used as feed for beef and dairy cattle, but the maximum haloxyfop intake would result from consuming 100% of pasture. The maximum residue found in pasture was 3.35 mg/kg (1995 Residue Evaluations, p.488), and with an assumed 80% moisture content this would be equivalent to 16.75 ppm in the feed on a dry-weight basis.

Since this feed level is higher than the highest level in the feeding studies (beef calves 10 ppm; lactating cows 2.5 ppm), the Meeting could not confirm the maximum residue levels for cattle products that were estimated by the 1995 JMPR and agreed to withdraw the provisional estimates for these commodities.

Poultry. Pulses and processed fractions of pulses and oil seed can be used as feed for poultry. Cereals are the main feed items, but the feed could contain up to 50% of pulses, 7% of rape seed meal and 30% of soya bean meal, and this composition would provide the maximum haloxyfop intake. The median intake level for this feed composition was calculated from the STMR for each feed item (pulses 0.03 mg/kg, rape seed meal 0.15 mg/kg and soya bean meal 0.03 mg/kg) to be 0.035 ppm (dry weight basis).

The residues in the muscle, liver, fat and eggs at a feeding level of 0.035 ppm were estimated from control residues (<0.01 mg/kg in each product) and the highest residues found in each product in the feeding study at 0.25 ppm by extrapolation to be <0.01, 0.01, 0.01 and <0.01 mg/kg respectively.

The Meeting confirmed the 1995 estimates of maximum residue levels in poultry products and estimated an STMR of 0.01 mg/kg for haloxyfop in chicken meat, chicken edible offal and chicken eggs.

Residues in rotational crops

Comprehensive studies were conducted with six rotational crops, using labelled or unlabelled haloxyfop. When lettuce, sugar beet and wheat were planted as rotational crops 25-148 days after treating soya beans or cotton as primary crops with unlabelled haloxyfop at a rate of 0.28 or 0.56 kg ai/ha, no residues were found in any of the mature rotational crops except green wheat forage at the LOD of 0.01 mg/kg, 110 days after treatment with 0.28 kg ai/ha. The limit of determination was 0.01 mg/kg for all substrates except wheat straw, for which it was 0.02 mg/kg.

When lettuce, wheat, soya beans, carrots or turnips were grown to maturity in soil which had been treated with phenyl-ring-labelled haloxyfop at 0.56 kg ai/ha 30 days before planting, the highest radioactive residues in the edible portions were found in lettuce and wheat grain and were 0.01 mg/kg haloxyfop equivalent. The radioactivity was too low for identification of the residue. 130 days after treatment of soya bean plants with phenyl-ring-labelled haloxyfop, the top 5 cm of soil was transferred to pots and sown with lettuce, soya bean, wheat and sugar beet in the laboratory. The total radioactivity was 0.01, 0.04, 0.02 and 0.01 mg/kg haloxyfop equivalent in lettuce, soya bean, wheat and sugar beet respectively and 0.02, 0.05, 0.02 and 0.02 mg/kg in soya bean forage, soya bean straw, wheat straw and sugar beet forage respectively. Again, the residue could not be identified owing to the low level of radioactivity.

The pyridinol 3-chloro-5-trifluoromethylpyridin-2-ol was found in soil as a major terminal degradation product under aerobic conditions (1995 Residue Evaluations, p.415) but it was not detected in the plants at harvest in any of the plant metabolism studies, although these included experiments with pyridinol-labelled haloxyfop.

The submitted data indicated that haloxyfop and its soil degradation products would not be absorbed or accumulate in plants to any significant extent.

The Meeting noted that the residues found in supervised trials on fodder crops reviewed by the 1995 JMPR were expressed on a wet-weight basis, although the Codex Classification of Food and Feeds indicates that MRLs for fodder and forage should preferably be set and expressed on a dry-weight basis. As the Meeting did not have information on the moisture content of the fodder crops for which the 1995 JMPR estimated provisional maximum residue levels, it agreed to withdraw the provisional estimates for fodder crops.

RECOMMENDATIONS

The Meeting estimated the maximum residue levels, STMR levels and STMR-P levels listed in the Tables below. The maximum residue levels are recommended for use as MRLs.

Definition of the residue, for compliance with MRLs and for estimation of dietary intake: haloxyfop esters, haloxyfop and its conjugates expressed as haloxyfop.

Commodity		Recommended MRL, mg/kg		PHI, days	Estimated STMR, for dietary intake estimation, mg/kg
CCN	Name	New	Previous max. residue level[1]		
AL 1021	Alfalfa forage (green)	W	5	21	
FI 0327	Banana	0.05*	0.05*	-	0
FC 0001	Citrus fruits	0.05*	0.05*	-	0
SO 0691	Cotton seed	0.2	0.2	-	0.09
OC 0691	Cotton seed oil, crude	0.5	0.5	-	0.1 (STMR-P)
AM 1051	Fodder beet	0.3	0.3	90	0.02
AV 1051	Fodder beet leaves or tops	W	0.3	90	
FB 0269	Grapes	0.05*	0.05*	-	0
SO 0697	Peanut	0.05	0.05	-	0.03
VP 0063	Peas (pods and succulent seeds)	0.2	-	-	0.02
FP 0009	Pome fruits	0.05*	0.05*	-	0
VD 0070	Pulses (dry)	0.2	0.2	-	0.03
VR 0589	Potato	0.1	0.1	-	0.04
SO 0495	Rape seed	2	2	-	0.17
OC 0495	Rape seed oil, crude	5	5	-	0.36 (STMR-P)
OR 0495	Rape seed oil, edible	5	5	-	0.28 (STMR-P)
CM 1206	Rice bran, unprocessed	0.02*	0.02*	-	0.02 (STMR-P)
CM 0649	Rice, husked	0.02*	0.02*	-	0
CM 1205	Rice, polished	0.02*	0.02*	-	0
OC 0541	Soya bean oil, crude	0.2	0.2	-	0.02 (STMR-P)
OR 0541	Soya bean oil, refined	0.2	0.2	-	0.02 (STMR-P)
VR 0596	Sugar beet	0.3	0.3	-	0.02
AV 0596	Sugar beet leaves or tops	W	0.3	-	
SO 0702	Sunflower seed	0.2	0.2	-	0.05
MM 0812	Cattle meat	W	0.01	-	
MO 0812	Cattle, Edible offal of	W	0.5	-	
MF 0812	Cattle fat	W	0.1	-	

Commodity		Recommended MRL, mg/kg		PHI, days	Estimated STMR, for dietary intake estimation, mg/kg
CCN	Name	New	Previous max. residue level[1]		
ML 0812	Cattle milk	W	0.05	-	
FM 0812	Cattle milk fat	W	0.5	-	
PM 0840	Chicken meat	0.01*	0.01*	-	0.01
PO 0840	Chicken, Edible offal of	0.1	0.1	-	0.01
PE 0840	Chicken eggs	0.01*	0.01*	-	0.01

[1] Provisional estimates of maximum residue levels made by the 1995 JMPR but not recommended for use as MRLs.

Raw agricultural commodity	STMR, mg/kg	Processed commodity	STMR-P, mg/kg
Sugar beet	0.02	Refined sugar	0.002
		Sugar beet pressed pulp	0.008
Rape seed	0.17	Rape seed meal	0.15
Soya bean	0.03 (Pulses (dry))	Soya bean meal	0.03

FURTHER WORK ON INFORMATION

Desirable

1. Information on the moisture content of fodder crops.

2. Ruminant feeding studies at a feeding level comparable to the maximum residue level found in fodder crops.

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(All unpublished)

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METHAMIDOPHOS (100)

[see also ACEPHATE (095)]

EXPLANATION

Methamidophos was first evaluated in 1976, with further reviews of residue aspects in 1979, 1981, 1984, 1990 and 1994. The 1994 JMPR withdrew the previous recommendations for MRLs for broccoli, Brussels sprouts, head cabbages, cauliflower, citrus fruits, egg plant, melons except watermelon, peaches and tomato which had been held at Step 7B by the 1992 CCPR (ALINORM 93/24, para 119-123). The manufacturer indicated that information on GAP and residue data would be available to support new MRLs for these commodities. This information was provided to the Meeting, together with information on analytical methods and residues in food in commerce or at consumption.

METHODS OF RESIDUE ANALYSIS

Analytical methods

In the supervised trials homogenized samples were extracted with ethyl acetate or a water-acetone mixture. The water-acetone extract was filtered and, after the addition of sodium chloride, partitioned with chloroform or methylene chloride. The samples were cleaned up on a silica gel column and analysed by GLC (Leary, 1971; Möllhoff, 1971; Luke, 1975; Lai and Fowler, 1989; Blass, 1994).

Recoveries of both methamidophos and acephate were generally >70% and the limit of determination was 0.01-0.02 mg/kg.

Stability of pesticide residues in stored analytical samples

The stability of methamidophos was studied in vegetables, pulses, oil seed, animal products, cereals and grasses as part of the stability studies on acephate. All samples except pinto beans and eggs were from crops or animals which had been treated with acephate. Pinto beans and eggs were fortified. Storage was for periods ranging from 28 days to more than a year at -20°C (Lai, 1987, 1988, 1989).

The results of the trials did not establish the stability of methamidophos in the treated commodities, because they contained substantially higher residues of acephate than of methamidophos and there was a possibility that acephate was degraded to methamidophos during storage. Methamidophos was stable in pinto beans and eggs at -20°C for periods of 461 and 175 days respectively, but was unstable in cow kidneys and cotton seed. The results are given in Table 1.

Table 1. Stability of acephate and methamidophos in samples stored at -20°C.

Commodity	Compound[1]	Storage period, days	Initial concentration[2], mg/kg	% of initial residue remaining	Reference
Celery	A	364	0.26-4.40	87-97	56
	M	364	0.02-0.29	243-300	
Celery	A	94	4.16-4.40	106-116	55
	M	94	0.23-0.29	93-148	
Snap beans	A	548	0.30-0.39	76.7-82.1	57
	M	548	0.12-0.15	75.0-80.0	
Snap beans	A	69	0.30-0.39	73.3-84.6	55
	M	69	0.12-0.15	75.0-86.7	
Pinto beans (dry)	A	461	0.23-0.24[3]	95.0-95.0	57
	M	461	0.09-0.10[3]	80.0-90.0	
Pigeon peas	A	418	8.11-9.74	104-110	55
	M	418	0.94-1.07	108-111	
Bell peppers	A	386	3.67-3.83	103-112	
	M	386	0.51-0.53	131-136	
Brussels sprouts	A	272	1.61-2.06	84-88	
	M	272	0.03-0.03	100-100	
Cotton seed	A	48	0.38-0.82	73.2-86.8	
	M	48	0.02-0.03	0.0-0.0	
Grass	A	269	0.52-0.70	78.6-100	
	M	269	0.10-0.14	78.6-90	
Bermuda grass	A	61	0.62-0.72	108.1-122.2	
	M	61	0.11-0.11	109.1-116.7	
Bermuda grass	A	60	1.88-2.85	98.2-101.6	
	M	60	0.31-0.44	102.3-106.5	
Fresh hay	A	58	6.95-7.36	72.0-85.8	
	M	58	0.49-0.54	75.5-83.3	
Spent hay	A	58	2.81-2.91	96.2-96.4	
	M	58	0.33-0.36	90.9-91.7	
Lettuce	A	28	0.29-0.31	84-93	
	M	28	0.02-0.02	50-100	
Rice grain	A	506	1.09-1.19	81-126	
	M	506	0.21-0.23	96-124	
Rice straw	A	507	0.17-0.21	90-94	
	M	507	0.06-0.06	83-83	
Eggs	A	175	0.15-0.16[3]	96.8-103	55
	M	175	0.07-0.08[3]	93.3-93.3	
Cow milk	A	202	0.04-0.79	98.7-150	
	M	202	0.02-0.12	58.3-100	
Cow kidneys	A	172	0.26-0.73	71.2-73.1	
	M	172	0.02-0.07	50.0-60.0	
Cow muscle	A	193	0.11-0.40	90.5-112	
	M	193	0.01-0.03	100-100	

[1] A: Acephate M: Methamidophos
[2] Initial concentrations were the residues found in the commodity at harvest or collection, except in pinto beans and eggs in which acephate and methamidophos were added to the untreated commodities
[3] Fortified separately with acephate and methamidophos

USE PATTERN

Information on use patterns was provided by the governments of Germany, The Netherlands and Poland and the manufacturers.

The use patterns for peaches, broccoli, head cabbages, cauliflowers, melons, egg plants and tomatoes are shown in Table 2.

Table 2. Registered uses of methamidophos on peaches, broccoli, head cabbages, cauliflowers, melons, egg plants and tomatoes. All spray applications.

Crop	Country	Form.	Application				PHI, days
			kg ai/ha	kg ai/hl	No.	Interval, days	
Peaches	Australia	EC		0.029	-	-	21
	France	SL	0.5				21
	Greece	SL	0.68				21
	Italy	EC	0.38-0.6				21
	Portugal	SL	0.8-1.0				21
	Spain	SL	0.75-1.1	0.05-0.075			35
	Uruguay	SL		0.036-0.06			28
Broccoli	Brazil	SL	0.3-0.6	0.06			21
	Canada	SL	0.53-1.1				14
	Mexico	SL	0.6-0.9				21
	USA	SL	0.56				14
	USA	SL	>0.56-1.1				21
	Venezuela	SL	0.24	0.06-0.12			14
Cabbages	Australia	SL	0.64-1.2	0.058-0.11	-	10	7
	Bolivia	EC	0.6				14
	Brazil	SL	0.3-0.6	0.06			21
	Canada	SL	0.53-1.1				7
	El Salvador	SL	0.6-0.84	0.06-0.09			15
	Germany	SL	0.36	0.09	2		14
	Indonesia	LC	0.19-0.49				14
	Mexico	SL	0.6-0.9				35
	New Zealand	SL	0.6-0.9				7
	Philippines	SL	0.56-1.9	0.11-0.19			15
	Poland	SL	0.22	0.036-0.11	1		21
	Thailand	SL	0.6-1.2				21
	USA	SL	0.56-1.1				35
Cauliflowers	Australia	SL	0.64-1.2	0.058-0.11	-	10	7
	Brazil	SL	0.3-0.6	0.06			21

Crop	Country	Form.	Application				PHI, days
			kg ai/ha	kg ai/hl	No.	Interval, days	
	Canada	SL	0.53-1.1				7
	Germany	SL	0.36	0.09	2		21
	Greece	SL	0.48-0.72	0.06-0.09			21
	Mexico	SL	0.6-0.9				28
	Philippines	SL	0.56-1.9	0.11-0.19			15
	USA	SL	0.56-1.1				28
	Venezuela	SL	0.24	0.06-0.12			14
Melons	Dominican Republic	SL	0.36				3
	Ecuador	SL	0.48-0.6				15
	Mexico	SL	0.6-0.9				7
	Uruguay	SL		0.048			14
	Venezuela	SL	0.6				14
Egg plants	Greece	SL	0.48-0.72				21
	Mexico	SL	0.6-0.9				14
	Philippines	SL	0.56-1.9	0.11-0.19			28
	Thailand	SL	0.6-1.2				21
	Venezuela	SL	0.6				14
Tomatoes	Australia	SL	0.32-1.2	0.029-0.11	-	14	4
	Brazil	SL	0.3-0.6	0.06			21
	Chile	SL	0.3-0.6				15
	Dominican Republic	SL	0.6-0.9				14
	Ecuador	SL	0.48-0.6				15
	El Salvador	SL	0.6-0.84	0.06-0.09			7
	Greece	SL	0.48-0.72				21
	Guatemala	SL	0.6-0.84	0.06-0.09			21
	Honduras	SL		0.06-0.09			21
	Mexico	SL	0.6-0.9				7
	New Zealand	SL	0.6-0.9				3
	Nicaragua	SL	0.6-0.84	0.06-0.09			21
	Peru	SL	0.6-1.2	0.12-0.18			14
	Philippines	SL	0.56-1.9	0.11-0.19			28
	Portugal	SL	0.6-1.2				21
	Spain	SL		0.05-0.075			7
	Thailand	SL	0.45-0.9				21
	Uruguay	SL	0.24-0.48	0.024-0.048			28
	Venezuela	SL	0.6				14

RESIDUES RESULTING FROM SUPERVISED TRIALS

Data from many supervised trials on peaches, broccoli, head cabbages, cauliflowers, melons, egg plants and tomatoes were submitted or resubmitted to the Meeting, but some reports lacked important information so the Meeting did not evaluate trials which lacked data on analytical recoveries or those with abnormally high residues in control samples and for which no representative chromatograms were supplied. In such cases. it was not clear whether the control samples were contaminated or the analysis was at fault. The trials which were considered to be unsuitable for evaluation are shaded in the Tables.

Residues in crops

Peaches. Ten supervised trials were carried out in Italy and Spain. The summarized data are shown in Table 3.

Table 3. Residues of methamidophos in peaches. All SL formulations.

Country Year	Application			PHI, days	Residues	Reference
	No.	kg ai/ha	kg ai/hl			
Italy 1994	2	0.68	0.057	0[1]	0.19	36
				0	1.1	
				7	0.41	
				14	0.26	
				21	0.16	
				28	0.11	
Italy 1994	2	0.68	0.057	0[1]	0.13	37
				0	0.67	
				21	0.12	
				28	0.06	
Italy 1994	2	0.6	0.05	0[1]	0.12	34
				0	0.39	
				21	0.09	
				28	0.04	
Italy 1994	2	0.6	0.05	0[1]	0.41	31
				0	0.95	
				7	0.55	
				14	0.28	
				21	0.27	
				28	0.13	
Spain 1994	2	0.6	0.048	0[1]	0.26	33
				0	1.3	
				6	0.59	
				14	0.27	
				21	0.09	
				28	0.09	
Spain 1994	2	1	0.054	0[1]	0.6	35
				0	1	
				21	0.27	
				28	0.15	
	1	0.72	0.048	28	<0.01	1

Country Year	Application			PHI, days	Residues	Reference
	No.	kg ai/ha	kg ai/hl			
Spain 1995				35	<0.01	
	1	1.1	0.072	28	0.11	
				35	0.07	
Spain 1995	1	0.86	0.048	28	0.12	1
				35	0.24	
	1	1.3	0.072	28	0.4	
				35	0.76	

[1] Sampling just before last application

Broccoli. Eighteen supervised trials were carried out in Brazil, Canada, Mexico and the USA. The results are shown in Table 4.

Table 4. Residues of methamidophos in broccoli.

Country Year	Application				PHI, days	Residues[1]	Reference
	Form.	No.	kg ai/ha	kg ai/hl			
Brazil 1987	SL	5	0.6	0.06	0	1.6	27[2]
					14	<0.01	
					21	<0.01	
					28	<0.01	
		5	1.2	0.12	21	<0.01	
Brazil 1995	SL	3	0.6	0.06	21	0.2	76
		3	1.2	0.12	21	0.6	
Canada 1995	EC	3	1.08		14	0.01, 0.01	79[3]
Canada 1972	EC	8	1.12	0.12	0	13.05	2[4]
					3	5.87	
					7	1.88	
					14	0.13	
Canada 1972	EC	8	1.12	0.14	0	0.95	3[4]
					3	0.94	
					7	0.99	
					14	0.41	
Mexico 1995	EC	2	0.9	0.3	21	<0.01, 0.08	46[4]
USA 1974	SC	5	1.12		0	2.15(H), 2.85(L)	12[4]
					7	0.92(H), 3.64(L)	
					14	0.42(H), 2.24(L)	
					21	0.14(H), 0.88(L)	
					28	0.04(H), 0.45(L)	
USA 1974	SC	3	1.12	0.48	21	0.06(H), 0.75(L)	11[4]
					28	0.09(H), 0.54(L)	

Country Year	Application Form.	No.	kg ai/ha	kg ai/hl	PHI, days	Residues[1]	Reference
					35	0.02(H), 0.24(L)	
					42	0.03(H), 0.07(L)	
	SC	3	1.12	0.34	14	0.03(H), 0.74(L)	4[4]
USA 1973					21	0.01(H), 0.11(L)	
					28	<0.01(H), 0.06(L)	
					35	<0.01(H), 0.03(L)	
					42	<0.01(H), <0.01(L)	
	SC	6	1.12	0.34	0	1.55(H), 7.15(L)	6[4]
USA 1973					7	1.10(H), 0.01(L)	
					14	0.12(H), 0.22(L)	
					21	0.02(H), 0.11(L)	
					28	<0.01(H), 0.02(L)	
	SC	3	1.12	0.2	14	<0.01(H), 0.54(L)	8[4]
USA 1973					21	<0.01(H), 0.06(L)	
					27	0.02(L)	
	SC	3	1.12	0.27	14	2.84(H), 4.98(L)	9[4]
USA 1974					21	0.01(H), <0.01(L)	
					26	<0.01(H), <0.01(L)	
					35	0.02(H), 0.04(L)	
					42	<0.01(H), 0.02(L)	
	SC	3	1.12	0.12	14	0.28	14[4]
USA 1973					21	0.02	
					28	0.03	
					35	0.01	
	SC	5	1.12	0.2	0	1.32(H), 22.93(L)	5[4]
USA 1973					7	0.33(H), 2.51(L)	
					13	0.15(H), 1.14(L)	
	SC	5	1.12	0.12	0	1.52	13[4]
USA 1974					7	0.21	
					14	0.05	
					21	0.01	
	SC	5	1.12	0.27	0	2.65(H), 7.3(L)	10[4]
USA 1974					7	2.53(H), 9.07(L)	
					14	1.27(H), 7.62(L)	
					21	<0.01(H), 0.08(L)	
					28	<0.01(H), <0.01(L)	

[1] (H) head (L) leaves
[2] The data were also submitted to the 1990 JMPR
[3] The 2 results were from duplicate plots. The higher values of each pair were used to estimate both maximum residue levels and STMRs
[4] No data on analytical recoveries

Head cabbages. Six supervised trials were carried out in Argentina, Brazil, Germany and Mexico. The results are shown in Table 5.

Table 5. Residues of methamidophos in head cabbages.

Country, Year	Application				PHI, days	Residues[1]	Reference
	Form.	No.	kg ai/ha	kg ai/hl			
Argentina 1995	EC	1	0.6	0.16	14 21 30	<0.05 <0.05 <0.05	63[1]
Brazil 1995	SL	3	0.6	0.06	21	0.08	77
		3	1.2	0.12	21	0.2	
Germany 1977	SL	3	0.36	0.06	0 7 14 21 28	1.95 0.23 0.2 0.06 0.07	15
Germany 1977	SL	3	0.36	0.06	0 7 14 21 28	<0.01 0.16 0.03 0.02 0.05	16
Mexico 1995	EC	2	0.9	0.3	35	<0.01, 0.02	65[1,2]

[1] No data on analytical recoveries
[2] The 2 results were from duplicate plots. The higher value was used to estimate both maximum residue levels and STMRs

Cauliflowers. Eleven supervised trials were carried out in Brazil, France, Germany, Mexico and the USA. The results are shown in Table 6.

Table 6. Residues of methamidophos in cauliflowers.

Country Year	Application				PHI, days	Residues[1]	Reference
	Form.	No.	kg ai/ha	kg ai/hl			
Brazil 1987	SL	6	0.6	0.06	0 14 21 28	2.5 <0.01 <0.01 <0.01	23[2]

Country Year	Application				PHI, days	Residues[1]	Reference
	Form.	No.	kg ai/ha	kg ai/hl			
		6	1.2	0.12	21	<0.01	
	SL	3	0.6	0.06	21	0.5	75
Brazil 1995		3	1.2	0.12	21	0.6	
	SL	1	0.94	0.06	14	0.64, 1.06	53[3]
France 1988		1	0.75	0.06	21	0.29, 0.84	
		2	0.75	0.06	21	0.19, 0.23	
	SL	2	0.36	0.06	0	2.15	17[4]
Germany					3	1.1(H), 1.6(L)	
1978					7	0.55	
					14	0.3	
					21	0.04	
					28	<0.01	
	SL	2	0.36	0.06	0	0.35	18[4]
Germany					3	0.25(H), 1.25(L)	
1978					7	0.07	
					14	0.02	
					21	0.01	
					28	<0.01	
	SL	2	0.36	0.06	0	0.55	19
Germany					7	0.01	
1978					14	<0.01	
					21	≤0.01	
					28	<0.01	
Mexico 1995	EC	2	0.9	0.3	35	0.07, 0.12	66[5]
	SL	9	1.12	N.S	0	0.19(H), 16.05(L)	7[5]
USA 1973					7	0.23(H), 9.38(L)	
					14	0.26(H), 6.01(L)	
					21	0.11(H), 5.5(L)	
					28	0.07(H), 1.55(L)	

[1] (H) head (L) leaves
[2] The data were also submitted to the 1990 JMPR
[3] The 2 results were from duplicate plots
[4] Data were also submitted to the 1981 JMPR
[5] No data on analytical recoveries

Melons. Four supervised trials were carried out in Argentina, Mexico and Spain. The results are shown in Table 7.

Table 7. Residues of methamidophos in melons.

Country Year	Application				PHI, days	Residues[1]	Reference
	Form.	No.	kg ai/ha	kg ai/hl			
Argentina	EC	1	0.6	0.16	14 21	0.98 0.44	70[1]
Mexico 1995	EC	2	0.9	0.3	7	0.23 1.63	71[1,2]
Spain 1995	SL	3	1.2	0.08	7	0.11	72
Spain 1995	SL	3	1.2	0.08	0 3 7	0.32 0.19 0.11	73

[1] No data on analytical recoveries
[2] The 2 results were from duplicate plots

Egg plants. Seven supervised trials were carried out in Argentina, Mexico, Spain and the USA. The results are shown in Table 8.

Table 8. Residues of methamidophos in egg plants.

Country Year	Application				PHI, days	Residues	Reference
	Form.	No.	kg ai/ha	kg ai/hl			
	EC	1	0.6	0.16	14	<0.05	I.67[1]
Argentina					21	<0.05	
1995					30	<0.05	
Mexico 1995	EC	2	0.9	0.3	14	0.77, 0.95	68[1,2]
Mexico 1995	EC	2	0.9	0.3	14	0.28, 0.34	69[1,2]
	EC	4	0.6	0.045	0	0.18	51
Spain 1987					3	0.1	
					7	0.05	
					10	0.04	
	SL	7	1.12	0.3	3	0.17	24[3]
USA 1987					7	0.12	
					14	0.06	
					21	0.04	
	SL	7	1.12	0.08	3	0.11	25[3]
USA 1987					7	0.1	
					14	0.06	
					21	0.03	
	SL	7	1.12	0.38	3	0.13	22[3]
USA 1987					7	0.12	
					14	0.12	

Country Year	Application				PHI, days	Residues	Reference
	Form.	No.	kg ai/ha	kg ai/hl			
					21	0.06	

[1] No data on analytical recoveries
[2] The 2 results were from duplicate plots
[3] Data were also submitted to the 1990 JMPR

Tomatoes. Twenty five supervised trials were carried out in Australia, Brazil, France, Italy, Mexico, Spain and Turkey. The results are shown in Table 9.

Table 9. Residues of methamidophos in tomatoes.

Country, Year	Application				PHI, days	Residues	Reference
	Form.	No.	kg ai/ha	kg ai/hl			
Australia 1995	EC	6	1.2	0.44	1 3 5 7	0.70, 0.77 0.75, 0.96 0.64, 0.74 0.66, 0.74	II.74[1]
		6	2.4	0.88	1 3 5 7	1.20, 1.70 0.96, 3.10 1.70, 1.70 1.90, 4.10	
Brazil 1988	SL	3	0.6	0.06	0 4 7 14 21	0.03 <0.01 <0.01 <0.01 <0.01	26
		3	1.2	0.12	21	<0.01	
Brazil 1995	SL	3	0.6	0.06	21	0.3	78
		3	1.2	0.12	21	1.2	
France 1988	SC	1	0.44	0.06	13 20	0.04, 0.04 0.03, 0.04	54[1]
		2	0.44	0.06	20	0.05, 0.06	
France 1988	SC	1	1.3	0.06	14 21	<0.02, <0.02 0.07, 0.08	52[1]
		2	1.3	0.06	21	<0.02, <0.02	
Italy 1988	EC	2	0.49	0.049	0 14 21	0.053 0.027 0.018	28[2]
Mexico 1995	EC	3	0.9	0.3	7	0.02, 0.03	62[1,2]

Country, Year	Application				PHI, days	Residues	Reference
	Form.	No.	kg ai/ha	kg ai/hl			
Spain 1986		2	0.72	0.045	1 3 7	<0.02 <0.02 <0.02	41
Spain 1986		4	0.73	0.045	1 3 7	0.03 0.03 <0,02	45
Spain 1986	SC	2	1	0.06	1 3 7	0.22 0.44 0.14	46
		4	0.98	0.06	1 3 7	0.15 0.07 0.12	
Spain 1986	SL	2	0.9	0.045	1 3 7	0.51 0.42 0.22	42[3]
Spain 1986	SL	4	0.91	0.045	1 3 7	0.46 0.4 0.47	43[3]
Spain 1986	SL	2	0.73	0.045	1 3 7	0.36 0.37 0.3	44[3]
Spain 1986	SL	4	0.73	0.045	1 3 7	0.84 0.54 0.39	45[3]
Spain 1986	SC	2	1.11	0.06	1 3 7	0.77 0.56 0.54	48[3]
Spain 1986	SC	4	1	0.06	1 3 7	0.83 0.81 0.4	49[3]
Spain 1984 Glasshouse	EC	3	1	0.05	0 3 8 14	0.2 0.22 0.32 0.2	21[4]
Spain 1984 Glasshouse	EC	3	1	0.05	0 3 8 14	0.25 0.25 0.29 0.29	20[4]
Turkey 1989	SL	2	0.6	0.06	0 7 14	0.17 0.1 <0.01	29

[1] The 2 results were from duplicate plots. The higher values from each pair were used to estimate both maximum residues and STMRs
[2] No data on analytical recoveries
[3] Abnormally high control values and no sample chromatograms
[4] The data were also submitted to the 1990 JMPR

FATE OF RESIDUES IN STORAGE AND PROCESSING

In processing

Peaches. Two studies on processing, one carried out in Italy, the other in Spain, were submitted to the Meeting. The harvested peaches were processed in the laboratory according to typical commercial practices, the outlines of which were as follows.

Washed fruit: sorting → washing → washed fruit.

Jam: washed fruit → peeling → stoning → cutting → cooking (3-4 minutes at 100°C) → jam.

Preserve: washed fruit → peeling → stoning → pasteurizing → preserve.

Juice: washed fruit → stoning → crushing → heating → pressing → centrifuging → diluting → pasteurizing → juice.

The results are shown in Table 10.

Table 10. Residues of methamidophos in processed products of peaches.

Field application, Country, Year	Sample	Residues, mg/kg	Reference
0.68 kg ai/ha	Fruit	0.11	36
0.057 kg ai/hl	Washed fruit	0.08	
2 applications	Juice[1]	0.05	
PHI 28 days	Jam	0.10	
Italy 1994	Preserve	0.09	
0.6 kg ai/ha	Fruit	0.09	33
0.048 kg ai/hl	Washed fruit	0.05	
2 applications	Juice[1]	0.02	
PHI 28 days	Jam	0.03	
Spain 1994	Preserve	0.02	

[1] Diluted with same volume water

Vegetables. Cooking studies were carried out on three vegetables containing acephate and methamidophos (Crossley, 1971). Field-treated samples of tomatoes, cabbages and broccoli were analyzed for acephate and methamidophos before and after boiling for 30 minutes. The results are shown in Table 11.

Table 11. Residues of acephate and methamidophos in vegetables before and after 30 minutes boiling.

Commodity	Residues, mg/kg			
	Acephate		Methamidophos	
	Before boiling	After boiling	Before boiling	After boiling
Tomatoes	0.93, 1.13	0.93, 1.09	0.12, 0.14	0.13, 0.15
Cabbages	2.08, 2.20	2.06, 2.08	0.22, 0.22	0.24, 0.25
Broccoli	8.38, 9.92	8.02, 7.12	0.98, 1.17	1.00, 1.10

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

National monitoring data

The government of The Netherlands reported monitoring data on methamidophos in several crops. The results are shown in Table 12.

Table 12. Monitoring data for methamidophos on several crops in The Netherlands, 1991-1994.

Commodity	Samples analyzed	No. in which residues found[1]	Detection frequency, %	Mean residues[2], mg/kg
Peaches	379	14	3.7	0.01, 0.02[3]
Nectarines	288	12	4.2	0.01
Grapes	999	5	0.5	<0.01
Strawberries	2976	2	0.07	<0.01
Tomatoes	330	2	0.6	<0.01
Sweet peppers	571	8	1.4	<0.01
Cucumbers	985	4	0.4	<0.01
Lettuce	865	6	0.7	<0.01, 0.02[3]
Celery	202	1	0.5	<0.01
Beans	1086	3	0.3	<0.01

[1] LOD = 0.01 mg/kg
[2] For samples with residues below the LOD, half the LOD is taken for the calculation of the mean residues
[3] Means for 1991-1993 and 1994 respectively

Market basket surveys

Market basket surveys for acephate and methamidophos were carried out in the USA in 1984 and 1985 (Lai, 1989c). From 26 to 62 commodities including fresh vegetables, fresh fruit, canned food,

meat and dairy products were collected from 24 locations. Acephate and methamidophos were found at or above the limit of determination (0.01 mg/kg) in samples of 6 and 7 commodities respectively. The residues found are shown in Table 13.

Table 13. Residues of acephate and methamidophos found at or above the limit of determination in market basket surveys carried out in the USA, 1984 and 1985 (Lai, 1989c).

Commodity	Residues, mg/kg		Reference
	Acephate	Methamidophos	
Cantaloupe	0.03	0.02, 0.02, 0.03, 0.10	57-3
Celery	0.01, 0.03, 0.04	0.04	
Cucumbers	-	0.04, 0.06	
Crisphead lettuce	0.01, 0.09	0.02	
Tomatoes	0.01, 0.02	0.02, 0.02, 0.03, 0.04, 0.10, 0.17	
Green sweet peppers	0.06, 0.72	0.02, 0.03, 0.26	
Canned snap beans	0.01, 0.02	0.01	

Farm gate to consumer studies

Farm gate to consumer studies were carried out on five crops in the USA in 1985 and 1986 (Lai, 1989b). Lettuce, snap beans, cauliflowers, Brussels sprouts and bell peppers were treated with acephate at the highest label rate and monitored for residues from harvest through typical commercial processes to the consumer. The results are shown in Table 14 (identical to Table 13 in the monograph on acephate but repeated for convenience).

Table 14. Residues of acephate and methamidophos in crisphead lettuce, snap beans, bell peppers, cauliflowers and Brussels sprouts from farm gate to consumer, USA (Lai, 1989b).

Application, Year	Description (Location)	Acephate		Methamidophos	
		mg/kg	% of field	mg/kg	%of field
0.63 + 1.12 kg ai/ha	Whole head lettuce (field)	0.30	100	0.02	100
	Head + cap leaf (cooler)	0.05	17	0.00	0
2 applications	Head + cap leaf (distributor)	0.06	20	0.00	0
PHI 21 days	Head + cap (market)	0.04	13	0.00	0
1985	Head + cap (supermarket shelf)	0.03	10	0.00	0
0.84 kg ai/ha	Fresh snap beans (field)	0.29	100	0.06	100
2 applications	Fresh snap beans (market)	0.10	35	0.02	36
PHI 24 days	Fresh snap beans (processing plant)	0.13	46	0.03	55
1985	Canned snap beans	0.05	18	0.02	36
	Frozen snap beans in butter sauce	0.03	11	0.00	0
1.5 kg ai/ha	Bell peppers (field)	3.8	100	0.52	100
7 applications	Bell peppers (packing shed)	2.8	75	0.43	83
PHI 9 days	Bell peppers (distributor)	2.7	71	0.45	87

Application, Year	Description (Location)	Acephate		Methamidophos	
		mg/kg	% of field	mg/kg	%of field
1986	Bell peppers (supermarket)	3.1	83	0.51	97
1.12 kg ai/ha	Cauliflower head (field)	0.80	100	0.10	100
6 applications	Trimmed head (cooler)	0.34	42	0.04	40
PHI 14 days	Curd after coring (processor)	0.33	41	0.04	40
1986	Curd after processing and freezing	0.25	31	0.04	40
	Processing waste	0.73	91	0.10	95
1.12 kg ai/ha	Fresh Brussels sprouts (field)	1.85	100	0.03	100
6 applications	Fresh sprouts after sorting	0.79	43	0.02	67
PHI 14 days	Sorting waste	1.6	86	0.02	67
1986	Sprouts after blanching & freezing	0.13	7	0.01	33
	Processing waste	9.4	508	0.15	500

NATIONAL MAXIMUM RESIDUE LIMITS

The following national MRLs were reported to the Meeting.

Country	Commodity	MRL, mg/kg	Commodity	MRL, mg/kg
Argentina	Alfalfa forage	0.1	Melon	0.5
	Alfalfa seed	0.1	Olive oil	0.1
	Almond	0.1	Peppers, Sweet	0.5
	Bean	0.1	Pome fruit	0.1
	Cereals	0.05	Potato	0.1
	Citrus fruit	0.5	Rice	0.05
	Clover forage	2	Soya	0.1

	Cotton seed	0.1	Stone fruit	0.1
	Garlic	0.1	Sunflower	0.1
	Grape	0.1	Tomato	0.5
	Leafy + other stem vegetables	0.2		
Australia	Egg plant	1	Goat milk	0.01*
	Sugar beet	0.05	Hops dry	5
	Brussels sprouts	1	Lettuce	1
	Cabbages	1	Peaches	1
	Cattle fat	0.01*	Peanut	0.02*
	Cattle meat	0.01*	Peanut fodder	10
	Cattle meat by-products	0.01*	Peanut forage	10
	Cattle milk	0.01*	Peppers, Sweet	2
	Cauliflowers	1	Potato	0.25
	Celery	2	Rape seed	0.1
	Citrus fruit	0.5	Sheep meat	0.01*
	Cotton seed	0.1	Sheep meat by-products	0.01*
	Cucumber	0.5	Sheep milk	0.01*
	Goat fat	0.01*	Soya	0.1
	Goat meat	0.01*	Tomatoes	2
	Goat meat by-products	0.01*	Tree tomatoes	0.01*
Austria	All food of animal origin	0.01	Other plant commodities	0.05
	Hops	5		
Belgium	Cabbages	0.1	Other plant commodities	N.D.[1]
	Hops	5		
Brazil	Bean	0.01	Peanut	0.1
	Broccoli	1	Peppers, Sweet	0.4
	Cabbages	1	Potato	0.1
	Cabbages, white	1	Soya	0.01
	Cauliflowers	1	Tomatoes	0.3
	Cotton seed	0.1		
Canada	Bean	0.3	Lettuce	1
	Egg plant	0.5	Melons	0.1*
	Broccoli	1	Peppers, Sweet	1
	Brussels sprouts	1	Potato	0.1*
	Cabbages	0.5	Rape seed	0.1*
	Cauliflowers	0.5	Tomatoes	0.5
	Cucumber	0.5		
Chile	Sugar beet	0.1	Peaches	1
	Cattle fat	0.01*	Potato	0.1
	Cattle meat	0.01*	Rape	0.1
	Goat fat	0.01*	Sheep fat	0.01*
	Goat meat	0.01*	Sheep meat	0.01*
	Lettuce, head	2	Tomatoes	2
	Milk	0.01*		
Cyprus	Egg plant	1	Lettuce	1
	Beets (Beta vulgaris) leaf	1	Meat	0.01
	Beets (Beta vulgaris) root	0.1	Milk	0.01
	Cabbages	1	Peaches	1
	Cauliflowers	2	Pepper, cayenne	1
	Celery	2	Peppers, Sweet	1

	Citrus fruit	0.5	Potato	0.1
	Cucumber	1	Tomatoes	2
Denmark	Artichoke	0.01*T	Meat, preparations of	0.01*
	Egg plant	0.2	Milk	0.01*
	Bean dry	0.01*T	Milk products	0.01*
	Berries and small fruits	0.01*	Mushroom	0.01*
	Berry, wild	0.01*	Nuts	0.01*
	Bulb vegetables	0.01*		
	Cabbages, Head	0.5	Other leafy vegetables	0.01*
	Cereals	0.01*	Other oil seed	0.01*
	Chicory, Witloof	0.01*	Other pulses	0.01*
	Citrus fruit	0.2	Other solanaceae	0.01*
	Corn, Sweet	0.01*	Other stem vegetables	0.01*
	Cotton	0.1	Other tropical/subtropical fruits	0.01*
	Cucurbits with edible peel	1	Pea dry	0.01*T
	Cucurbits with inedible peel	0.01*	Peppers, Sweet	0.01*T
	Eggs without shell	0.01*	Pome fruit	0.01*T
	Escarole	0.2	Potato	0.01*
	Flowering brassicas	0.01*T	Poultry fat	0.01*
	Grape	0.01*T	Poultry meat	0.01*
	Herbs	0.01*	Poultry meat by-products	0.01*
	Hops	2	Root and tuber vegetables	0.01*
	Kohlrabi	0.01*	Rubus species (Cane fruit)	0.01*
	Leafy brassicas	0.01*T	Spinach & similar	0.01*
	Leek	0.01*T	Stone fruit	0.01*T
	Legume vegetables	0.01*T	Strawberry	0.01*T
	Lentil dry	0.01*	Tea	0.1*
	Mammalian, fat	0.01*	Tomatoes	0.5
	Mammalian, meat	0.01*	Watercress	0.01*
	Mammalian, meat by-products	0.01*		
European Union	Artichoke	0.01*T	Meat, preparations of	0.01*
	Egg plant	0.2	Milk	0.01*
	Bean dry	0.01*T	Milk products	0.01*
	Berries and small fruits	0.01*	Mushroom	0.01*
	Berry, wild	0.01*	Nuts	0.01*
	Bulb vegetables	0.01*	Other cucurbits	0.01*
	Cabbages, Head	0.5	Other leafy vegetables	0.01*
	Cereals	0.01*	Other oil seed	0.01*
	Chicory, Witloof	0.01*	Other pulses	0.01*
	Citrus fruit	0.2	Other solanaceae	0.01*
	Corn, Sweet	0.01*	Other stem vegetables	0.01*
	Cotton	0.1	Other tropical/subtropical fruits	0.01*
	Cucumber	1	Pea, dry	0.01*T
	Cucurbits with inedible peel	0.01*	Peppers, Sweet	0.01*T
	Eggs with shell	0.01*	Pome fruit	0.01*T
	Flowering brassicas	0.01*T	Potato	0.01*
	Grape	0.01*T	Poultry fat	0.01*
	Herbs	0.01*	Poultry meat	0.01*
	Hops	2	Poultry meat by-products	0.01*
	Kohlrabi	0.01*	Root and tuber vegetables	0.01*

	Leafy brassicas	0.01*T	Rubus species (Cane fruit)	0.01*
	Leek	0.01*T	Spinach & similar	0.01*
	Legume vegetables	0.01*T	Stone fruit	0.01*T
	Lentil dry	0.01*	Strawberry	0.01*T
	Lettuce	0.2	Tea	0.1*
	Mammalian, fat	0.01*	Tomatoes	0.5
	Mammalian, meat	0.01*	Watercress	0.01*
	Mammalian, meat by-products	0.01*		
Finland	Fruit	0.2	Vegetables	0.2
France	Egg plant	0.2	Other fruits	0.01
	Cabbages, Head	0.5	Other oil seed	0.01
	Cereals	0.01	Other vegetables	0.01
	Citrus fruit	0.2	Pome fruit	0.3
	Cotton	0.1	Potato	0.01
	Cucumber	1	Stone fruit	0.3
	Grape	0.3	Tea	0.1
	Hops	2	Tomatoes	0.5
	Lettuce	0.2		
Germany	Animal fat	0.01	Lettuce, head	0.2
	Egg plant	0.2	Meat	0.01
	Cabbages, Head	0.5	Meat, preparations of	0.01
	Cauliflowers	0.2	Milk	0.01
	Citrus fruit	0.2	Milk products	0.01[2]
	Cotton seed	0.1	Other plant commodities	0.01
	Cucumber	1	Peaches	1
	Egg products	0.01[2]	Peppers, Sweet	1
	Eggs	0.01	Pome fruit	0.2
	Hops	2	Tea	0.1
	Leafy brassicas	0.2	Tomatoes	0.5
India	Cotton seed	0.1	Safflower seed	0.1
Israel	Egg plant	1	Garlic	0.1
	Beet, Sugar	0.05	Melons	1
	Brassica vegetables	1	Onion, Bulb	0.1
	Celery	2	Peppers, Sweet	1
	Cotton seed	0.1	Potato	0.1
	Cucumber	0.5	Tomatoes	2
Italy	Artichoke	0.01*T	Meat, preparations of	0.01*
	Egg plant	0.2	Milk	0.01*
	Bean dry	0.01*T	Milk products	0.01*
	Beet, Sugar	0.15	Mushroom	0.01*
	Berries and small fruits	0.01*	Nuts	0.01*
	Berry, wild	0.01*	Other cucurbits with edible peel	0.01*
	Bulb vegetables	0.01*	Other leafy vegetables	0.01*
	Cabbages, Head	0.5	Other oil seed	0.01*
	Cereals	0.01*	Other solanaceae	0.01*
	Chicory, Witloof	0.01*	Other stem vegetables	0.01*
	Citrus fruit	0.2	Pea	0.01*T
	Corn, Sweet	0.01*	Peppers, Sweet	0.01*T
	Cotton seed	0.1	Pome fruit	0.15[3]
	Cucumber	1	Potato	0.01*

	Cucurbits with inedible peel	0.01*	Poultry fat	0.01*
	Eggs	0.01*	Poultry meat	0.01*
	Flowering brassicas	0.01*T	Poultry meat by-products	0.01*
	Herbs	0.01*	Pulses	0.01*
	Hops	2	Root and tuber vegetables	0.01*
	Kohlrabi	0.01*	Rubus-Species (Cane fruit)	0.01*
	Leafy brassicas	0.01*T	Spinach & similar	0.01*
	Leek	0.01*T	Stone fruit	0.15[3]
	Legume vegetables	0.01*T	Strawberry	0.15[3]
	Lentil dry	0.01*	Tea	0.1*
	Lettuce	0.2	Tomatoes	0.5*
	Mammalian fat	0.01*	Tropical and subtropical fruits	0.01*
	Mammalian meat	0.01*	Watercress	0.01*
	Mammalian meat by-products	0.01*		
Japan	Beet, Sugar	0.05	Lettuce	1
	Broccoli	1	Other fruits	0.1
	Cabbages	1	Peaches	1
	Cauliflowers	1	Potato	0.25
	Cotton seed	0.1	Rape	0.1
	Hops	5	Soya	0.05
Kenya	Cattle fat	0.01*T	Milk	0.01*T
	Cattle meat	0.01*T	Sheep fat	0.01*T
	Goat fat	0.01*T	Sheep meat	0.01*T
	Goat meat	0.01*T		
Luxembourg	Cabbages	0.1	Potato	0.01
Malaysia	Egg plant	1	Peaches	0.25
	Citrus fruit	0.5	Pepper, cayenne-	1
	Cucumber	1	Peppers, Sweet	1
	Fat and oil edible	0.1	Potato	0.1
	Melons, water-	0.5	Tomatoes	1
Mexico	Alfalfa	2	Cotton	0.1
	Egg plant	1	Cucumber	1
	Broccoli	1	Lettuce, head	1
	Brussels sprouts	1	Melons	0.5
	Cabbages	1	Potato	0.1
	Capsicum (Peppers, Chilli)	1	Soya	0.05
	Cauliflowers	1	Tomatoes	1
	Celery	1		
Netherlands	Aubergine	0.2	Lettuce	0.2
	Cabbages, Head	0.5	Melons	1
	Citrus fruit	0.2	Other plant commodities	N.D.[1]
	Cotton seed	0.1	Tea	0.1*
	Flowering brassicas	0.1	Tomatoes	0.5
	Hops	2		
New Zealand	Brassica vegetables	1	Leafy vegetables	0.5
	Citrus fruit	0.5	Potato	0.1
	Fruiting vegetables except tomatoes	0.2	Tomatoes	0.1
Spain	Artichoke	0.2	Other cucurbits with inedible peel	0.01
	Egg plant	0.2	Other leafy vegetables	0.01

	Bean dry	0.01	Other legume vegetables	0.01
	Bean pods and/or immature seeds	0.1	Other oil seed	0.01
	Beet, Sugar	0.05	Other pulses	0.01
	Berry, wild	0.01	Other solanaceae	0.01
	Bulb vegetables	0.01	Other stem vegetables	0.01
	Cabbages	0.5	Pea, dry	0.01
	Cereals	0.01	Pea, pods and/or immature seeds	0.2
	Chicory, Witloof	0.01	Peppers, Sweet	1
	Citrus fruit	0.2	Pome fruit	0.2
	Corn, Sweet	0.01	Potato	0.01
	Cotton seed	0.1	Root and tuber vegetables	0.01
	Cucumber	1	Rubus species (Cane fruit)	0.01
	Flowering brassicas	0.01	Spices	0.01
	Food, dry	0.01	Spinach & similar	0.01
	Forage crops a. straw	0.01	Stimulant plants	0.01
	Grape	0.01	Stone fruit	0.2
	Herbs	0.01	Strawberry	0.01
	Hops	2	Sugar cane	0.01
	Kohlrabi	0.01	Tea	0.1
	Leafy brassicas	0.01	Tea plant infusion	0.1
	Leek	0.05	Tobacco	0.01
	Lettuce	0.2	Tomatoes	0.5
	Mushroom	0.01	Tropical and subtropical fruits	0.01
	Nuts	0.01	Watercress	0.01
	Other berries and small fruits	0.01		
Sri Lanka	Bean	1	Cowpea	1
	Beets (Beta Vulgaris)	1	Potato	0.1
	Cabbages	1		
Sweden	Fruit	0.2	Vegetables	0.2
	Potato	0.02*		
Taiwan	Asparagus	0.1	Longan	0.2
	Bamboo	0.1	Mango	0.2
	Bean, Mung	0.03	Mustard	0.1
	Bean, Adzuki	0.03	Onion	0.1
	Cabbages	0.5	Pe-tsai	0.1
	Cabbages, Chinese	0.5	Peanut	0.03
	Carrot	0.1	Potato	0.1
	Cauliflowers	0.5	Radish	0.1
	Celery	0.5	Rape	0.5
	Dasheen	0.1	Rice	0.5
	Garlic	0.5	Shallot	0.5
	Ginger	0.1	Soya	0.03
	Leek	0.5	Spinach	0.5
	Litchi	0.2	Water spinach	0.5
UK	Almond	0.01*	Mushrooms, wild, edible	0.01*
	Asparagus	0.01*	Mustard seed	0.01*
	Egg plant	0.2	Nut, para-	0.01*
	Avocado	0.01*	Walnut	0.01*
	Banana	0.01*	Oats	0.01*

	Barley	0.01*	Olive	0.01*
	Beetroot	0.01*	Onion	0.01*
	Berry, wild	0.01*	Orange	0.2
	Blackberry	0.01*	Other berries and small fruits	0.01*
	Blueberry	0.01*	Other brassica vegetables	0.5
	Boysenberry	0.01*	Other bulb vegetables	0.01*
	Brussels sprouts	0.5	Other cereals except rice	0.01*
	Cabbages, Head	0.5	Other citrus fruits	0.2
	Cardoon	0.01*	Other cucurbits with edible peel	0.01*
	Carrot	0.01*	Other cucurbits with inedible peel	0.01*
	Cashew nut	0.01*	Other herbs	0.01*
	Celeriac	0.01*	Other leafy vegetables	0.01*
	Celery	0.01*	Other nuts	0.01*
	Celery, bleached	0.01*	Other oil seed	0.01*
	Chard, Swiss	0.01*	Other pulses	0.01*
	Chervil, Garden	0.01*	Other root & tuber vegetables	0.01*
	Chestnut	0.01*	Other solanaceae	0.01*
	Chicory, Witloof	0.01*	Other stem vegetables	0.01*
	Chives	0.01*	Other tropical/subtropical fruits	0.01*
	Coconut	0.01*	Parsley, leaf	0.01*
	Corn, Sweet	0.01*	Parsley, turnip-rooted	0.01*
	Cotton seed	0.1	Parsnip	0.01*
	Cranberry	0.01*	Passion fruit	0.01*
	Cress, Garden	0.01*	Peanut	0.01*
	Cucumber	1	Pecan nut	0.01*
	Cumquat (Kumquat)	0.01*	Pine	0.01*
	Currant, Red, White, Black	0.01*	Pineapple	0.01*
	Date	0.01*	Pistachio	0.01*
	Eggs	0.01*	Pomegranate	0.01*
	Escarole	0.01*	Poppy seed	0.01*
	Fennel, Italian	0.01*	Potato, early	0.01*
	Fig	0.01*	Potato, ware	0.01*
	Flax/Linseed seed	0.01*	Pomelo	0.2
	Garlic	0.01*	Pumpkin	0.01*
	Gherkin	0.01*	Radish	0.01*
	Gooseberry	0.01*	Rape seed	0.01*
	Grapefruit	0.2	Raspberry	0.01*
	Hazel nut	0.01*	Rhubarb	0.01*
	Hops	2	Rice	0.01*
	Horseradish	0.01*	Rutabaga	0.01*
	Kiwifruit	0.01*	Rye	0.01*
	Kohlrabi	0.01*	Salsify, black	0.01*
	Lamb's lettuce	0.01*	Sesame seed	0.01*
	Lemon	0.2	Shallot	0.01*
	Lentil	0.01*	Soya	0.01*
	Lettuce	0.2	Spinach & similar	0.01*
	Lime, sour	0.2	Spring onion	0.01*
	Lychee	0.01*	Sunflower seed	0.01*
	Macadamia nut	0.01*	Sweet potato	0.01*
	Maize (Corn)	0.01*	Tea	0.01*
	Mandarin	0.2	Tomatoes	0.5

	Mango	0.01*	Topinambur	0.01*
	Meat	0.01*	Triticale	0.01*
	Meat, preparations of	0.01*	Turnip, edible	0.01*
	Melons	0.01*	Watercress	0.01*
	Melons, water-	0.01*	Wheat	0.01*
	Milk	0.01*	Yam	0.01*
	Mushrooms, cultivated	0.01*	Zucchini	0.01*
Uruguay	Tomatoes	0.1		
USA	Aubergine	1	Cotton seed	0.1*
	Beet, Sugar, root	0.02	Cucumber	1
	Beet, Sugar, tops or leaves	0.5	Lettuce	1
	Broccoli	1	Melons	0.5
	Brussels sprouts	1	Pepper, Cayenne	1
	Cauliflowers	1	Peppers, Sweet	1
	Cabbages	1	Potato	0.1*
	Celery	1	Tomatoes	1

* At or about the limit of determination; negligible residue tolerance
T Temporary MRL
[1] <0.01 mg/kg
[2] MRL refers to the whole product
[3] If not adopted by 01.01.1998, will become 0.01 mg/kg

APPRAISAL

Methamidophos was first evaluated in 1976, with further reviews of residue aspects in 1979, 1981, 1984, 1990 and 1994. The 1994 JMPR withdrew the previous recommendations for MRLs for broccoli, Brussels sprouts, head cabbages, cauliflower, citrus fruits, eggplant, melons except watermelon, peaches and tomato which had been held at Step 7B by the 1992 CCPR (ALINORM 93/24, para 119-123). The manufacturer indicated that information on GAP and residue data would be available to support new MRLs for these commodities. This information was provided to the Meeting, together with information on analytical methods and residues in food in commerce or at consumption.

Analytical methods

Samples from the supervised trials were analysed by GLC. Recoveries of both acephate and methamidophos were generally >70%, with limits of determination of 0.01-0.02 mg/kg.

These methods were considered suitable for use in supervised trials and for enforcement.

Stability of residues in stored analytical samples

Studies of the storage stability of acephate and methamidophos were carried out with vegetables, pulses, oilseed, animal products, cereals and grasses using samples which had been treated with acephate. The stability of methamidophos in the macerated or ground samples was not established by the results as most of the samples contained substantially higher residues of acephate than of methamidophos, and there may have been some conversion of acephate to methamidophos during

storage.

Validity of data

In view of the difficulty of determining methamidophos caused by its high polarity, the Meeting did not evaluate trials which lacked data on analytical recoveries or in which recoveries were below 70%, trials without analysis of control samples and/or sample chromatograms, or trials with abnormally high control values and for which sample chromatograms were not supplied.

Supervised trials

Where data were available from applications of both methamidophos and acephate the results of trials with methamidophos are discussed first and the acephate trials are indicated by a sub-heading. Trials on citrus fruits and Brussels sprouts were with acephate only and are considered last.

Peaches. Four Italian trials with 0.6-0.68 kg ai/ha and a PHI of 21 days were according to GAP in France, Greece and/or Italy (0.38-0.68 kg ai/ha, 21 days PHI) and residues were 0.09-0.27 mg/kg. Five of the six Spanish trials (0.72-1.3 kg ai/ha, 28-35 days PHI) were according to Spanish GAP (0.75-1.1 kg ai/ha, 35 days PHI) with residues of <0.01-0.76 mg/kg. One of these, at 1.0 kg ai/ha, included a PHI of 21 days and was therefore comparable with Portuguese GAP (0.8-1.0 kg ai/ha, 21 days PHI). The residue was 0.27 mg/kg. The sixth Spanish trial was not according to Spanish GAP, but included conditions (0.6 kg ai/ha, 21 days PHI) which complied with GAP in France, Greece and Italy. The residue after 21 days was 0.09 mg/kg.

The residues in the 4 Italian trials carried out at maximum GAP were 0.09, 0.12, 0.16 and 0.27 mg/kg, and those in the five Spanish trials detailed above were 0.07, 0.09, 0.24, 0.27 and 0.76 mg/kg. The residues from the 9 trials in rank order (median underlined) were 0.07, 0.09, 0.09, 0.12, 0.16, 0.24, 0.27, 0.27 and 0.76 mg/kg.

The Meeting estimated a maximum residue level of 1 mg/kg and an STMR of 0.16 mg/kg for peaches, based on residues of methamidophos from the application of methamidophos.

Broccoli. Two Brazilian trials and one Canadian trial complied with their national GAP (Brazil 0.3-0.6 kg ai/ha, 0.06 kg ai/hl, 21 days PHI, Canada 0.53-1.1 kg ai/ha, 14 days PHI). The residues were <0.01 and 0.2 mg/kg in Brazil and 0.01 mg/kg in Canada.

Two Canadian (1972), one Mexican and nine American trials were also according to GAP (Mexico 0.6-0.9 kg ai/ha, 21 days PHI; USA 0.56-1.1 kg ai/ha, 14 or 21 days PHI). Residues were 0.13-0.41 mg/kg in Canada, <0.01-0.08 mg /kg in Mexico and <0.01-0.14 mg/kg in America. However information on analytical recoveries was lacking so the Meeting could not evaluate the trials.

Residues from the application of acephate. One Australian and two Japanese trials with acephate complied with GAP (Australia 0.98 kg ai/ha, 0.098 kg ai/hl, 14 days PHI; Japan 0.05 kg ai/hl, 14 days PHI). In the Australian trial the spray concentration of 0.21 kg ai/hl was higher than the GAP concentration but the dosage rate complied with GAP. The residues of methamidophos were <0.02-0.08 mg/kg and 0.017-0.566 mg/kg in Australia and Japan respectively. One Brazilian trial with acephate did not include data on methamidophos. Two French and two Spanish trials on acephate were reported but no information on GAP was available. An Italian trial did not comply with Italian

GAP.

The Meeting could not estimate a maximum residue level.

Cabbages. One Brazilian trial complied with GAP (0.3-0.6 kg ai/ha, 0.06 kg ai/hl, 21 days PHI). The residue was 0.08 mg/kg.

Two German supervised trials with 0.36 kg ai/ha, 0-28 days PHI, and 3 applications were comparable with German GAP (0.36 kg ai/ha, 14 days PHI, 2 applications) except in the number of applications. The Meeting considered that the additional application would not have a significant effect on the results. The residues were 0.05 and 0.2 mg/kg.

The results of seven US trials submitted to the 1990 JMPR were rejected by the 1994 JMPR. The present Meeting found the trial conditions (1.1 kg ai/ha, 6 application, 21-35 days PHI) were comparable with current GAP (0.56-1.1 kg ai/ha, 35 days PHI) and could evaluate the data. The residues were <0.01 (5) and <0.01-0.03 mg/kg in six trials and 0.76-1.1 mg/kg in the seventh. The Meeting agreed not to consider the residues from the seventh trial since they seemed to be of a different population from others and because of difficulties encountered with the evaluation of data without the original studies.

Four trials in Germany were not evaluated in 1990 but the trial conditions (0.3 kg ai/ha, 2 application, 0-28 days PHI) were comparable with GAP (see above). The residues were <0.01, <0.01, 0.01 and 0.09 mg/kg at 14 days PHI.

One Argentinian and one Mexican trial were comparable with Brazilian or Mexican GAP but information on analytical recoveries was lacking so the Meeting could not evaluate the trials.

Residues from the application of acephate. Three of four French and two Japanese trials with acephate complied with their national GAP (France 0.075 kg ai/hl, 7 days PHI; Japan 0.025-0.05 kg ai/hl, 7 days PHI). The residues of methamidophos were 0.01 (2) and 0.09 mg/kg in France and 0.010 and 0.138 mg/kg in Japan.

One supervised trial on acephate in The Netherlands (0.75 kg ai/ha, 14 days PHI, 1 application) was within the limits of GAP (0.75 kg ai/ha, 14 days PHI, 6 applications) and the residues of methamidophos were 0.038-0.050 mg/kg.

The residues of methamidophos from its application according to maximum GAP were 0.08 mg/kg in Brazil, <0.01 (5) and 0.03 mg/kg in the USA and <0.01 (2), 0.01, 0.05, 0.09 and 0.2 mg/kg in Germany.

The residues of methamidophos from the application of acephate according to maximum GAP were 0.01 (2) and 0.09 mg/kg in France, 0.010 and 0.117 mg/kg in Japan and 0.05 mg/kg in The Netherlands.

The residues from 19 trials in rank order were <0.01 (7), 0.01 (4), 0.03, 0.05 (2), 0.08, 0.09 (2), 0.12 and 0.2 mg/kg.

The Meeting estimated a maximum residue level of 0.5 mg/kg and an STMR of 0.01 mg/kg, based on the residues of methamidophos from the use of acephate or methamidophos on cabbages.

Cauliflowers. Two Brazilian trials complied with GAP (0.3-0.6 kg ai/ha, 0.06 kg ai/hl, 21 days PHI). The residues were <0.01-0.5 mg/kg. Reports of three French trials were submitted without information on GAP.

Six supervised trials in Germany were reported to the 1981 JMPR. Reports of three were resubmitted to the present Meeting. The trial conditions (0.36 kg ai/ha, 2-3 applications, 0, 14, 21 and 28 days PHI) complied with GAP (0.36 kg ai/ha, 2 application, 21 days PHI). The residues were <0.01, <0.01 and 0.01 mg/kg (reported in the 1981 JMPR monograph) and <0.01, 0.01 and 0.04 mg/kg.

One supervised trial was carried out in Mexico, but there was no comparable GAP and recovery data were lacking. One American trial was reported to the Meeting and the conditions accorded with American GAP (0.56-1.1 kg ai/ha, 28 days PHI). The residue was 0.07 mg/kg, but again critical information, including recovery data, was lacking.

Residues from the application of acephate. One Australian and two Japanese supervised trials on acephate were according to national GAP (Australia 0.98 kg ai/ha, 0.098 kg ai/hl, 3 days PHI; Japan 0.05 kg ai/hl, 14 days PHI). In the Australian trial the spray concentration (0.24 kg ai/hl) was higher than the GAP concentration but the kg ai/ha rate complied with GAP. The residues of methamidophos were 0.05-0.20 mg/kg at PHIs of 3-7 days in Australia and <0.005-0.228 mg/kg at 14 days in Japan.

Five supervised trials in The Netherlands were according to GAP (0.75 kg ai/ha, 14 days PHI, 6 applications) except that there were only 1-4 applications, but it seems that the number of applications does not influence the residue significantly. The Meeting considered that the results were valid for the estimation of a maximum residue level and an STMR. The residues of methamidophos were <0.01-0.03 me/kg.

Four supervised trials in France and one in Germany were well conducted but the conditions were not comparable with any available GAP. One Brazilian trial with acephate did not include analyses for methamidophos.

The residues of the 2 Brazilian and 6 German trials with methamidophos at maximum GAP were <0.01 and 0.5 mg/kg, and <0.01(3), 0.01(2) and 0.04 mg/kg respectively. The residues of methamidophos from applications of acephate at maximum GAP were 0.2 mg/kg in Australia, 0.006 and 0.23 mg/kg in Japan and <0.01 (3), 0.03 and 0.018 in The Netherlands.

The residues from the 16 trials in rank order were 0.006, <0.01(7), 0.01(2), 0.018, 0.03, 0.04, 0.2, 0.23 and 0.5 mg/kg.

The Meeting estimated a maximum residue level of 0.5 mg/kg and an STMR of 0.01 mg/kg for residues of methamidophos in cauliflower arising from the use of acephate or methamidophos.

Melons. Two Spanish trials were reported without information on relevant GAP.

Two supervised trials in Mexico were reviewed in 1990. The trial conditions (0.6 kg ai/ha, 0, 3, 7/8, and 14 days PHI) complied with GAP (0.6-0.9 kg ai/ha, 7 days PHI) and the residues were 0.05 and 0.08 mg/kg in the whole fruit, 0.06 mg/kg in the pulp and 0.13 mg/kg in the peel. In another

Mexican trial with the same conditions the residues were 0.23-1.63 mg/kg, but critical information including data on analytical recoveries was lacking.

A single supervised trial was carried out in Argentina, but there was no relevant GAP and critical information was again lacking.

The Meeting could not estimate a maximum residue level.

Egg plants. One Spanish, one Argentinian and three American trials were reported to the Meeting without information on relevant GAP.

Conditions in two Mexican trials were comparable to Mexican GAP (0.6-0.9 kg ai/ha, 14 days PHI) with residues of 0.28 and 0.95 mg/kg, but critical information was lacking.

The Meeting could not estimate a maximum residue level.

Tomatoes. An Australian trial was carried out at higher spray concentration (0.44 kg ai/hl) than GAP (0.029-0.11 kg ai/hl), but at a dose rate within the GAP range (0.32-1.2 kg ai/ha, 4 days PHI). The residues were 0.64-0.96 mg/kg at 3-5 days PHI.

Two Brazilian trials reflected GAP (0.3-0.6 kg ai/ha, 0.06 kg ai/hl, 21 days PHI) and the residues were <0.01 and 0.3 mg/kg. In one of them the residue at 14 days PHI was determined and this could be related to GAP in Chile and Ecuador where the PHI is 15 days. The residue was <0.01 mg/kg. Four French supervised trials were reported without information on GAP, but the conditions were comparable with GAP in Portugal (0.6-1.2 kg ai/ha, 21 days PHI). The residues were <0.02, 0.03-0.04, 0.05-0.06 and 0.07-0.08 mg/kg.

Twelve Spanish trials at 0.045-0.06 kg ai/hl, 7-8 days PHI, complied with Spanish GAP (0.05-0.075 kg ai/hl, 7 days PHI) but six of them showed abnormally high control values and were without sample chromatograms, so were not used for the estimation of a maximum residue level. The residues from the other 6 trials were <0.02 (2), 0.12, 0.14, 0.29 and 0.32 mg/kg.

Trials in Italy and Mexico were comparable with Greek and Mexican Gap respectively but critical information was lacking. One supervised trial in Turkey was reported to the Meeting without information on relevant GAP.

Residues from the application of acephate. One Australian, one Spanish and two Japanese supervised trials with acephate reflected national GAP (Australia 0.98 kg ai/ha, 0.098 kg ai/hl, 3 days PHI; Japan 0.025-0.05 kg ai/hl, 1 days PHI; Spain 0.038-0.11 kg ai/hl, 14 days PHI). In the Australian trial the spray concentration (0.35 kg ai/hl) was high but the dose rate (kg ai/ha) accorded with GAP. The residues of methamidophos were 0.40 and 0.50 mg/kg in Australia and 0.03 mg/kg in Spain. In Japan the residues at 7 days (higher than at 1 and 3 days) were 0.072 and 0.106 mg/kg in one trial and 0.085 and 0.123 mg/kg in the other.

Seventeen French supervised trials with acephate were reported to the Meeting with no information on GAP, but four of them (0.62-0.83 kg ai/ha, 13-15 days PHI, 1-3 applications) could be related to Polish GAP (0.75 kg ai/ha, 14 days PHI, 1 application), since it seems that the number of application does not significantly affect the residue level. Furthermore these four and six other trials (0.03-0.075 kg ai/hl, 13-15 days PHI) were according to Spanish GAP (0.038-0.11 kg ai/hl, 14

days PHI). The residues of methamidophos at 13-21 days ranged from <0.02 mg/kg to 0.44 mg/kg.

The residues of methamidophos from its use at maximum GAP were 0.96 mg/kg in Australia, <0.01 and 0.3 mg/kg in Brazil, <0.02 and 0.08 mg/kg in France (according to Portuguese GAP) and 0.12, 0.14, 0.29 and 0.32 mg/kg in Spain.

The residues of methamidophos from the use of acephate at maximum GAP were 0.5 mg/kg in Australia, 0.089 and 0.104 mg/kg in Japan, 0.03 mg/kg in Spain, and 0.04, 0.06, 0.16 and 0.16 mg/kg in France.

The residues from the 17 trials in rank order were <0.01, <0.02, 0.03, 0.04, 0.06, 0.08, 0.089, 0.104, 0.12, 0.14, 0.16, 0.16, 0.29, 0.3, 0.32, 0.5 and 0.96 mg/kg.

The Meeting estimated a maximum residue level of 1 mg/kg and an STMR level of 0.12 mg/kg, based on the residues of methamidophos from the uses of acephate or methamidophos on tomatoes.

Residues of methamidophos in citrus fruits and Brussels sprouts resulting from the application of acephate

Citrus Fruits. Six supervised trials, two on Satsuma mandarins, two on Natsudaidai, one on Kabosu (sour orange) and one on Yuzu (lemons and limes), were carried out with acephate applied according to GAP in Japan. (0.025-0.05 kg ai/hl, 30 days PHI) The residues of methamidophos were 0.07 and 0.11 mg/kg in Satsuma mandarins, 0.06 and 0.33 mg/kg in Natsudaidai, 0.031 mg/kg in Kabosu and 0.044 mg/kg in Yuzu.

A single New Zealand residue trial with acephate on mandarins complied with GAP (0.075 kg ai/hl, 14 days PHI) and the residue of methamidophos was 0.29 mg/kg.

A trial in Greece with acephate on oranges reported without information on GAP, but the conditions (0.031 kg ai/ha, 20 days PHI) were comparable with Italian GAP (0.024-0.036 kg ai/hl, 21 days PHI). The residue of methamidophos was 0.05 mg/kg.

Since the Meeting was unable to estimate a maximum residue level for acephate in citrus fruits it could not estimate the maximum residue level of methamidophos arising from the use of acephate.

Brussels sprouts. Two Australian supervised trials with acephate were considered to be comparable with Australian GAP (0.98 kg ai/ha, 0.098 kg ai/hl, 3 days PHI) since the dose rate was 0.98 kg ai/ha although the spray concentration was high (0.21 kg ai/hl). The residues of methamidophos were 0.05-1.0 mg/kg. One of two US trials with acephate complied with US GAP (0.56-1.1 kg ai/ha, 14 days PHI). The residues of methamidophos on the trimmed heads were 0.01 and 0.02 mg/kg.

The results did not require any change of the existing CXL for Brussels sprouts (1 mg/kg).

Processing studies

Peaches. The concentration factors from 2 trials were 0.73 and 0.56 for washed fruit, 0.91 and 0.44 for juice, 0.91 and 0.33 for jam, and 0.82 and 0.22 for preserve. The mean concentration factors were

respectively 0.65, 0.68, 0.62 and 0.52. The Meeting estimated STMR-P levels of 0.10, 0.11, 0.10 and 0.08 mg/kg for washed fruit, juice, jam and preserve by calculation from the STMR for peaches (0.16 mg/kg) and the mean concentration factors.

In cooking studies on tomatoes, cabbages and broccoli, 30 minutes boiling had no measurable effect on the levels of methamidophos residues.

Monitoring data

In monitoring in The Netherlands a total of 8681 samples of peaches, nectarines, grapes, strawberries, tomatoes, sweet peppers, cucumbers, lettuce, celery and beans were analysed for methamidophos during 1991-1994. Detection frequencies ranged from 0.07% for strawberries to 4.2% for nectarines and the highest mean residue was 0.02 mg/kg, found in peaches and lettuce.

Market basket surveys

Market basket surveys for methamidophos were carried out at 24 locations in the USA in 1984 and 1985. Methamidophos was found in samples of 7 of 62 of collected commodities. The highest residue was 0.26 mg/kg in a sample of green sweet peppers.

RECOMMENDATIONS

Since methamidophos has been listed by ;the CCPR as a candidate for periodic review but not yet scheduled, and in view of the difficulties encountered by the present Meeting in evaluating the available data without the original studies, the Meeting recommended that the CCPR should schedule methamidophos for periodic review.

The Meeting estimated the maximum residue levels shown below for methamidophos on the basis of the residues found in supervised trials with applications of methamidophos and acephate.

Definition of the residue for compliance with MRLs and for estimation of dietary intake: methamidophos.

Commodity		Recommended MRL (mg/kg)		PHI, days	Estimated STMR, mg/kg, for dietary intake estimation
CCN	Name	New	Previous		
VB 0041	Cabbages, Head	0.5	W	14-35	0.01
VB 0404	Cauliflower	0.5	W	21	0.01
FS 0247	Peach	1[1]	W	21-35	0.16
VO 0448	Tomato	1	W	4-21	0.12

W: withdrawal recommended by 1994 JMPR

[1] Based on the residues from the use of methamidophos. The other recommendations are based on residues from the use of methamidophos or acephate

The estimated STMR-P levels for methamidophos in the food commodities listed in the Table below are recommended for use in estimates of dietary intake.

Raw agricultural commodity	STMR (mg/kg)	Processed commodity	STMR-P (mg/kg)
Peaches	0.16	Washed fruit	0.10
		Juice (100 % basis)	0.11
		Jam	0.10
		Preserve (canned fruit)	0.08

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PROPOXUR (075)

EXPLANATION

Propoxur was evaluated by the JMPR in 1973, 1977, 1981, 1983 and 1991. At the 1994 CCPR, several delegations expressed the opinion that the MRLs recommended by the 1991 JMPR for head lettuce and potatoes were based on very old data. The manufacturer stated that new data on potatoes would be available for the 1996 JMPR and that additional studies were scheduled for lettuce. New data from supervised trials on these commodities were provided to the Meeting.

METHODS OF RESIDUE ANALYSIS

Analytical methods

Samples from the newly submitted supervised trials were analysed by HPLC with on-line reaction and fluorometric detection. The method determines both propoxur and its metabolite 2-hydroxy-propoxur (2-OH-propoxur). The homogenized sample is extracted with dichloromethane and the dichloromethane evaporated. The residual material is transferred to an Extrelut cartridge with 20 ml of saturated aqueous sodium chloride solution and 2 x 25 ml of dichloromethane. The cartridge is eluted with a further 50 ml of dichloromethane and the eluate evaporated to dryness. The dry residue is dissolved in 2 ml of methanol for analysis. A reverse-phase HPLC system is used with an acetonitrile/water gradient. The column eluate is passed successively through an on-line hydrolysis reactor to produce methylamine and a derivatization reactor to form a fluorophore with *o*-phthalaldehyde and 2-mercaptoethanol. Fluorometric detection is at 340 nm excitation and 455 nm emission.

The mean recoveries from potatoes were 86% for propoxur and 84% for 2-OH-propoxur at fortification levels of 0.02-0.2 mg/kg, and from lettuce 95% for propoxur and 93% for 2-OH-propoxur at 0.04-1.0 mg/kg. The limits of determination were 0.02 mg/kg for potatoes and 0.04 mg/kg for lettuce (Blass, 1990).

In the older trials samples were analysed by a colorimetric method (lettuce in the 1960s) or by GLC (lettuce and potatoes in the 1970s). The colorimetric method is based on measurement of the reaction product of *o*-isopropoxyphenol and aminoantipyrine at 490 nm. In the GLC method the compound determined is methyl *N*-methylcarbamate which is formed in the injection port by transesterification of propoxur with methanol.

USE PATTERN

Information on use patterns was provided by the governments of Germany, The Netherlands and Poland, and the manufacturer. The uses on potatoes and lettuce are given in Table 1.

Table 1. Registered uses of propoxur on lettuce and potatoes. All spray applications. The figures in parentheses should not be compared with the application rates in the supervised trials: they are included only for reference.

Crop	Country	Form.	Application			PHI, days
			kg ai/ha	kg ai/hl	No.	
Lettuce	Germany	SL	0.18-0.24	0.03-0.04	2	7
	Italy	WP	(0.20-0.50)	0.025-0.05	1	10
	Italy	WP	(0.2-0.3)	0.025-0.038	2	10
	Netherlands	WP	0.20-0.40	0.05	1	14/21[1]
Lettuce	Netherlands	WP	0.15-0.30	0.037	2	14/21[1]
(glasshouse)	Netherlands	EC	0.40-0.60		2	14/21[1]
Potatoes	Brazil	EC	0.48-0.60	(0.048-0.075)	7	14
	Germany	SL	0.24	0.04	1	14
	Germany	SL	0.18	0.03	2	14
	Italy	EC	(0.08-0.20)	0.01-0.02	2	10
	Italy	WP	(0.38-0.53)	0.047-0.066	1	10
	Italy	WP	(0.20-0.50)	0.025-0.063	2	10
	Kuwait	EC	0.30-0.75	(0.03-0.13	1	
	Kuwait	WP	0.50-0.75	(0.05-0.13)	1	
	Poland	WP	0.30-0.50	(0.075-0.30)	2	7
	Rumania	WP	0.50	(0.063-0.083)	4	
	Saudi Arabia	EC	(0.18-0.42)	0.03-0.07	2	
	Saudi Arabia	WP	(0.30-0.45)	0.05-0.075	1	
	Netherlands	WP	0.50		2	14
	United Arab Republic	EC	0.30-0.75	(0.03-0.13)	1	
	Uruguay	EC	(0.20-0.40)	0.05	4	14

[1] 14 days from March to September and 21 days from October to February

RESIDUES RESULTING FROM SUPERVISED TRIALS

Data on residues in lettuce and potatoes were submitted to the Meeting by the manufacturer and the government of Poland (Tables 2 and 3).

Table 2. Residues of propoxur and its metabolite 2-OH-propoxur in potatoes.

Country Year	Application				PHI, days	Residues, mg/kg		Reference
	Form.	No	kg ai/ha	kg ai/hl		Propoxur	2-OH-propoxur	
Germany 1994 (6 locations)	SL	1	0.24	0.04	14 -15	≤0.02 (6)	<0.02 (6)	3
Poland 1994	EC	1	0.5		3 7	<0.02 ≤0.02		4

Country Year	Application				PHI, days	Residues, mg/kg		Reference
	Form.	No	kg ai/ha	kg ai/hl		Propoxur	2-OH-propoxur	
					14	<0.02		
Poland 1993	EC	2	0.24	0.04	23	<0.01		4

Table 3. Residues of propoxur and 2-OH-propoxur in lettuce under field conditions.

Country, Year	Application				PHI, days	Residues, mg/kg		Ref.
	Form.	No	kg ai/ha	kg ai/hl		Propoxur	2-OH-propoxur	
Lettuce	WP	2	0.24	0.04	0	2.9, 3.8, 5.1, 5.7	<0.04 (4)	2
Germany					3	0.34, 0.54, 0.67, 0.73	<0.04 (4)	
(4 locations)					4	0.32, 0.36, 0.52, 0.62	<0.04 (4)	
1991 (June)					7	0.05, 0.07, 0.10, 0.13	<0.04 (4)	
					14	<0.04 (4)	<0.04 (4)	

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

The governments of The Netherlands and Poland reported monitoring data for propoxur on several crops. The result are given in Tables 4 and 5.

Table 4. Monitoring data on propoxur in crops in Poland, 1994.

Commodity	No. of samples analysed	No. of samples with detectable residues[1]	Detection frequency, %	Residues, mg/kg
Apples	121	0		
Cabbage, white	114	2	1.8	0.1, 0.3
Carrot	18	0		
Cauliflower	30	0		
Celery	23	0		
Cherry, sour	41	0		
Currants, black	58	0		
Currants, red, white	20	0		
Onion, Bulb	21	0		
Parsley	30	0		
Potatoes	88	0		
Tomato (glasshouse)	167	0		

[1] The LOD was not reported

Table 5. Monitoring data on propoxur in crops in The Netherlands, 1991-1994.

Commodity	No. of samples analysed	No. of samples with detectable residues[1]	Detection frequency, %	Mean residues,[2] mg/kg

Commodity	No. of samples analysed	No. of samples with detectable residues[1]	Detection frequency, %	Mean residues,[2] mg/kg
Apples	87	2	2.3	<0.05
Bananas	3	1	33.3	<0.05
Celery	29	2	6.9	<0.05
Cucumbers	644	2	0.3	<0.05
Currants	620	13	2.1	<0.05
Egg plant	8	1	12.5	<0.05
Endive	104	4	3.8	<0.05
Leek	10	4	40.0	<0.05
Lettuce	2845	9	0.3	<0.05
Plums	536	2	0.4	<0.05
Raspberries	267	4	1.5	0.10
Strawberries	3343	21	0.6	<0.05
Sweet peppers	1129	7	0.6	<0.05
Wheat	185	7	3.8	<0.05

[1] LOD = 0.05 mg/kg
[2] For samples with residues below the LOD, half of the LOD is taken for calculation of the mean residues

NATIONAL MAXIMUM RESIDUE LIMITS

The following national MRLs were reported to the Meeting.

Country	Crop	MRL, mg/kg	Remarks
Australia	Potatoes	10	
Austria	Beet, sugar	1	
	Fruit	1	
	Hop	50	
	Other food of animal origin	0.05	
	Other plant commodities	0.5	
	Vegetables	1	
Belgium	All plant commodities	N.D.	<0.05 mg/kg
	Eggs	0.5	
	Fruit	3	
	Meat	0.05*	
	Milk	0.05*	
	Milk products	0.05*	
	Poultry meat	0.5	
	Vegetable	3	
Brazil (Propoxur and its metabolite expressed as propoxur)	Apple	3	
	Eggplant	3	
	Broccoli	3	
	Cabbage	3	

Country	Crop	MRL, mg/kg	Remarks
	Cabbage, white	3	
	Cacao	0.03	
	Cauliflower	3	
	Citrus fruit	3	
	Cotton seed	0.03	
	Cucurbits	3	
	Garlic	3	
	Grassland	5	
	Meat	0.05	
	Milk	0.05	
	Onion	3	
	Peach	3	
	Peanut	0.03	temporary
	Pepper, cayenne	3	
	Pepper, sweet	3	
	Plum	3	
	Potatoes	0.5	
	Poultry	0.03	temporary
	Soya	0.03	temporary
Chile	Apple	3	
Propoxur and its metabolites	Cereals	0.5	
express as propoxur	Cherry	3	
	Meat	N.D.	<0.05 mg/kg
	Milk	N.D.	<0.05 mg/kg
	Peach	3	
	Pear	3	
	Plum	3	
	Potatoes	0.5	
	Rice husked	0.1	
European Community	Fruit	3	
	Vegetables	3	
Finland	All plant commodities	3	
Germany	Fruit	3	
	Hop	50	
	Other plant commodities	0.05	
	Vegetables	3	
Greece	All plant commodities	3	
Italy	Fruit	3	
	Potatoes	3	
	Tobacco	3	
	Vegetables	3	
Japan	Cereals except rice	0.5	

Country	Crop	MRL, mg/kg	Remarks
	Fruit	1	
	Potatoes	0.5	
	Rice	1	
	Vegetables	2	
Kenya	Apple	3	temporary
	Blackberry	3	temporary
	Cherry	3	temporary
	Currant, red	3	temporary
	Gooseberry	3	temporary
	Legume animal feed, green	5	temporary
	Meat	0.05*	temporary
	Milk	0.05*	temporary
	Other vegetables	3	temporary
	Peach	3	temporary
	Pear	3	temporary
	Plum	3	temporary
	Rice husked	0.1	temporary
	Root vegetables	0.5	temporary
	Strawberry	3	temporary
Luxembourg	Fruit	3	
	Other plant commodities	0.05	
	Vegetables	3	
Malaysia	Apple	3	
	Cereals grain	0.5	
	Cherry	3	
	Rice Milled	0.1	
	Strawberry	3	
Netherlands	Eggs	0.5	
	Fruit	3	
	Meat	0.05*	
	Chicken meat	0.5	
	Milk	0.05*	
	Potatoes	0.5	
	Rice	0.1	
	Vegetables	3	
	Other food commodities	N.D.	<0.05 mg/kg
Poland	Strawberry	0.2	
	Fruit, other	3	
	Potatoes	0.1	
	Vegetables, other	3	
	Rape seed	0.5	
Portugal	Fruit	3	

Country	Crop	MRL, mg/kg	Remarks
	Potatoes	0.05	
	Vegetables	3	
South Africa	Grape	0.05	
	Grape	0.05	export tolerance
Spain	Cereals	0.05	
	Food dry	0.05	
	Forage crops straw	1	
	Fruit	3	
	Hop	0.05	
	Nuts	3	
	Oil plants seed	0.05	
	Potatoes	0.05	
	Pulses	0.05	
	Spices	0.05	
	Stimulant plants	0.05	
	Sugar plants	0.05	
	Tea	0.05	
	Tobacco	0.05	
	Vegetables	3	
Taiwan	Banana	1	
	Banana without peel	0.1	
	Papaya	1	
	Papaya without peel	0.1	
	Pineapple	1	
	Pineapple without peel	0.1	
	Rice	0.1	
Turkey	Apple	2	
	Milk	0.05	
	Pear	2	
Uruguay	Apple	3	
	Pear	3	

* at the lower limit of determination

APPRAISAL

Propoxur was evaluated by the JMPR in 1973, 1977, 1981, 1983 and 1991. At the 1994 CCPR, several delegations expressed the opinion that the MRLs recommended by the 1991 JMPR for head lettuce and potatoes were based on very old data. The manufacturer stated that new data on potatoes would be available for the 1996 JMPR and that additional studies were scheduled for lettuce. New data from supervised trials on these commodities were provided to the Meeting, together with information on GAP, analytical methods and monitoring surveys.

Analytical methods

Analyses in the new supervised trials were by HPLC with on-line derivatization and fluorometric detection. This method allows the determination of both propoxur and its metabolite 2-hydroxy-propoxur (2-OH-propoxur).

Recoveries from lettuce and potatoes were 86-95% for propoxur and 84-93% for 2-OH-propoxur and the limits of determination were 0.02 mg/kg in potatoes and 0.04 mg/kg in lettuce.

The method was considered suitable for use in supervised trials and for enforcement.

Field trials data

The Meeting evaluated newly submitted data from supervised trials on lettuce and potatoes and re-evaluated data reviewed by earlier Meetings in the light of current GAP. The Meeting agreed not to estimate STMRs for these commodities until a periodic review was undertaken, since CXLs exist for many commodities and metabolic studies were not submitted to the Meeting.

Lettuce. Six supervised trials in The Netherlands in 1963 and 1971 under glass were reported in the 1973 JMPR monograph. The residues were 0.9-20.2 mg/kg at 0-13 days and 0.5-0.8 mg/kg at 14-17 days from single applications of 0.6-0.9 kg ai/ha. The details of the trials were not clear and the treatments did not match current glasshouse GAP in The Netherlands (0.15-0.30 kg ai/ha for WP and 0.40-0.60 kg ai/ha for EC, PHI 14 days from March to September and 21 days from October to February, 2 applications).

Eight supervised field trials carried out in Germany during 1961-1964 were reported in the 1991 JMPR monograph. Samples were analysed by a very old colorimetric method and details of 4 trials (0.3-0.75 kg ai/ha, one application, 0-18 days PHI) were not clear. In the other 4 trials the conditions (0.15 or 0.6 kg ai/ha, one application, 0-8 days PHI) were not comparable with GAP in Germany (0.18-0.24 kg ai/ha, 0.03-0.04 kg ai/hl, 2 applications, 7 days PHI) or The Netherlands (0.20-0.40 kg ai/ha, one application, 14 or 21 days PHI). The trials data could not be used for evaluation.

In three field trials in 1975, reported in 1991, the conditions (0.24 kg ai/ha, 0.04 kg ai/hl, 3 applications, 0-9 days PHI) were comparable to German GAP except in the number of applications and the residues were 0.01, 0.2 and 0.3 mg/kg at 7 days PHI. The Meeting considered that the effect of the number of applications would not be significant because propoxur residues were observed to decrease rapidly during the first 7 days after application.

In 1991, supervised trials were carried out at four locations in Germany according to German GAP with the current HPLC analytical method. The residues of propoxur were 0.05, 0.07, 0.10 and 0.13 mg/kg, and of 2-hydroxy-propoxur <0.04 mg/kg in each trial.

The Meeting used the three 1975 and four 1991 German trials to estimate maximum residue levels. The residues were 0.01, 0.05, 0.07, 0.10, 0.13, 0.2 and 0.3 mg/kg.

The Meeting estimated a maximum residue level of 0.5 mg/kg for propoxur in head lettuce to replace the existing CXL of 3 mg/kg.

Potatoes. Seven supervised trials in Germany in 1971-1975 were reported in the 1991 monograph. Although most of these trials were at higher dose rates or shorter PHIs than required by GAP in Germany and The Netherlands, all of the residues were below the limit of determination of 0.1 mg/kg.

In 1994, supervised trials were carried out at six locations in Germany according to German GAP (0.24 kg ai/ha, 1 application, 14 days PHI). Analyses by the new HPLC analytical method showed propoxur and 2-hydroxy-propoxur residues to be below the limit of determination (0.02 mg/kg).

The government of Poland provided data on three supervised trials to the Meeting. The trials were in 1993 and 1994 and according to GAP (0.3-0.5 kg ai/ha, 2 applications, 7 days PHI). The residues were below the limit of determination (0.01 or 0.02 mg/kg). The Meeting concluded that adequate data on propoxur residues in potatoes determined by a modern method of analysis with an LOD of 0.02 mg/kg were now available.

The Meeting estimated a maximum residue level of 0.02* mg/kg for propoxur in potatoes to replace the existing CXL (0.1* mg/kg).

Monitoring data

In monitoring in Poland for propoxur in 1994, 731 samples of apples, white cabbages, carrots, cauliflowers, celery, sour cherries, black, red and white currants, bulb onions, parsley, potatoes and tomato (glasshouse) were analysed. No residues were found in any samples except two of white cabbage at 0.1 and 0.3 mg/kg, but information on the LOD was not available. The detection frequency was 0.3% for all samples and 1.8% for white cabbage. The residues found in white cabbage were below the national MRL (vegetables: 3 mg/kg).

Comprehensive monitoring was carried out in The Netherlands from 1991 to 1994 on 9810 samples of apples, bananas, celery, cucumbers, currants, egg plant, endive, leeks, lettuce, plums, raspberries, strawberries, sweet peppers and wheat. The overall detection frequency was 0.81% and the detection frequency for individual commodities ranged from 0.3% for cucumbers and lettuce to 40% for leeks. The mean residues in all crops were below the national MRL (fruit and vegetables 3 mg/kg, other food commodities <0.05 mg/kg).

RECOMMENDATIONS

The Meeting estimated the maximum residue levels shown below, which are recommended for use as MRLs.

Definition of the residue for compliance with MRLs: propoxur

Commodity		Recommended MRL (mg/kg)		PHI, days
CCN	Name	New	Previous	
VL 0482	Lettuce, Head	0.5	3	7
VR 0589	Potato	0.02*	0.1*	7-14

REFERENCES

(all unpublished)

Blass, W. 1990. Method for the determination of propoxur and 2-hydroxy-propoxur residues in plant materials using on-line coupling of high pressure liquid chromatography (HPLC) with a post-column fluorometric labelling technique, Report No.: RA-504/90, Method No.: 00170, Bayer AG, Germany.

Netherlands, 1996. Reports of the government of The Netherlands on monitoring, 1991-1994.

Poland, 1996. Reports of the government of Poland on supervised trials on potatoes and monitoring of various crops.

Seym, M. 1992. Determination of residues of Unden flüssing 200 EC in/on head lettuce under actual use conditions in the Federal Republic of Germany, Report No.: RA-2124/91 (0012-91, 0013-91, 0014-91, 0015-91), Bayer AG,

Seym, M. and Nüsslein, F. 1995. Determination of residues of Unden SL 200 in/on potato in Germany, Report No.: RA-2007/94 (0029-94, 0030-94, 0031-94, 0032-94, 0033-94, 0034-94), Bayer AG.

TEBUFENOZIDE

IDENTITY

ISO common name: tebufenozide

Chemical name

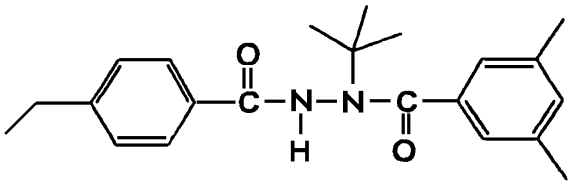
IUPAC:
N-*tert*-butyl-*N*′-(4-ethylbenzoyl)-3,5-dimethylbenzohydrazide

CA:
3,5-dimethylbenzoic acid 1-(1,1-dimethylethyl)-2-(4-ethylbenzoyl)hydrazide

CAS No: 112410-23-8

Synonyms: RH-75992, HOE-105540, MIMIC

Structural formula:



Molecular formula: $C_{22}H_{28}N_2O_2$

Molecular weight: 352

Physical and chemical properties

Pure active ingredient (Kelly, 1992b)

Vapour pressure: 2×10^{-8} torr (3×10^{-6} Pa)

Melting point: 191-191.5°C

Octanol/water partition coefficient:	mean K_{ow} 17,906. log K_{ow} 4.25
Solubility:	0.83 mg/l in water 6.5 g/100 ml in acetone 10.8 g/100 ml in methanol 24 g/100 ml in methylene chloride
bulk density:	0.28 g/ml
Hydrolysis:	stable at pH 5, 7, or 9.
Photolysis:	degraded photolytically in pond water with a half-life of 67 days (Reynolds, 1992b). Degraded photolytically when adsorbed on soil with a half-life of 98 days (Reynolds, 1991).

Technical material (Kelly, 1992a)

Purity:	94-97% (average 96%)
Melting point:	188.5-190.0°C
Stability:	stable at ambient temperatures for at least two years

Formulations

The main formulations are two aqueous suspension concentrates and a wettable powder. The suspension concentrates are the 2 SC containing 240 g ai/l and the 200 SC containing 200 g ai/l. The wettable powder contains 700 g ai/kg. Several other formulations have been developed for use specifically in rice: a 7.5 g ai/kg wettable powder, an 8 g ai/kg wettable powder, a 100 g ai/kg wettable powder, and a 1 g ai/kg granule.

METABOLISM AND ENVIRONMENTAL FATE

Radiolabels

Three different ^{14}C compounds were used. One labelled in the ring carbons of the 4-ethylphenyl ring, referred to as "A-ring labelled", the second labelled in the ring atoms of the 3,5-dimethylphenyl ring and referred to as "B-ring labelled" and the third with the central carbon of the *tert*-butyl group lebelled and referred to as "t-butyl labelled".

In studies where it was possible or likely that the entire molecule would be metabolized all three labelled compounds were used to allow for the isolation and identification of fragments of the parent compound. In other studies only one label was used.

Animal Metabolism

The metabolism of tebufenozide has been studied in rats, lactating goats, and laying hens. In each case the test material was administered orally. All the studies included test material labelled in all three positions.

Rats. The metabolism of tebufenozide in male and female rats was studied using a single high dose (250 mg/kg), a single low dose (3 mg/kg), or a pulse dose (3 mg/kg) given after dosing for two weeks with unlabelled test material. The low-dose study was with all three radiolabels, the high-dose study with the B-ring and t-butyl labels and the pulse dose study with only the t-butyl label. Each test group consisted of 5 male or 5 female rats. Pooled samples of urine and faeces from each group, each containing >95% of the excreted radioactivity, were extracted and analysed (Hawkins *et al.*, 1992b).

For the determination of metabolites samples from the 5 animals in each test group were combined for analysis. After dosing, urine and faeces were collected at approximately 24-hour intervals for 7 days.

Almost all the radioactivity (94-104%) in all test groups was eliminated in the faeces. Low levels (0.5-8.0%) were excreted in the urine and only traces of radioactivity were left in the carcase, blood and tissues or excreted as carbon dioxide or other volatile compounds.

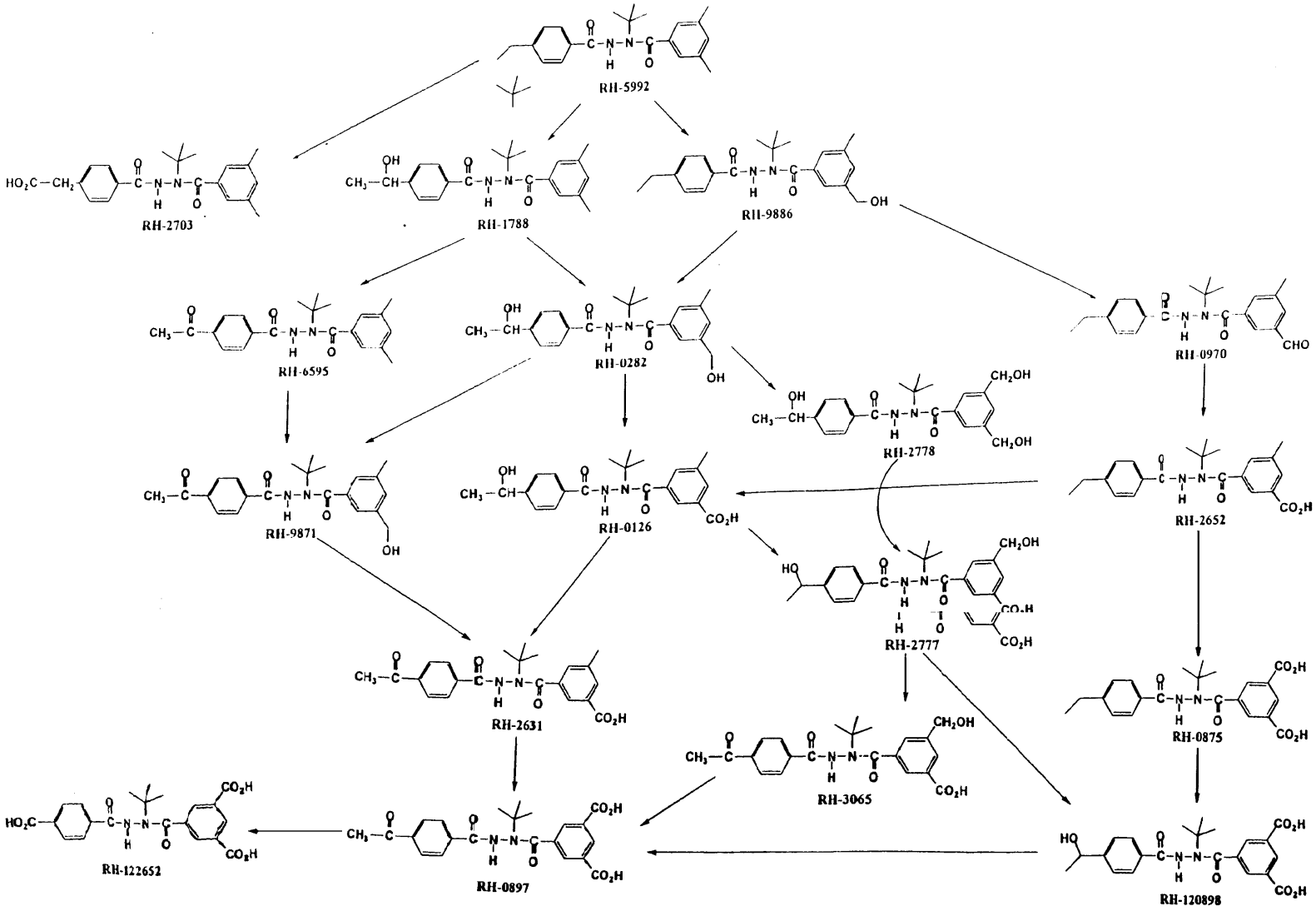
The extent of metabolism in the high- and low-dose groups was found to be highly dependent on the dose. When fed a nominal dose of 250 mg/kg, <4% of the dose was metabolized. When fed a nominal dose of 3 mg/kg about 46% was metabolized.

No qualitative differences in metabolism between the different labelled compounds were seen in the faeces samples from either high or low doses where multiple labels were used. Most of the activity in the high-dose faeces was due to parent tebufenozide. In the low-dose samples the parent accounted for 35-43% of the dosed activity. In addition to the parent compound a total of eleven metabolites were identified in the high-dose faeces samples: RH-6595, RH-1788, RH-9886, RH-2631, RH-9871, RH-0126, RH-0282, RH-0897, RH-120898, RH-0875, and RH-0970. All of these metabolites except RH-0970 were also found in the low-dose faeces samples together with four others, RH-2703, RH-3065, RH-2777, and RH-2778, which were not identified in the high-dose samples. The structures of the metabolites are shown in Figure 1.

In the pulse dose study, male and female rats were dosed with 30 ppm of unlabelled compound in the diet for two weeks before administration of a single 3 mg/kg dose of labelled tebufenozide. The metabolism in these rats was qualitatively the same as in the rats receiving only a low single dose of the test material, but higher levels of the more highly oxidized metabolites were observed (Hawkins *et al.*, 1993).

The faeces samples showed a similar metabolic profile to the low-dose samples, but they did not contain RH-9871 or RH-2703 and contained one additional metabolite, RH-122652. The parent compound accounted for 26-39% of the dose.

Figure 1. Proposed metabolic pathways of tebufenozide in rats.



No tebufenozide was found in the urine nor were several of the less polar faecal metabolites, but eleven of the metabolites identified in the faeces were also found in the urine, together with one additional whole-molecule metabolite, RH-2652. Although no differences were observed between the metabolites in the faeces produced from the three different labelled compounds the same was not true of the metabolites in the urine samples; small amounts of cleavage products containing only the A-ring or only the B-ring label (none greater than 1.5% of the dosed activity) were also seen but not identified. Some compounds with the B-ring label were similar in chromatographic behaviour to some of the whole-molecule metabolites.

As can be seen from Figure 1, all identified metabolites result from oxidation of the alkyl substituents of the aromatic rings of tebufenozide, particularly at the carbons adjacent to the rings. On the A-ring side of the molecule, this can result in either a secondary alcohol or a ketone, or an acid produced by oxidation of the terminal carbon of the ethyl group. On the B-ring side there are two positions which can be oxidized to combinations of alcohols acids.

Table 1. Tebufenozide and its metabolites in the combined urine and faeces of rats (Hawkins *et al.*, 1992, 1993).

Compound (see Figure 1)	% of ^{14}C in dose					
	High-dose[1]		Low-dose[1]		Pulse dose[2]	
	Male	Female	Male	Female	Male	Female
Tebufenozide	96.6	99.7	43.5	34.6	39.3	26.1
RH-6595	0.48	0.40	2.5	2.8	0.6	0.8
RH-2652	0.002	0.12				
RH-1788	0.19	0.31	0.58	0.8	1.7	2.0
RH-2631	0.11	0.1	10.0	1.0	3.5	3.8
RH-9886	0.19	0.28	1.04	0.99	0.2	0.26
RH-2703			0.26		0.44	0.18
RH-0126	0.22	0.03	3.83	3.3	5.7	4.5
RH-0282	1.4	1.1	2.64	13.9	11.1	16.3
RH-0875	0.06	0.11	3.8	0.1	0.57	
RH-9871	0.14	0.38	1.74	1.54	0.84	1.23
RH-0897	0.07	0.03	1.81	0.12	3.35	0.34
RH-120898	0.04		2.2	3.1	6.1	15.2
RH-2778		0.003	0.11	8.3	3.5	2.7
RH-0970	0.4	0.5				
RH-2777	0.006		0.41	0.34	0.39	10.9
RH-3065	0.02		1.63	1.05	2.49	0.41
RH-122652					1.24	4.41
Unknowns[3]	0.19	0.14	1.1	6.6	3.0	4.7
TOTAL	100.1	103.2	77.1	78.5	84.0	93.8

[1] Hawkins *et al.*, 1992
[2] Hawkins *et al.*, 1993
[3] Only whole-molecule compounds

Livestock. The fate of tebufenozide fed to lactating dairy goats was studied by dosing three goats orally with tebufenozide labelled in all three parts of the molecule at the equivalent of approximately 50 ppm in the feed for 7 consecutive days. The goats weighed between 45 and 60 kg. Milk, urine, and faeces were collected during the dosing period and the animals were killed within 24 hours after the last dose. No adverse effects of the dosing were observed. The samples collected for analysis were composite fat, consisting of equal portions of omental and perirenal fat, kidneys, liver, and composite muscle consisting of equal portions of longissimus dorsi, semimembranosus, and triceps muscle (Bender *et al.*, 1995). The total radioactivity recovered in the excreta, tissues, and milk was 88.2% of the A-ring dose, 87.3% of the B-ring, and 90.0% of the t-butyl. Table 2 shows the recovery of the administered activity in the separate samples.

Table 2. Recovery of ingested radioactivity from goats fed with [^{14}C]tebufenozide (Bender *et al.*, 1995).

Sample	% of total dose of ^{14}C		
	A-ring ^{14}C	B-ring ^{14}C	t-butyl ^{14}C
Faeces	79.0	78.3	81.1
Urine	8.9	8.6	7.8
Liver	0.07	0.12	0.4
Composite fat	0.14	0.15	0.26
Composite milk	0.09	0.08	0.26
Composite muscle	0.02	0.06	0.16
Kidney	<0.01	<0.01	0.01
Heart	<0.01	<0.01	<0.01
Total	88.2	87.3	90.0

The test compound was eliminated mainly in the faeces where 79.0%, 78.3% and 81.1% of the total dose was recovered from the A-ring, B-ring, and t-butyl labels respectively. The urine accounted for 8.9% of the A-ring, 8.6% of the B-ring, and 7.8% of the t-butyl activity. Thus 87% to 89% of the administered dose was eliminated from the body via the excreta. Less than 0.3% of the dose was excreted in the milk during the 7-day dosing period: 0.09%, 0.08%, and 0.26% of the total dose from the A-ring, B-ring, and t-butyl labels respectively. Body tissues contained small amounts of activity: fat contained the highest percentage of the dose in the A-ring and B-ring samples, 0.14% and 0.15%, and the liver the highest amount from the t-butyl label (0.4% of the dose). Detectable residues were found in the muscle, heart, and kidney of all the goats. Residue levels in the tissues expressed as tebufenozide equivalents are shown in Table 3.

Table 3. Residues of ^{14}C as tebufenozide equivalents in the milk and tissues of goats (Bender *et al.*, 1995).

Sample	^{14}C, mg/kg tebufenozide equivalents		
	A-ring ^{14}C	B-ring ^{14}C	t-butyl ^{14}C
Liver	0.50	0.99	2.87
Fat	0.17	0.14	0.29
Kidney	0.04	0.06	0.30
Muscle	0.007	0.02	0.06
Milk (day 2)	0.07	0.07	0.16

Milk, fat, liver, kidney, muscle, and urine samples were analysed to determine the nature of the residue. Milk samples were extracted with chloroform, methanol and water, fat samples with hexane and methanol, and muscle, kidney and liver with various solvent mixtures which included acetonitrile, water, chloroform and methanol. Urine samples were extracted with ethyl acetate and butanol. The quantitative determination of the metabolites was carried out by TLC, GLC, HPLC and MS.

Residue levels of ^{14}C as tebufenozide in the milk remained relatively constant throughout the dosing period. With the exception of a single residue of 0.12 mg/kg seen on day 6 from the B-ring label, residues in the A- and B-ring milk samples remained consistently in the range 0.05-0.07 mg/kg throughout the 7 days. Residues in the t-butyl milk were approximately twice those found in the other samples, from 0.09 mg/kg on day 1 to a maximum of 0.17 mg/kg on day 5.

The total radioactive residue (TRR) in the milk samples from day 2 corresponded to 0.07 mg/kg from the A- and B-ring labels and 0.16 mg/kg from the t-butyl label. More than 90% of the A- and B-ring activity was extractable with, or partitioned into, organic solvents. Only slightly more than 40% of the original t-butyl activity could be extracted in the same manner: most of the remainder, 37% of the TRR, remained in the aqueous fraction and was characterized as arising from polar, non-volatile small molecules, probably lactose and/or amino acids. The t-butyl label also had the highest proportion of unextractable activity, nearly 10%. Hydrolysis with dilute acid or base released about one third of this activity, all of which was insoluble in ethyl acetate.

In the milk, all 3 labels appeared in the same residues, generally in similar concentrations. The milk contained two major components of the TRR. One or more fatty acid conjugates of the B-ring alcohol RH-9886 represented 17-24% of the total activity from the A- and B-ring labels (0.01-0.017 mg/kg). Tebufenozide was detected at a concentration of approximately 0.01 mg/kg from each of the labels, and represented 13.7% of the TRR. Three other alcohol metabolites were also identified: RH-0282, RH-9871 and RH-9886.

Residue levels of ^{14}C as tebufenozide in fat samples ranged from 0.143 mg/kg from the B-ring label to 0.286 mg/kg from the t-butyl. More than 90% of the activity in the A- and B-ring labelled fat samples was extractable with or partitioned into organic solvents, but only about 55% of the t-butyl activity was in these fractions. Almost 18% of the t-butyl activity was found in the aqueous fraction, with close to 27% of the activity remaining in the post-extraction solids (PES). After saponification, most of the PES activity was released and partitioned into both aqueous and organic extracts. Radioactive residues in the fat comprised solely the parent compound and three fatty acid conjugates of RH-9886, the conjugated alcohol present in milk. The fatty acids were identified by mass spectrometry

as palmitic, oleic, and stearic. The relative amounts of the parent and the conjugates varied in the three samples. Residues of tebufenozide were highest in the B-ring labelled fat, contributing 69.1% of the total residue at a concentration of 0.1 mg/kg. The lowest concentration of the parent, <0.02 mg/kg, was found with the t-butyl label, where it represented only 5.4% of the TRR.

The liver contained the highest concentration of residues with all 3 labels of any of the tissues analysed, ranging from 0.5 mg/kg from the A-ring to 2.9 mg/kg from the t-butyl label. The 3-6-fold higher residue levels in the t-butyl sample suggested some fragmentation of the parent molecule.

The major component of the A- and B-ring labelled liver was RH-2703, in which the terminal group of the ethyl substituent on the A-ring has been oxidized to carboxyl. This compound was also present in large quantities in the t-butyl labelled liver, but that also showed an approximately equal amount of one of the fatty acid conjugates of RH-9886. Numerous oxidative degradation products were present with all 3 labels. The major metabolites identified with the t-butyl label were volatile: 2-propanol and acetaldehyde. No residues of the parent were seen in the liver.

Residues in kidney samples were identified as a small amount of unmetabolized parent, together with several of its metabolites. Residues of tebufenozide were well below 0.01 mg/kg with each of the labels and represented between 0.9 and 12.8% of the TRR. Residues of RH-9886, RH-0282, and RH-2631 were also identified. The highest concentration of any single metabolite was 0.015 mg/kg of RH-0282 in the B-ring labelled sample (23.3% of the TRR in the kidney).

Muscle contained the lowest levels of radioactive residues of any samples analysed. The TRR ranged from less than 0.01 mg/kg with the A-ring label to 0.06 mg/kg with the t-butyl label. The parent and 3 of its metabolites were identified with all three labels. Samples with the B-ring label contained the highest amount of unmetabolized parent, 45.1% of the TRR, at a concentration of 0.008 mg/kg. The concentrations of tebufenozide with the A-ring and t-butyl labels were 0.002 and 0.005 mg/kg respectively (32.6% and 8.9% of the TRR). Three alcohol metabolites were detected: RH-9886, RH-0282, and RH-2778. All were present at concentrations below 0.004 mg/kg. The distribution of the identified residues is shown in Table 4.

Table 4. Residues identified in goat milk and tissues (Bender *et al*, 1995).

Compound	% of total ^{14}C in sample[1]				
	Milk (day 2)	Fat	Liver	Kidney	Muscle
Tebufenozide	13.7	47.4	-	11.4	38.9
Fatty acid conjugates(s) of RH-9886	20.8	20.5	5.0	-	-
RH-0282	9.3	-	2.7	15.7	24.8
RH-9871	6.7	-	1.2	-	-
RH-9886	1.0	-	2.4	12.1	10.1
RH-2703	-	-	47.4	-	-
RH-2777	-	-	1.6	-	-
RH-2778	-	-	-	-	13.9
RH-0126	-	-	3.4	-	-
RH-2631	-	-	-	15.0	-
2-propanol	-	-	44.3	-	-
Acetaldehyde	-		5.5	-	-

[1] Mean percentage of A- and B-ring labels in milk, fat, kidney and muscle, and in liver for RH-0282, 9886, 2703 and 0126. Percentage of t-butyl label in liver for other compounds.

Tebufenozide is extensively metabolized by multiple oxidative transformations in goats. In most samples the parent compound represented only a small proportion of the TRR and the same residues were found with all 3 labels, indicating that the metabolites contained the intact molecular skeleton of the parent molecule. One of the resulting alcohols, RH-9886, was found conjugated to fatty acids in milk and fat. Low levels of two other alcohol metabolites and a carboxylic acid were also found in milk. Liver was exceptional in that residues of the parent compound were not found: the major metabolites identified with the t-butyl label were 2-propanol and an approximately equal amount of RH-2703. The proposed metabolic pathways of tebufenozide in lactating goats are shown in Figure 2.

Poultry. Laying hens (6 groups of 10 hens each, 25 weeks old) were dosed by capsule with labelled tebufenozide for 7 days at a level equivalent to 30 ppm in the feed. The average feed intake was 110 g/bird/day. Another group of 10 hens served as the control. Two of the 6 groups received the A-ring label, another two the B-ring and the remaining two the t-butyl label. Samples of excreta and eggs were collected daily for the 7-day dosing period, and tissues samples were collected after all the birds were killed 24 hours after the final dose. The recovery of the ^{14}C in the A- and B-label groups was nearly quantitative, but in the t-butyl group only about 80%, probably owing to extensive degradation to produce the volatile metabolites identified in liver and possibly loss as CO_2. The total radioactivity in the tissues varied with the label, and was generally highest with the t-butyl. Significantly different TRRs from the three labels implied extensive breakdown of the molecule. The average residues found in tissues and eggs are shown in Tables 5 and 6 (Schuck and Sharma, 1996).

Table 5. ^{14}C residues in hen tissues (Schuck and Sharma, 1996).

Sample	^{14}C, mg/kg as tebufenozide, mean of 2 groups		
	A-ring label	B-ring label	t-butyl label
Liver	0.13	0.18	3.95
Fat	0.13	0.06	0.16
Thigh muscle	0.02	0.01	0.07
Breast muscle	0.006	0.0000	0.03
Kidney	0.13	0.12	0.97

Figure 2. Proposed metabolic pathways of tebufenozide in goats.

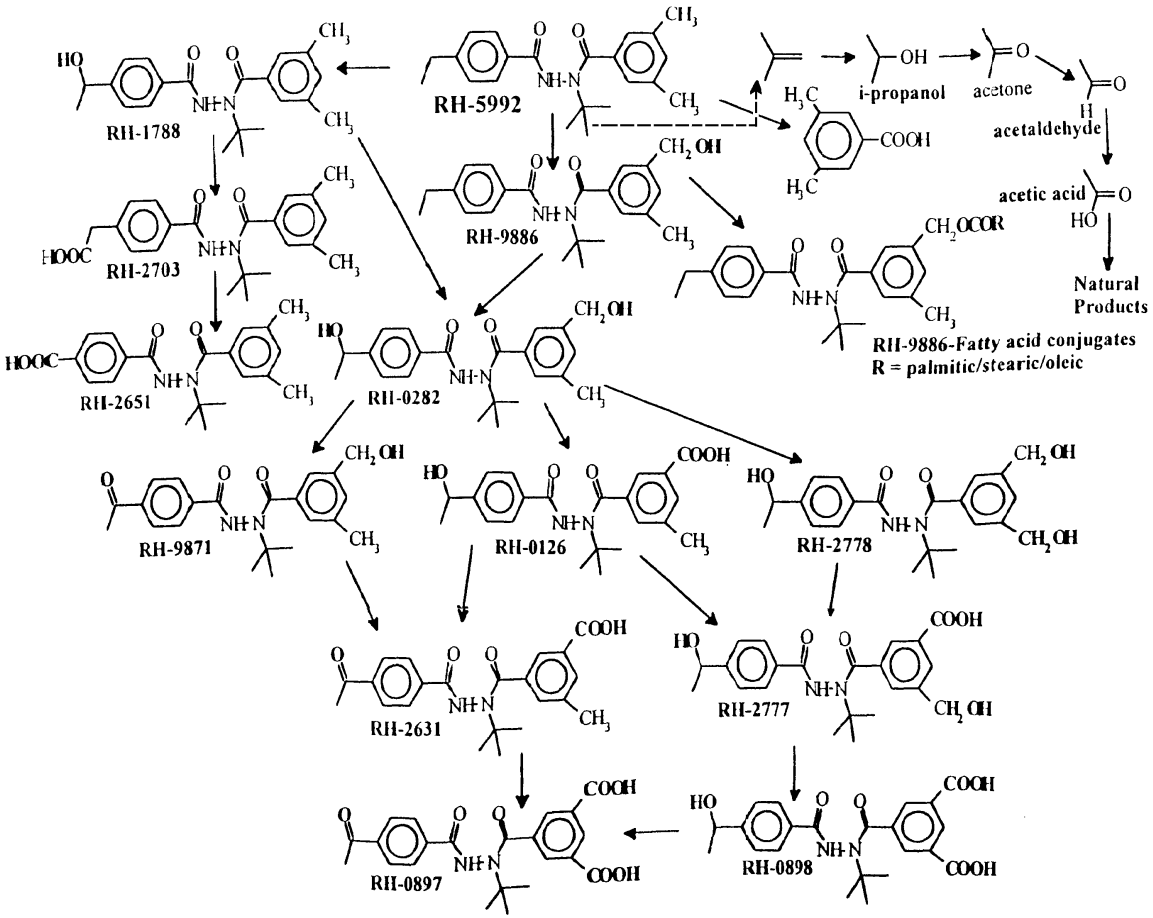


Table 6. ^{14}C residues in whole eggs.

Label	^{14}C, mg/kg as tebufenozide, mean of 2 groups						
	Day 1	Day 2	Day 3	Day 4	Day 5	Day 6	Day 7
A-ring	0	0.02	0.03	0.04	0.06	0.06	0.07
B-ring	0	0	0.01	0.04	0.02	0.03	0.03
t-butyl	0	0.03	0.05	0.04	0.09	0.11	0.13

The hens dosed with the t-butyl label showed the highest TRR in eggs in almost all the samples collected.

The analysis of eggs showed only traces of whole-molecule metabolites such as the parent compound, RH-9886, RH-9871 and a fatty acid conjugate of RH-9886. The analysis of fat also showed a high percentage of radiocarbon from the A-ring and t-butyl labels incorporated into the fatty acids themselves.

The t-butyl labelled liver sample contained a very high percentage of the TRR as a volatile residue. More than 30% was shown to consist of two volatile components, 2-propanol and acetaldehyde, and another volatile residue was identified as acetic acid. RH-2277, RH-2778, RH-0897, RH-0126 and RH-0282 were detected in small quantities.

The residue in muscle was very low. Solvent-extractable residues in thigh muscle amounted to about 0.03 mg/kg or less and the remainder was extractable after treatment with proteolytic enzymes.

Excreta were the main source of metabolites in this study: most of the residue was soluble in organic solvents and only a small proportion was water-soluble. The compounds were identified by TLC and HPLC, and further confirmed by mass spectrometry. Tebufenozide, RH-0126, RH-0282, RH-2777 and RH-2778 were present in amounts exceeding 10%. RH-1788, RH-9886, RH-9871, RH-0897 and 3,5-dimethylbenzoic acid were also detected.

Table 7. Metabolites identified in the eggs, tissues and excreta of hens.

Compound	Residues, mg/kg				
	Eggs[1]	Liver	Fat	Muscle	Excreta
Tebufenozide	0.005		0.18	nd	Detected
RH-9886	0.001[2]				Detected
RH-0282	0.003	Detected	0.03	0.008	Detected
RH-9871	0.003				Detected
RH-9886 conj.	0.002[3]		0.01		
RH-1788/9886				0.005[3]	Detected
RH-2631				0.004[4]	
RH-2778		Detected		<LOD	Detected
Rh-0126		Detected		<LOD	Detected
RH-0897		Detected			

Compound	Residues, mg/kg				
	Eggs[1]	Liver	Fat	Muscle	Excreta
RH-2777		Detected		<LOD	Detected
3.5-dimethylbenzoic acid					Detected
acetaldehyde + 2-propanol		2.56			Detected
acetic acid		0.45			
others & unknown	0.04[5]		0.01[3]		
Natural product incorporation	>20%		>30%		

[1] A-ring label groups collected on day 5, B-ring and t-butyl label groups on day 7
[2] A-ring label only
[3] A-ring and t-butyl label
[4] A- and B-ring labels
[5] t-butyl label only

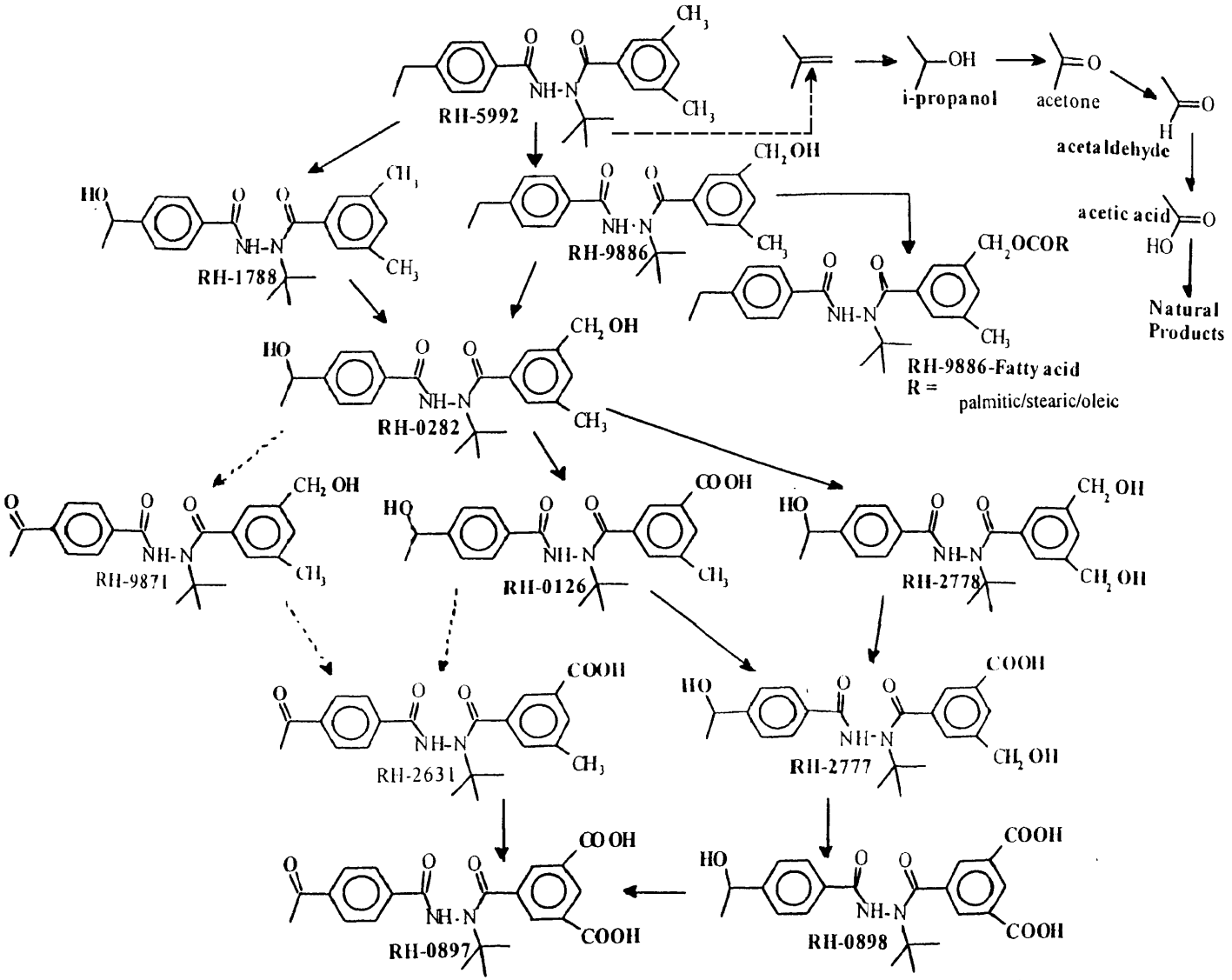
The metabolic degradation of tebufenozide in hens proceeded via oxidation of the ethyl and methyl groups of the A- and B- rings, hydrolysis of the amide portions which released the free benzoic acids and oxidative degradation of the *tert*-butyl group which resulted in the small volatile molecules 2-propanol, acetaldehyde and acetic acid. A proposed degradation pathway in hens is shown in Figure 3.

Fish. To study the kinetics of the uptake and elimination of tebufenozide, Bluegill sunfish were continuously exposed to a nominal concentration of 50 µg/l for 29 days. Each of three groups was exposed to [^{14}C] tebufenozide with one of the three labels. Thirty-five fish were then transferred from each of the three exposure aquaria to their respective depuration aquaria for a 15-day depuration period in fresh water, to determine the half-life for the loss of tebufenozide from tissues. Fish, divided into edible and inedible components, and water samples were taken at eight intervals during the exposure period and at 5-day intervals during the depuration phase, and analysed by radioassay (Christensen, 1992).

The concentrations of ^{14}C in the edible and inedible tissues and whole bodies of the fish reached a statistically determined steady state during the first day of exposure. The mean steady state concentration in the tissues and the bioconcentration factor (BCF) for each of the three radiolabels is shown below.

Label	^{14}C, mg/kg as tebufenozide, and bioconcentration factors in fish					
	Edible tissue		Inedible tissue		Whole body	
	mg/kg	BCF	mg/kg	BCF	mg/kg	BCF
A-ring	0.46	8.7	4.30	81	2.20	42
B-ring	0.32	5.9	8.3	150	3.80	70
t-butyl	0.41	8.0	4.5	88	2.20	43

Figure 3. Proposed metabolic pathways of tebufenozide in hens.



The reported half-life for depuration was less than three days for all labels. By the last (15th) day of depuration at least 90% of the radioactivity had been eliminated from the fish.

In another study to determine the rate and extent of tebufenozide bioconcentration in fish, Bluegill sunfish were also exposed for 29 days to nominal concentrations of 50 µg/l of tebufenozide labelled at the three sites as before. The concentration in the fish rapidly reached a steady state level. The BCF was 7.5 for the edible tissue and 52 for the whole body. Other fish were exposed to tebufenozide labelled in the A-ring at a nominal concentration of 300 mg/l for 14 days for the identification of metabolites.

The main residue in the fish extracts was unmetabolized tebufenozide but eight metabolites, RH-0126, RH-2777, RH-2778, RH-2652, RH-2631, RH-0282, RH-9886 and the A-ring ketone/B-ring diol of tebufenozide, were also isolated and identified (Dong and Hawkins, 1993).

The metabolic profiles in the edible and inedible tissues were the same: they are shown in Table 8. No other metabolites constituted more than 10% of the residue in any tissue. The proposed metabolic pathways for tebufenozide in fish are shown in Figure 4.

Figure 4. Proposed metabolic pathways of tebufenozide in fish.

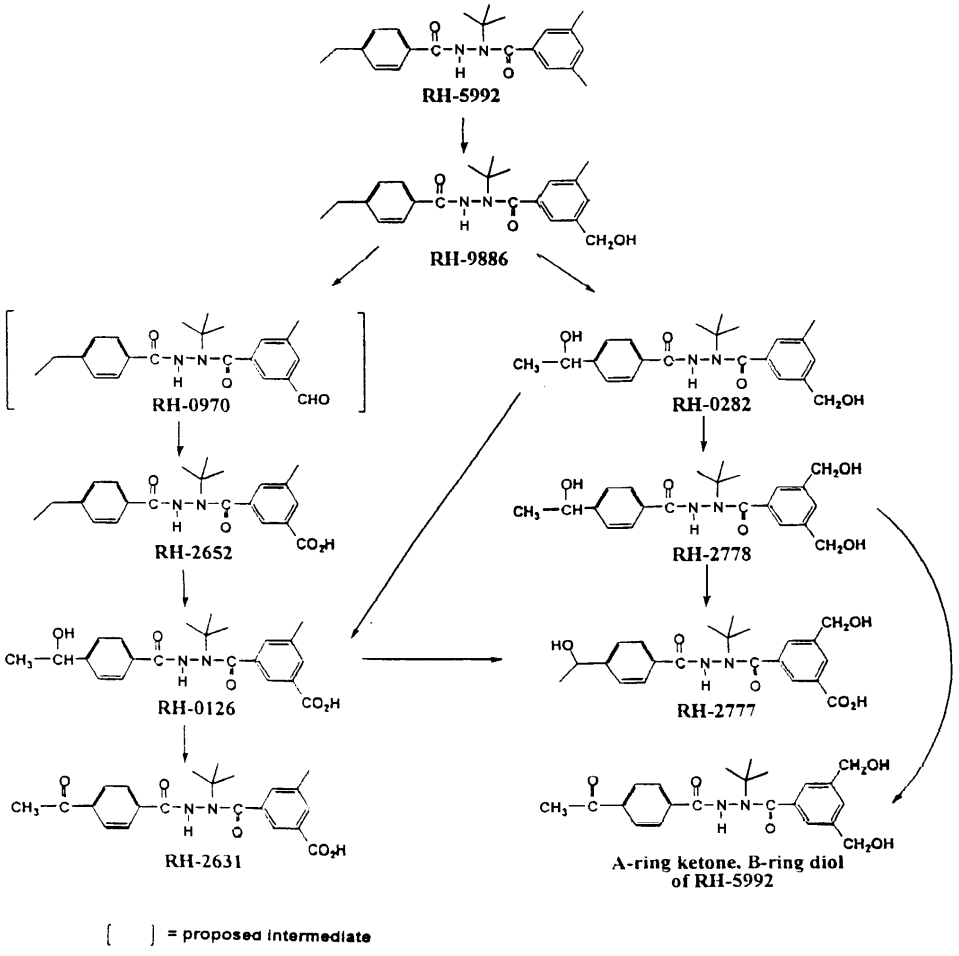


Table 8. Residues in edible and inedible tissues of fish exposed to [^{14}C]tebufenozide (Dong and Hawkins, 1993).

Compound	% of total residue			
	Edible tissue		Inedible tissue	
	t-butyl label	B-ring label	t-butyl label	B-ring label
Tebufenozide	45.6	58.8	39.4	17.3
RH-0126	10.5	17.8	19.7	21.8
RH-2652	2.3	1.9	3.6	4.2
RH-2778	5.3	3.6	2.7	5.0
RH-2777	2.9	5.0	1.8	3.8
RH-0282	2.0	0.9	0.2	0.2
RH-2631	*	*	1.3	2.5
RH-9886	*	*	*	*
A-ring ketone/ B-ring diol	*	*	*	0.2

* Below the limit of detection in fish exposed to 50 µg/l, but observed in fish exposed to 300 µg/l

Plant metabolism

Plant metabolism was studied in apples, grapes, rice, and sugar beet. All plants except apples, were treated with tebufenozide with all three radiolabels, apples only with material labelled in the A-ring.

Grapes. Grape vines (mature Concord) were treated once with [^{14}C]tebufenozide applied as a 10% EC formulation at a nominal rate of 1.2 kg ai/ha. Application was with a backpack compressed air sprayer. Samples of leaves and fruit were collected 0, 15 and 31 (harvest) days after treatment. Leaves were taken only to determine the total radioactivity, not for metabolite identification (Hawkins *et al.*, 1991).

Samples were extracted with aqueous methanol which extracted more than 98% of the ^{14}C of all three labels.

Residues in the extract were partitioned between ethyl acetate and water. The ethyl acetate fractions were analysed by HPLC and TLC. Only one radioactive component was observed and confirmed to be parent tebufenozide, which is evidently not metabolized by grapes. The three labels gave similar results, which are shown in Table 9.

Table 9. Residues in grapes and leaves after application of [^{14}C]tebufenozide to vines (Hawkins, 1991).

Sample	^{14}C, mg/kg as tebufenozide, at intervals (days)								
	A-ring label			B-ring label			t-butyl label		
	0	15	31	0	15	31	0	15	31
Leaves	187.3	41.4	93.4	136	39.3	36.4	229.4	119	115
Grapes	1.95	0.72	1.0	0.35	0.69	1.27	3.15	1.64	2.45

Rice. The metabolism of tebufenozide in rice was studied in a field experiment in California. Four 7.2 m^2 plots were lined with plastic, filled with 45 cm of a sandy loam soil and flood-irrigated after planting until final harvest. Tebufenozide, labelled in all three positions, was applied three months after planting at a nominal rate of 1.2 kg ai/ha, higher than the expected use rate. One plot served as a control. Straw, grain, paddy soil and paddy water samples were collected before and immediately after application, then at 15 days, 30 days, and at mature harvest after 64 days (Randazzo, 1992).

Straw and grain samples were chopped in a blender and ground to a fine powder in a mill. Mature grain was hulled before processing. The total radioactivity in the rice samples was determined by combustion analysis. The results are given in Table 10.

Table 10. Total radioactivity in rice treated with [^{14}C]tebufenozide (Randazzo, 1992).

Sample	Days after treatment	^{14}C, mg/kg as tebufenozide		
		A-ring label	B-ring label	t-butyl label
Straw	15	25.23	38.16	30.0
Straw	30	36.59	27.1	37.46
Straw	64	62.3	68.32	23.68
Immature grain	15	3.06	3.0	3.11
Immature grain	30	2.28	3.18	2.6
Mature grain (hulled)	64	0.33	0.40	0.29
Hulls	64	7.03	13.86	11.19

The residues in the straw, grain and hull samples were extracted with acidified aqueous acetonitrile and partitioned into organic and aqueous fractions. Most of the activity was found in the organic fraction, which was analysed after solid-phase extraction by TLC and HPLC with radiometric detection. The average recoveries were 80.6-99.1% from straw, 92-105% from grain, and 97% from hulls. The main residue in all the samples was found to be the parent compound. Four metabolites were isolated, none of which accounted for more than 10% of the sample activity. The metabolites were identified by mass spectrometry. The proposed metabolic pathway is shown in Figure 5. The metabolic profile in the final harvest samples is shown in Table 11.

Table 11. Residues in rice at harvest after treatment with [^{14}C]tebufenozide.

Sample	Compound	% of total recovered radioactivity		
		A-ring label	B-ring label	t-butyl label
Straw	Tebufenozide	78.7	77.9	71.9
	RH-0970	1.2	1.2	1.1
	RH-6595	1.1	1.3	1.3
	RH-1788	1.8	2.0	2.8
	RH-9886	1.3	1.2	1.5
Hulls	Tebufenozide	58.2	63.0	62.6
	RH-0970	1.0	1.2	1.6
	RH-6595	4.8	3.6	3.9
	RH-1788	9.4	6.6	6.9
	RH-9886	1.1	1.2	1.3
Grain	Tebufenozide	51.7	49.5	52.0
	RH-0970	1.5	1.4	1.5
	RH-6595	4.6	4.8	4.8
	RH-1788	8.8	9.4	9.8
	RH-9886	1.2	1.1	1.2

The post-extraction solids contained 9.5-17% of the total activity. The activity from one straw and one grain sample was released by mild basic hydrolysis and found to represent mainly the parent compound, with small amounts of the metabolites found in the primary extraction.

Sugar beet. Metabolism in sugar beet was studied in a field experiment in California. The three ^{14}C-labelled versions of tebufenozide were each isotopically diluted with the corresponding ^{13}C-labelled or unlabelled compound.

Each labelled tebufenozide was used as a 5% EC to treat a separate plot of sugar beet at a nominal rate of 2.24 kg ai/ha. One plot was untreated as a control. Sugar beet tops and roots were harvested at 0, 30, 61, and 120 days after treatment. The samples taken at day 0 were not analysed.

Root samples were rinsed with water, air-dried and homogenized in liquid nitrogen. Leaf samples were homogenized without rinsing. The total radioactivity in the samples, determined by combustion radioanalysis and expressed as mg/kg tebufenozide equivalent, are shown in Table 12. The residue levels in the control samples were all <0.01 mg/kg (Wu, 1993a).

Table 12. Residues in sugar beet after treatment with [^{14}C]tebufenozide (Wu, 1993a).

Sample	Days after treatment	^{14}C, mg/kg as tebufenozide		
		A-ring label	B-ring label	t-butyl label
Tops	30	2.75	4.09	2.63
	61	0.76	1.13	0.96
	120	0.44	0.27	0.56
Roots	30	0.40	0.84	0.44
	61	0.35	0.66	0.57
	120	0.16	0.23	0.13

Most of the residue, 38-59% in the final harvest beet tops and 59-83% in the roots, was found to be the parent compound, confirmed by isolation and mass spectrometry. In addition twelve metabolites were isolated and identified, none of which accounted for more than 10% of the total activity. Four of them (RH-2703, RH-2631, RH-0126, and RH-0897) were also found as conjugates in small quantities. The proposed metabolic pathways are shown in Figure 6, and the distribution of metabolites with the B-ring label in the final harvest samples is shown in Table 13.

Figure 5. Proposed metabolic pathways of tebufenozide in rice.

tebufenozide

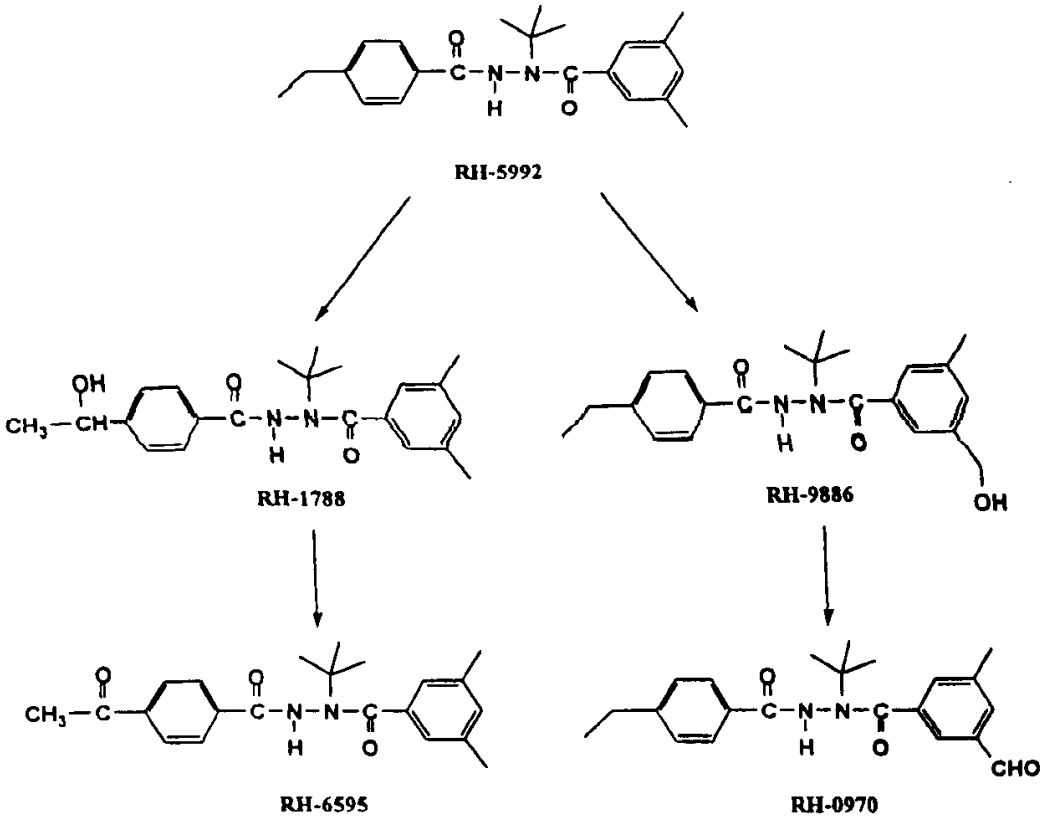
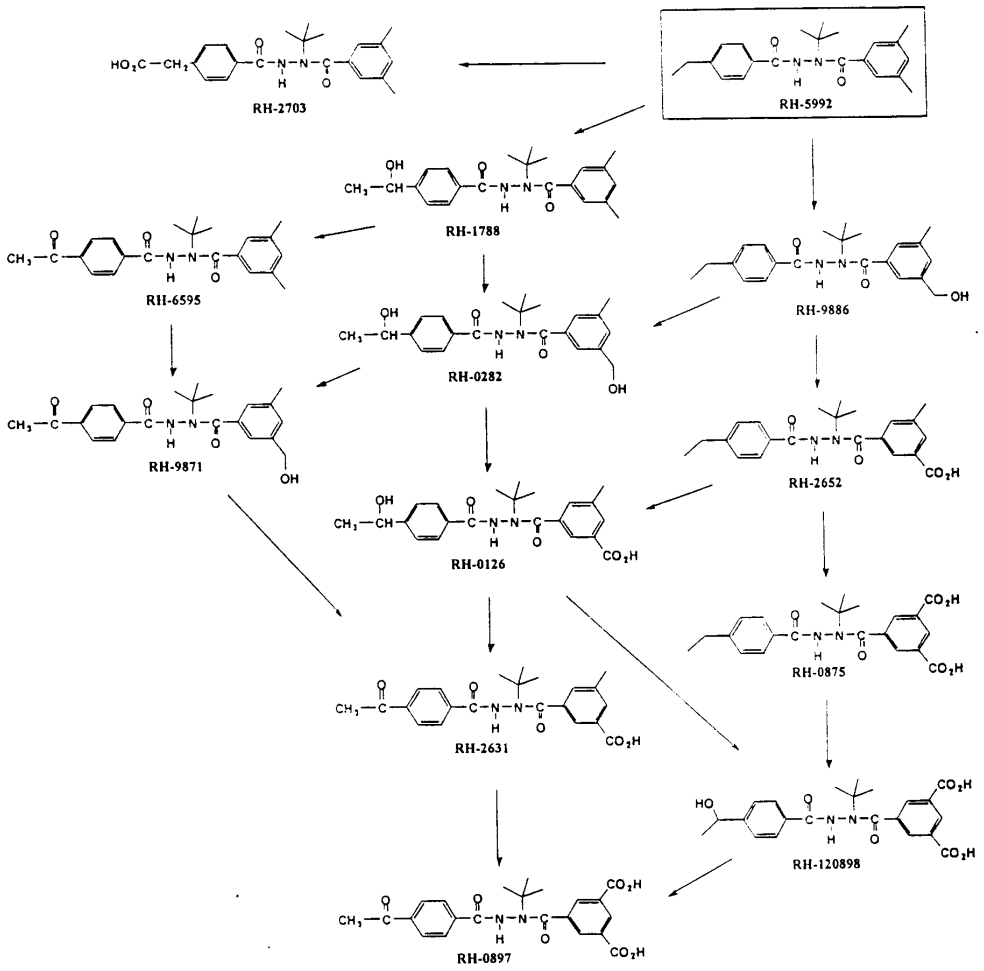


Table 13. Distribution of radioactivity in sugar beet treated with tebufenozide labelled in the B-ring (Wu, 1993a).

Compounds	% of total radioactivity	
	Tops	Roots
Tebufenozide	41.4	66.6
RH-6595	0.65	2.8
RH-9886	0.05	0.06
RH-112652	0.04	-
RH-1788	1.7	0.8
RH-2703	2.6	0.8
RH-2703 conj.	2.3	0.73
RH-9871	0.1	0.01
RH-2631	3.4	-
RH-2631 conj.	0.68	0.16
RH-0282	2.3	2.8
RH-0126	1.96	0.86
RH-0126 conj.	3.6	-
RH-0875	0.7	0.09
RH-0897	3.5	1.0
RH-0897 conj.	6.5	2.5
RH-120898	-	2.2

The main residue found in the tops and roots was the parent compound. Metabolism via oxidation of the alkyl substituents on both aromatic rings produced metabolites with varied sites and degrees of oxidation. None of the metabolites exceeded 10% of the total residue. No differences were observed between the products with the three radiolabels.

Figure 6. Proposed metabolic pathways of tebufenozide in sugar beet.



Apples. Tebufenozide can be applied to apples at various times during the whole growing season from bloom until shortly before harvest. Metabolism was studied in a field trial simulating early-season and mid-season applications at high rates, with tebufenozide uniformly labelled with ^{14}C in the A-ring. One apple tree was treated twice with a 35-day interval at 1.12 kg ai/ha giving a total of 2.24 kg ai/ha. Foliage was sampled after the first treatment, and fruit and foliage were both sampled before and immediately after, 29 days after, and 68 days after the second treatment when the fruit was ripe at the final harvest (Wu, 1993b).

Fruit and foliage samples were homogenized and the total radioactive residues determined by combustion radioanalysis. The results are given in Table 14.

Table 14. Residues in apple foliage and fruit after application of [^{14}C]tebufenozide (Wu, 1993b).

Sample	^{14}C, mg/kg as tebufenozide	
	Fruit	Foliage
Post-treatment 1	-	106
Pre-treatment 2	1.34	23
Post-treatment 2	5.34	188
29 days after 2	0.32	48
Final harvest (68 days after 2)	0.21	27

The metabolitic profiles of the fruit samples at both 29 and 68 days after the second treatment were determined by analysis of the organic extracts by TLC and HPLC with radiometric detection. Residues left in the aqueous layers (12-13% of the total) were hydrolyzed by treatment with cellulase and determined similarly. The main residue was found to be the parent compound. Four metabolites were isolated and identified, none of which represented more than about 6% of the sample activity. One metabolite, RH-1788, was found in both free and conjugated forms. RH-0282 and RH-2778 were found only as conjugates. The metabolic profiles in the fruit samples are shown in Table 15.

Table 15. Metabolite profiles in apple samples after application of [^{14}C]tebufenozide.

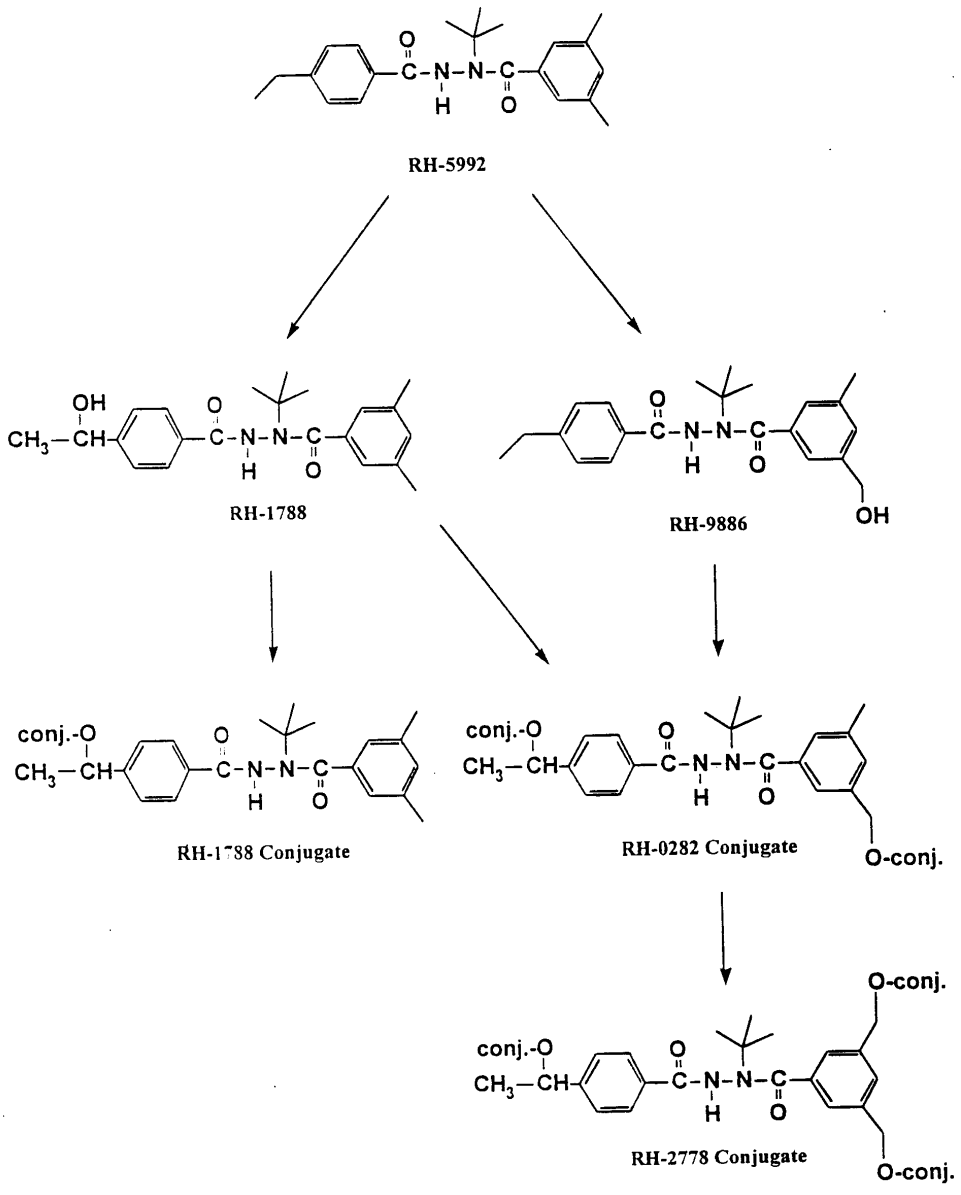
Compound	^{14}C at 29 days		^{14}C at 68 days (harvest)	
	% of total	mg/kg as tebufenozide	% of total	mg/kg as tebuconozide
tebufenozide	71.2	0.22	77.26	0.165
RH-1788	4.96	0.021	2.49	0.008
RH-1788conj.	1.52		1.47	
RH-9886	0.21	0.001	0.20	0.0000
RH-0282 conj.	6.02	0.02	4.32	0.009
RH-2778 conj	2.46	0.008	2.71	0.006
Total	86.37	0.27	88.45	0.188

The metabolic profile in the foliage sample from the final harvest was also determined. The residue was almost entirely parent tebufenozide (>93%). The only metabolite identified was RH-0282, present in both free and conjugated forms at levels of 0.24 and 0.52%, respectively, of the total activity. At least 13 unknowns were also present in the foliage, none acounting for more than 1.88% of the total activity.

The proposed metabolic pathways for tebufenozide in apples are shown in Figure 7.

tebufenozide

Figure 7. Proposed metabolic pathways of tebufenozide in apples.



Note: "conj." marks possible locations of conjugates.

Environmental fate in soil

A study of aerobic degradation was conducted with two soils, a loam and a sandy loam, at a nominal dose rate of 1 mg/kg of [^{14}C]tebufenozide. The soil samples were incubated in the dark at 25ºC for one year, in bottles with traps for CO_2 and volatile organic compounds. The three labels were used (Reynolds, 1992e).

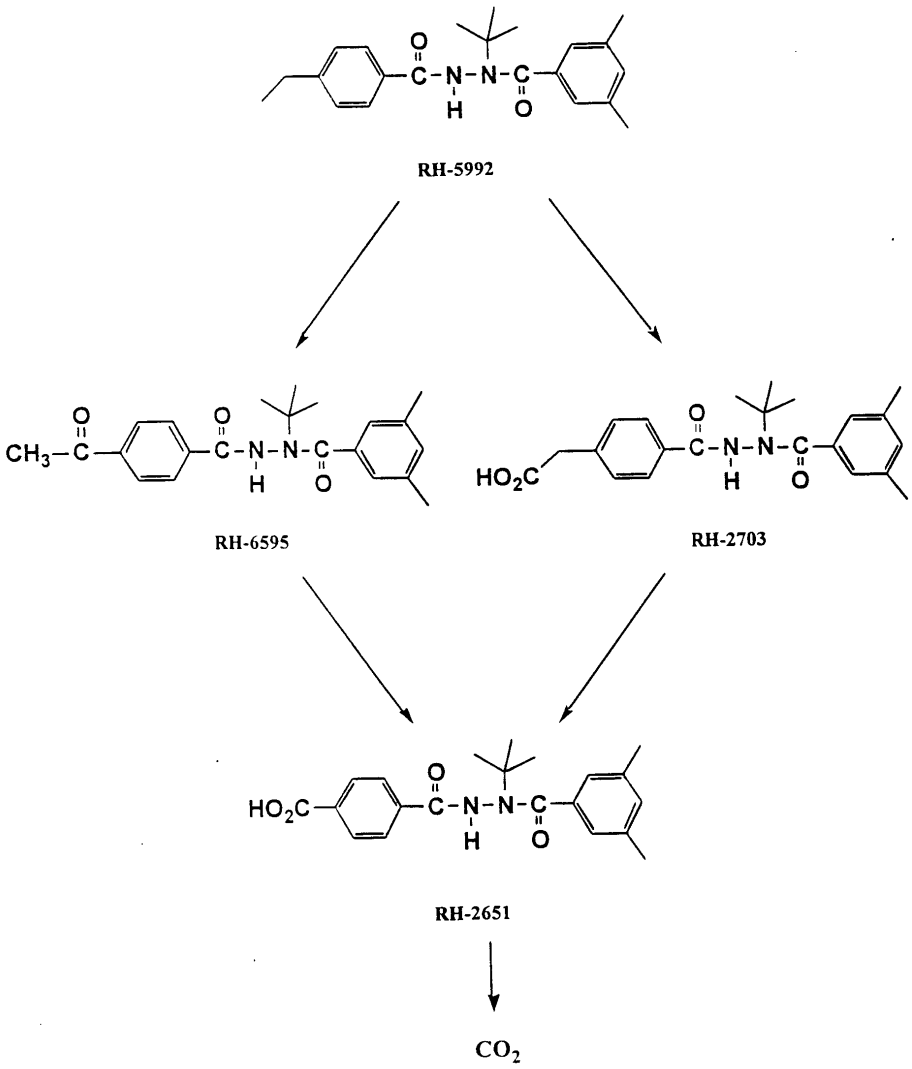
Duplicate samples were taken on days 0, 1, 3, 7, 14, 30, 60, 90, 120, 180, 270, and 365 and analysed by TLC and/or HPLC. The identities of the parent compound and major metabolites were confirmed by using a second chromatographic technique and by mass spectrometry. The average recovery of ^{14}C was >94%.

Three degradation produces were observed in addition to the parent compound and CO_2, the ketone RH-6595 and the carboxylic acids RH-2651 and RH-2703. All three are products of oxidation of the ethyl group on the A-ring of the molecule. Five other products were detected but not identified; none of them accounted for more than 5.5% of the applied activity in any sample.

The nature and amounts of the products from the three different labels were very similar in each soil, but the rates of degradation in the two soils were very different. In the loam soil (California), only 7-9% of the parent compound remained by day 365 and 54-62% of the total applied radioactivity had been converted to $^{14}CO_2$ by the end of the study. The calculated half-life of tebufenozide was 105 days. In the sandy loam soil (New Jersey) the rate of degradation was much slower; 61-71% of the parent compound remained by the end of the study and only 2-5% of the initial tebufenozide was mineralized: its calculated half-life was 704 days. It was suggested that this soil may have been atypical, but there was no explanation for the low rate of degradation.

Soil-bound residues increased gradually from all three labels as the study progressed to 12.3-16.6% in the sandy loam and 22.4-26.9% in the loam. They were characterized by various methods. Mild acidic extraction solubilized 2.6-7.0% of the total applied ^{14}C and acid hydrolysis released about 2.9-7.1%. The remainder was fractionated into humic and fulvic acids and humin, with most of the activity recovered from the humic and fulvic acid fractions. The proposed degradation pathways of tebufenozide in soil are shown in Figure 8. The half-life observed in the New Jersey soil was inconsistent with all other reported information on soil degradation.

Figure 8. Proposed degradation pathways of tebufenozide in soil.



The degradation of tebufenozide was also investigated under aerobic conditions in four German soils, a low organic sand (Speyer 2.1), a high organic sand (Speyer 2.2), a sandy loam (Speyer 2.3) and a loamy sand (SLV). Soil samples dosed with [^{14}C]tebufenozide at a rate of either 0.193 kg ai/ha or 0.257 mg ai/kg of dry soil were incubated at 20 ± 2ºC in the dark for 120 days at 40% maximum water capacity. The degradation of tebufenozide was continuous in all four soils, with half-lives between 27.8 and 31.5 days and disappearance times for 90% of the initial concentration from 92.2 to 104.5 days (Schanne 1995a). The characteristics of the soils are shown below.

	Low org. sand	High org. sand	Sandy loam	Loamy sand
Organic C, %	0.98	2.50	1.11	1.07
Clay,%	3.25	4.25	9.44	10.16
Silt, %	9.46	9.16	27.71	35.23
Sand, %	87.28	86.59	62.81	54.6
Cation exchange capaciy, meq/100g	3.76	10.26	9.47	5.33
Water capacity, g/100 g dry soil	21.0	41.3	31.4	34.7
Microbial biomass, mg/100 g dry	start 12.6	start 21.1	start 19.4	start 12.6
soil	final 7.8	final 31.8	final 10.4	final 22.5
pH	5.5	5.5	1.1	1.0

The DT-50 and DT-90 values calculated for the four soils are shown below.

	DT-50, days	DT-90, days
Low organic sand	31.1	103.2
High organic sand	28.2	93.6
Sandy loam	27.8	92.2
Loamy sand	31.5	104.5

Carbon dioxide and non-extractable residues reached a maximum of 38% and 42.8% of the applied radioactivity respectively, indicating that these are the major terminal residues. At day 120, up to 12% of the applied activity was associated with the humin fraction of the soil organic matter and up to 31% with humic and fulvic acids. The degradation products RH-6595 (ketone), RH-2651 and RH-2703 (carboxylic acids) were again identified.

Photodegradation on soil. Sandy loam soil treated with 10 mg/kg of A-ring-labelled tebufenozide was maintained at 25ºC and irradiated with a xenon arc lamp for 30 days with a 12-hour light/dark cycle. The study was designed to trap any volatile materials and minimize thermal decomposition (Reynolds, 1992d).

Samples were analysed after 0, 3, 7, 21 and 30 days of irradiation. They were extracted with acidified acetonitrile/water, partitioned with methylene chloride and analysed by TLC or HPLC with radiometric detection. The half-life of tebufenozide was calculate to be 98 days under the test conditions.

A total of seven photoproducts were detected, none accounting for more than 5.3% of the applied ^{14}C in any sample. The most abundant was the ketone RH-6595; the B-ring aldehyde RH-0970 was also identified.

Adsorption/desorption was studied in five different soil types, clay, loam, loamy sand, sandy clay loam and loamy sand, with [^{14}C]tebufenozide labelled in the A-ring. The soil pH ranged from 5.6 to 7.8 and the organic matter content from 0.8% to 3.6% (Hawkins, 1992).

Replicate experiments were conducted with each of the five soils at four aqueous concentrations from 0.0516 to 0.755 mg/kg in 0.01 M calcium chloride. In each experiment, the soil and aqueous solution of the test compound were equilibrated for 24 hours, and the phases then separated and radioassayed. The soil was desorbed twice with fresh calcium chloride solution for 24 hours with radioassay after each desorption. All equilibrations were carried out at 25 ± 1ºC.

The average K_{oc} for the soils was 572, 928 and 1168 for adsorption, first desorption and second desorption respectively. On this basis the potential mobility would be classified as low.

A laboratory column leaching study was carried out in accordance with the BBA guideline. Three soils were selected: a sand with low organic matter (0.7%), a sand with high organic matter (2.29%), and a sandy loam with an organic matter content of 1.34% (Knoch, 1993). After sieving, the soils were packed in glass columns of 5 cm diameter and 40 cm length to produce a soil depth of 30 cm. Formulated tebufenozide was added at 37.91 µg of ai to each soil column to simulate the application of 0.192 kg ai/ha. Leaching was by application of an artificial rainfall of 393 ml within 48 hours (simulating a rainfall of 200 mm). The leachate was collected from the first and second 24-hour periods and analysed.

The tebufenozide in the leachate from the sandy soil after 48 h was <2% of that applied. In sandy loam soil the amount of tebufenozide in the first 24 h leachate was <2% of that applied, and over the entire period 0.95 Ì g was recovered in the leachate, 2.5% of the added tebufenozide. In the loamy sand column no significant amounts of tebufenozide were observed in the leachate during the first 24h. By the end of the leaching period the leachate from one of the duplicate columns contained 5.5% of the applied tebufenozide and that from the second column contained none.

A column leaching study was carried out with aged residues in clay loam, sand, sandy loam and loam. Radiolabelled tebufenozide was mixed with the soils at a nominal rate of 1 mg/kg and the mixtures aged for 30 days, yielding the residues shown below (Reynolds, 1992b).

Compound	% of applied ^{14}C			
	Clay loam	Sand	Sandy loam	Loam
Tebufenozide	52.3	71.4	52.7	66.4
RH-6595	4.11	3.05	3.65	2.38
RH-2651	5.98	2.06	15.22	0.18
RH-2703	1.21	4.87	2.62	0.95

Duplicate glass columns were filled to a height of 30 cm with each of the sieved soils. The corresponding soil with the aged residue was applied to the top of each column and the columns were eluted with 0.01 M calcium chloride solution. The columns were divided into 6-cm sections, and the eluates, soil sections and dose plugs were radioassayed. The distribution of the radioactivity is shown in Table 16.

Table 16. Distribution of radioactivity in aged soil columns (Reynolds, 1992).

Sample	% of ^{14}C recovered from column			
	Clay loam	Sand	Sandy loam	Loam
Treated plug	52.0	46.9	60.2	91.8
Segment 1 (0-6 cm)	25.2	3.36	3.85	5.53
Segment 2	11.6	4.81	3.50	1.58
Segment 3	2.44	5.66	3.84	0.22
Segment 4	0.71	6.55	3.5	0.10
Segment 5	0.27	6.96	2.27	0.02
Eluate	7.76	25.8	22.9	0.77

Analysis of the eluates indicated that the two carboxylic acids were mobile in all four soils, and the parent tebufenozide was somewhat mobile in sand.

A similar study was carried out with a sandy soil with a low organic matter content. The soil was treated with [^{14}C]tebufenozide and aged in air for 40 days before leaching. After ageing, 17.5% of the radioactivity was unextractable, 11.3% had been converted to carbon dioxide and 0.3% had been volatilized. The remainder of the extractable applied ^{14}C was associated with tebufenozide (46.7%), RH-6595 (6.6%), RH-2651 (9.1%), RH-2703 (4.6%), and two unknowns present at 1.6% and 5.2 %.

After one day of leaching the highest level of radioactivity in the eluate was 0.3%, and after the second day the average activity was 6.9% of that applied. Analysis of the eluates showed that no tebufenozide or ketone (RH 6595) was present in the leachate, whose activity was due to the presence of two carboxylic acids (Schanne, 1995b).

Field dissipation studies were carried out in the USA, Canada, Japan and Germany to determine the persistence and mobility of tebufenozide and its soil degradation products.

In two trials in California, the test material was applied directly to bare sandy soils with low organic matter at a rate of 1.12 kg ai /ha. Under these conditions, residues of tebufenozide and its products were found only in the top 30 cm of the soil: no downward movement of the compounds was observed. The degradation of tebufenozide at the two sites was similar. The half-lives determined from the exponential decline were 53 and 39 days. The trials demonstrated that even when tebufenozide was applied at the maximum rate and to unprotected soil it did not persist and residue levels wwere below 0.01 mg/kg after one year or less (Hawkins, 1993).

Two similar studies were conducted at two other US locations. The soil at a New York site was a sandy loam at the top, becoming a loam below 15 cm and a silt loam below 60 cm; the soil at a Washington site was a low-organic sand. Each site received four treatments 14 days apart of 0.56 kg ai/ha giving a total of 2.24 kg ai/ha. All treatments were made to bare soil using the 2SC formulation of tebufenozide. Residues of tebufenozide and its degradation products were found only in the top 45 cm. The half-lives determined from the exponential decline were 52 and 31 days at New York and Washington respectively. This study also shows that tebufenozide residues are near or below 0.01 mg/kg after a year or less (Hawkins, 1994).

Field dissipation studies were conducted at two sites in Japan, with cultivated loam soils of high (6.49%) and low (1.45%) contents. At each site tebufenozide was applied three times at one-week intervals at a rate of 0.4 kg ai/ha. Soil from the top 10 cm was sampled on days 0, 7, 14, 30, 55 or 60, and 90 and analysed for tebufenozide and its degredation products. The half-life of tebufenozide was 6 days in the soil with high organic matter and 19 days in that with low organic matter (Yajima, 1992).

Other field dissipation studies were carried out at four different Japanese sites under paddy conditions, with tebufenozide applied at a rate of 0.3 kg ai/ha. Soil from the top 10 cm was sampled according to a regular schedule for a total of 240-365 days. The half-lives in the four soils ranged from 4.2 to 30 days (Chong, 1992).

The dissipation of tebufenozide under field conditions was also investigated at four locations in Germany. The test substance was applied at a rate of 0.192 kg ai/ha to bare soil and samples were collected from 0-10 cm, 10-20 and 20-40 cm depths before and after application, and approximately 1 week, 2 weeks, and 1, 2, 3, and 5 months after application. The soils were a loamy sand, two sandy loams and a sandy silt loam. Soil samples were analysed for residues of tebufenozide and its products, with an LOD for each of the analytes of 0.01 mg/kg. The DT50 and DT90 values for tebufenozide in each soil are shown below (Sochor and Holzwarth, 1995).

	Disappearance times of tebufenozide, days			
	Loamy sand	Sandy loam	Sandy loam	Sandy silt loam
DT 50	108	10	13	5
DT 90	968	112	43	137

Three dissipation studies were conducted in different regions of Canada in orchard soils. The soils at the three sites were characterized as loamy fine sand, silty loam, and very fine sand. In all the trials the test compound was applied four times at a rate of 0.28 kg ai/ha. Samples taken at intervals 0 to 368 days were divided into 0-7.5 cm, 7.5-25 cm, 25-50 cm and 50-60 cm sections for analysis (MacLeod, 1995a,b.c).

The first study, with loamy fine sand, was in a mature orchard of malus trees with a ground cover of bunch-type grass. The concentration of tebufenozide shortly after application was low, the maximum concentration being reached in the top 7.5 cm on day 122, probably owing to test material moving from the vegetation into the soil (MacLeod, 1995a).

The trial with silty loam was adjacent to an apple orchard. The concentration of tebufenozide in the top 7.5 cm after the last application was 0.29 mg/kg and was below the limit of determinatiaon (0.02 mg/kg) in the sample taken 284 days after the last application. The half-life of tebufenozide was 75 days.

The third trail was in an area typical of the fruit growing region. Applications were directly on to the bare soil surface. Residues of tebufenozide on day 0 averaged 1.29 mg/kg in the top 7.5 cm and decreased to 0.12 mg/kg in the sample taken 368 days after the last treatment. The calculated half-life was 135 days.

Environmental fate in water/sediment systems

The solubility of tebufenozide in water was determined by the shake-flask method, using aqueous mixtures of the labelled test compound to provide concentrations of 5, 10, 25 and 50 mg/l if all the tebufenozide dissolved. The concentration after the final equilibrium averaged 0.83 mg/l in the four tubes (Kelly, 1992b).

The hydrolysis of tebufenozide was studied in sterile aqueous solutions buffered to pH 5, 7, and 9, containing 0.5 mg/l of [^{14}C]tebufenozide and less than 1% by volume of acetonitrile, and maintained at 25 ± 1ºC for 30 days in the dark. Aliquots of the solutions were analysed after 0, 3, 7, 14, 21, and 30 days for the test compound and possible hydrolysis products by solid-phase extraction, LSC, TLC and HPLC.

The total recoveries of applied radioactivity averaged 105-109% for the three systems. The only significant radioactive compound in the solutions was confirmed by normal-phase TLC and reverse-phase HPLC to be tebufenozide. No hydrolysis products were detected (Reynolds, 1992a).

The photolysis of tebufenozide, labelled in the A-ring, was studied in a 0.5 mg/l solution in natural pond water maintained at 25 ± 1ºC. The sample tubes were irradiated with a Xenon lamp, with 12-hour light/dark cycles, for 30 days. CO_2 and other volatile materials in the head-spaces of the sample tubes were retained in a series of traps. Duplicate sample tubes were removed from the photolysis chamber and the solutions analysed for the test compound and possible photoproducts at 0, 3, 7, 14, 21 and 30 days.

Analyses were by partition with organic solvent and liquid scintillation counting of the two phases, followed by TLC and HPLC of the organic phase with radiometric detection. No aqueous phase contained more than 5% of the applied radioactivity and no significant amount of volatile material was formed.

Tebufenozide was degraded with a half-life of 67 days under the conditions of the experiment. The major product was the ketone RH-6595, produced at a maximum level of 5.3% of the original ^{14}C. Eight other photoproducts were detected, none exceeding 3.5% of the applied activity in any sample. Almost no degradation occurred in the dark control. Recoveries of the applied radioactivity were 96-106% (Reynolds, 1992c).

In a study of aerobic aquatic degradation two hydorsoils and their respective paddy waters were incubated with tebufenozide labelled at all three sites at a nominal rate of 1 mg/kg in the dark at 25 ± 1ºC for one year. The sediments were Arkansas silty clay and California clay loam (Reynolds, 1992g). The characteristics of the two systems are shown in Table 17.

Table 17. Characteristics of water/sediment systems.

	Arkansas silty clay		California clay loam	
	soil	water	soil	water
Sand, %	12		30	
Silt, %	42		34	
Clay, %	46		36	
Organic matter, %	2.0		2.6	
pH	6.9	6.7	7.8	6.9
O_2 (dissolved, ppm)		8.10		6.85

Duplicate samples were taken for analysis on days 0, 1, 3, 7, 14, 30, 60, 120, 180, 270 and 365. Volatile compounds were trapped throughout the study. At each sampling, the soil and supernatant water were separated by decanting and centrifuging and analysed separately. The radioactive components in each phase were extracted and determined by TLC and/or HPLC, and the identities of the parent compound and the major products were confirmed by mass spectrometry. The distribution of radioactivity and the nature and amounts of the products from the three labels were similar. The results are summarized in Tables 18 and 19.

Table 18. Average distribution of radioactivity at various intervals in Arkansas and California soils.

Days incubation	% of ^{14}C							
	Arkansas				California			
	Supernatant	Solids	PES[1]	Traps	Supernatant	Solids	PES[1]	Traps

Days incubation	% of ^{14}C							
	Arkansas				California			
	Supernatant	Solids	PES[1]	Traps	Supernatant	Solids	PES[1]	Traps
0	55.8	43.4	0.74	0	53.4	45.9	0.69	0
1	45.2	48.8	0.5	0.02	39.7	63.0	2.4	0.01
3	36.4	54.6	3.18	0.04	40.6	60.4	3.7	0.01
7	26.0	64.6	2.1	0.05	22.4	75.1	5.9	0.02
14	17.5	75.1	5.8	0.05	19.8	78.4	7.0	0.03
30	16	74.7	7.5	0.27	12.7	74.8	15.3	0.06
59-60	21.3	57.4	15.2	1.2	13.0	79.7	12.9	0.4
88-90	23.9	64.5	8.2	2.9	15.9	78.3	9.3	1.9
120	24.4	59.4	10	5.2	19.1	65.0	14.9	6.9
179	21.4	51.2	13.7	10.5	18.4	48.4	20.3	18.6
270	31.1	29.9	15.4	20	11.7	23	24	34.2
360	29.2	24.5	20.7	30.1	5.56	11.5	22.7	47.4

[1]Post-extraction solids

Three major compounds were observed in the study in addition to tebufenozide and CO_2. These were the ketone RH-6595 and the two carboxylic acids RH-2651 and 2703, all products of oxidation of the ethyl group on the A-ring of molecule. Five additional degradation products were also found and characterized by their chromatographic behaviour but not identified. None of these accounted for more than 5.7% of the applied activity in any sample. Up to 47% of the total applied ^{14}C was converted to $^{14}CO_2$ by the end of the study.

Sediment-bound residues increased gradually as the study progressed to 19-23% of the ^{14}C from all three labels in both hydrosoils. The bound residues after 358-366 days were characterized by means of various techniques. Mild acidic extraction solubilized 5-6% of the total applied ^{14}C and acid hydrolysis released about 4%. The remainder was fractionated into humic and fulvic acids and humin, with the activity predominantly in the fulvic acid fraction.

The half-lives of the parent compound under the aerobic aquatic test conditions (average of the three labels) were 101 days in the California clay loam and 99 days in the Arkansas silty clay.

Table 19. Distribution of residues in the aquatic degradation of tebufenozide.

Day	Percentage of ^{14}C present as				
	Tebufenozide	RH-6595	RH-2651	RH-2703	CO_2
Arkansas silty clay, A-ring label					
0	98.29	0	0	0	0
1	91.70	0.13	0	0	0.02
3	89.53	0.92	0	0	0.04

Day	Percentage of ^{14}C present as				
	Tebufenozide	RH-6595	RH-2651	RH-2703	CO_2
7	88.11	2.26	0	0	0.05
14	90.01	0	0	0.07	0.05
30	76.77	5.52	0	0	0.27
59	60.17	2.14	2.63	8.71	1.23
90	64.31	4.88	4.16	8.78	2.94
120	49.05	6.92	9.49	9.43	5.19
178	30.78	6.56	14.26	11.14	10.53
269	12.71	2.56	32.07	2.59	20.02
366	7.02	1.07	35.45	0	30.08
California clay loam, B-ring label					
0	98.48	0	0	0	0
1	96.92	0.29	0	0	0
3	94.92	0.70	0	0	0.01
7	95.67	0.32	0	0	0.01
14	91.46	0.42	0	0	0.02
30	86.63	0	0	0	0.05
60	80.80	5.10	0	0	0.35
88	64.99	8.10	2.57	4.74	2.04
120	58.18	4.99	2.86	4.62	6.09
179	31.11	4.26	8.46	4.66	18.08
270	15.17	2.50	6.40	1.11	35.78
358	7.94	0.78	1.88	0.05	47.00

A companion study of anaerobic aquatic degradation with a flooded silt loam hydrosoil, was again in the dark at 25 ± 1ºC for one year at a nominal rate of 1 mg/kg of tebufenozide, uniformly labelled with ^{14}C in the A-ring and also with ^{13}C in the two methyl groups on the B-ring to aid mass spectral identification if needed. Duplicate samples were taken for analysis on days 0, 3, 7, 14, 30, 60, 90, 120, 179, 270 and 365. Volatile products were trapped throughout the study (Reynolds, 1992f).

Two significant compounds were found apart from tebufenozide and CO_2: the ketone, which reached a maximum of 8.0% of the applied activity at day 179, and the carboxylic acid RH-2651 which reached 11.6% at day 120. Eight other compounds were found and characterized. One was the carboxylic acid RH-2703, the others were not identified. None of these minor products exceeded 4% of the applied activity in any sample. The calculated half-life of tebufenozide was 179 days.

METHODS OF RESIDUE ANALYSIS

Analytical Methods

A number of methods have been developed to analyse specifically for tebufenozide in various crops and processed fractions. They depend on GLC or HPLC and have been validated.

Fruits. An HPLC method was used to determine tebufenozide residues in apples, grapes, kiwifruit, apple juice and wine. Apples are extracted by blending with 0.1 N HCl/methanol (1:9). The extract is partially purified by partitioning first with hexane, which is discarded, then with methylene chloride. The methylene chloride layer is concentrated and further purified by column chromatography on basic alumina or Florisil. The residue is determined by HPLC with UV detection. The limit of determination (LOD) in apples was 0.02 mg/kg, with an average recovery of 81.3 ± 11.5% (Deakyne *et al.*, 1994b). The LODs were 0.01 mg/kg in grapes and 0.005 mg/kg in wine with recoveries of 87 ± 11.3% and 81 ± 12.4% respectively (Deakyne *et al.*, 1994a). The average recovery from kiwifuit was 108 ± 5.7%.

In a revised version of this method (Deakyne *et al*., 1995) the identity of the residue was confirmed by HPLC-MS after the same extraction and purification procedure. This revised method was validated for kiwifruit with a limit of detection of 0.01 mg/kg and was used to analyse kiwifruit from the 1994-5 residues trials in New Zealand (Deakyne *et al.*, 1995).

Schuld and Holzwarth (1994a) described a method for the determination of tebufenozide in apples, grapes, apple juice and wine by GLC. Samples (25 g) of apples and grapes are extracted with acetone and the acetone removed in a rotary evaporator. The remaining aqueous phase is extracted with n-hexane and the extract cleaned up by silica gel chromatography. Juice and wine (20 g) are concentrated on an Extrelut® column which is eluted with n-hexane. Tebufenozide is methylated with methyl iodide and the derivative extracted from the reaction solution with n-hexane. The *N*-methyl derivative is determined by gas chromatography with a nitrogen-phosphorus detector (NPD). The lower level of the practical working range for the method is 0.02 mg/kg for apples and grapes and 0.01 mg/kg for juice and wine. Recoveries ranged from 72% to 128% for grapes, 74% to 129% for apples, 77% to 109% for wine, and 63 to 159% for apple juice with mean recoveries of ≥80% for all samples.

The method was modified to complete the determination by GS-MS rather than GLC with an NPD. The limit of determination was 0.02 mg/kg.

In another GLC method (Mellet, 1993a) samples of grapes, must and wine are again extracted with acetone and, after evaporation of the solvent, extracted from the remaining aqueous phase with hexane. This extract is partitioned with acetonitrile and the residues are further purified on a silica gel column. Tebufenozide is then methylated with methyl iodide in the presence of sodium hydride and dimethyl sulfoxide. The *N*-methyl derivative is again determined by gas chromatography with an NPD. The limit of determination was 0.01 mg/kg in all three types of sample, with recoveries from grapes, must, and wine of 71-128%, 66-130%, and 54.5-116% respectively.

In a method for kiwifruit (skin and edible pulp) described by Tillman (1995d) residues are extracted with acetone and cleaned up as in Mellet (1993a), but tebufenozide is determined (without derivsatizaion) by HPLC with an isocratic mobile phase and UV detection at 220 nm. The method was validated for kiwifruit with an average recovery of 96.9% at 0.2 mg/kg.

Nuts. In a method described by Cui and Desai (1994a) residues of tebufenozide are Soxhlet-extracted from pecans with methanol. Sodium chloride solution is added to the extract and a hexane partition removes the oils. The residues are then extracted into methylene chloride. The solvent is evaporated and the residue cleaned up on a basic alumina column. Solid-phase extraction on carbon provides an optional additional clean-up. Tebufenozide is determined by HPLC with UV detection at 240 nm. Mass spectrometry can be used for confirmation. The average recovery was 81.9 ± 10.2%, with a reported limit of determination of 0.01 mg/kg.

A method for the analysis of walnuts described by Cui *et al.* (1993a) is similar to that for pecans, with further clean-up. The organic phase is concentrated to dryness and cleaned up on three columns, the first of carbon, the second a C-18 phase, and the third basic alumina. The final eluate is dried and dissolved in 30% acetonitrile/water for gradient HPLC (mobile phase A: 10% methanol in water; mobile phase B: acetonitrile) on an Adsorbosphere C-18 5µ column with a UV detector. The limit of determination was 0.01 mg/kg and recoveries from fortified samples averaged 91.1 ± 11.8%. A Supelco LC-DP 5µ column is used for confirmation.

This method was revised, first to incorporate the LC-DP confirmatory method with a new solvent system (Cui *et al.,* 1994) and subsequently to include an additional confirmatory method using HPLC-MS (Cui *et al.,* 1995).

Vegetable crops (cabbage, lettuce, spinach, broccoli, celery and mustard greens). Residues can be determined according to Chen *et al.* (1993). Samples are blended with 0.1N HCl/methanol (1:9). The extract is partitioned with methylene chloride and the concentrated methylene chloride layer is cleaned up chromatographically. Extracts of lettuce, cabbage, mustard greens and spinach are cleaned up on a single basic alumina column. Broccoli and celery extracts are passed through three small solid-phase extraction (SPE) tubes which contain successively carbon, basic alumina, and cyano adsorbent. Tebufenozide is determined by HPLC with UV detection. The limit of determination (LOD) was 0.01 mg/kg for all vegetables except celery which had a LOD of 0.05 mg/kg.

This method also was revised to add the options of a basic alumina chromatography clean-up step and HPLC-MS confirmation method (Chen *et al.,* 1994a).

Ishii and Higuchi (1993) described an HPLC method to analyse Chinese kale. Samples are extracted with acetone, which is evaporated. The residue is taken up in dichloromethane and cleaned up by Florisil and alumina column chromatography. The reported detection limit was 0.01 mg/kg, with an average recovery of 95.2%.

Chilli peppers were analysed by the method described above with an additional Florisil column before the alumina column chromatography (Ishii and Higuchi, 1994). The reported detection limit was 0.01 mg/kg and the average recovery was 96.6%.

Rice (grain and straw). A method for residues of tebufenozide and two metabolites in rice grain was described by Komatsu and Yabusaki (1992a). Samples (10g) are first soaked in water for two hours before extraction with acetone. A portion of the acetone extract is concentrated to 10 ml before being dissolved in water and cleaned up on a column of granular diatomite adsorbent. The fraction containing tebufenozide, the ketone RH-6595 and the corresponding alcohol RH-1788 is dried under nitrogen, partitioned with hexane and acetonitrile, and cleaned up further by silica gel chromatography. RH-6595 is reduced to RH-1788 with sodium borohydride. After the reduction step the combined residues are methylated with methyl iodide and sodium hydride in a mixture of benzene and dimethyl sulfoxide. The *N*-methyl-tebufenozide and the *N*-methyl-*O*-methyl-RH-1788 are determined by GLC with an NPD. The limit of detection of all three compounds was 0.005 mg/kg. The average recoveries of tebufenozide, RH-1788 and RH-6595 were 72%, 96% and 96% respectively. Residues of RH-1788 and RH-6595 would of course in practice be reported as one combined value (quantified as RH-1788).

The method for rice straw (Komatsu and Yabusaki, 1992b) is virtually identical to that used for the grain except for the sample size: 5 g of straw is extracted. The limit of detection of all three compounds in straw was 0.04 mg/kg, with average recoveries of tebufenozide, RH-1788 and RH-6595 of 78%, 82%, and 74% respectively.

Tea and brewed tea. An HPLC method for analysing dry and brewed tea was described by Ishii (1995). Dry samples are extracted with water/acetone, filtered, partitioned with dichloromethane, and the extract transferred to acetone. The acetone solution is cleaned up by coagulation and filtration through Celite, then extrated with dichloromethane before clean-up on Florisil and alumina columns. The limit of detection was 0.05 mg/kg and the average recovery 86.7%. Brewed tea is cleaned up by precipitation with zinc acetate and extraction with dichloromethane, followed by Florisil and alumina column chromatography. The residue is again determined by HPLC. The limit of detection was 0.01 mg/kg and the average recovery 91.6%.

A GLC method to analyse tea and brewed tea for tebufenozide, RH-6595 and RH-1788 described by Komatsu and Yabusaki (1993) is similar to the same authors' method for rice. Dry tea is extracted with acetone and cleaned up by chromatography on a porous kieselguhr column, coagulation, and chromatography on silica gel. RH-6595 is reduced to RH-1788 with sodium borohydride, tebufenozide and RH-1788 are methylated with methyl iodide, and the methylated compounds are partitioned with hexane. After clean-up by silica gel column chromatography, the residues are determined by GLC with an NPD. The limit of detection was 0.01 mg/kg for both tebufenozide and RH-1788 and the average recoveries of tebufenozide, RH-1788, and RH-6595 were 95%, 86%, and 100% respectively. Filtered brewed tea is partitioned with dichloromethane and cleaned up on a silica gel column. The analysis is completed by reduction, methylation, clean-up and GLC as before. The limit of detection was again 0.01 mg/kg for both analytes, with average recoveries of 90%, 74%, and 72% for tebufenozide, RH-1788, and RH-6595 respectively.

Soil. Tebufenozide and its degradation products, the ketone RH-6595 and the carboxylic acids RH-2703 and 2651, are easily extractable from soil with methanol/0.5N HCl (3:1). This extract is partitioned twice with dichloromethane. After concentration, the acids are esterified with diazomethane and the mixture is cleaned up on a Florisil column. Tebufenozide and its metabolites are eluted with ethyl acetate/hexane/acetone. The eluant is evaporated and the residue reconstitued in methanol/water for analysis by HPLC. Recoveries at fortification levels from 0.02 to 0.1 mg/kg were 85-87% for tebufenozide and above 90% for its metabolites (MacLeod, 1995a).

Water. Tebufenozide residues are extracted from water with methylene chloride after the addition of sodium chloride. The methylene chloride is evaporated and the residue dissolved in the mobile phase for determination by HPLC. An extra clean-up by silica gel chromatography may be necessary for stream waters. The mean recovery from all types of water was 98.4 ± 8.8% at fortification levels of 0.1 to 5 ì g/l. The reported LOD was 0.1 ì g/l (Deakyne *et al*., 1992).

Stability of residues in stored analytical samples

The stability of tebufenozide in stored analytical solutions was investigated in the USA (Chen *et al.*, 1994e). Standard solutions of tebufenozide at concentrations from 0.01 to 1.0 mg/l in either methanol/water or acetonitrile/water were stored for various periods and compared with freshly made standards by HPLC injection and integration of the peaks. Statistical analysis of the results indicated that working standards of tebufenozide were stable for a minimum of 6 months.

The stability of tebufenozide residues was studied in samples of apples, apple juice, grapes, wine, rice, walnuts and lettuce.

Apples and apple juice. A study of the stability of residues of tebufenozide in apple samples during frozen storage is in progress. Untreated apple samples were homgenized, and control and fortified samples were stored at -15 ± 1ºC. The results reported so far (Deakyne and Chen, 1995a) show that tebufenozide is stable for at least six months in apples when stored frozen. The percentage of the residue remaining aftere six months storage ranged from 90.8% to 108% with no indication of a systematic decrease with time.

Additional information on the stability of tebufenozide residues in apples during frozen storage (-15ºC) was obtained by analysing field-treated samples, storing them for two additional years and re-analysing the same samples (Deakyne, 1995b; Burnett *et al*., 1994). The results are shown below.

Sample	First analysis		Second analysis	
	Storage period, days	mg/kg	Storage period, days	mg/kg
2	199	0.44	924	0.53
			924	0.42
4	190	0.5	915	0.5
			915	0.4
8	173	0.67	917	0.61
			922	0.52
6	156	0.79	876	1

In another study carried out in Japan, untreated apple samples were fortified with a solution of tebufenozide and its metabolites RH-6595 and RH-1788 at a level of 1 mg/kg. Samples were stored at -20ºC for 190-202 days. The average recoveries after storage were 94% for tebufenozide, 87% for RH-1788 and 69% for RH-6595 (Komatsu and Yabusaki, 1994).

The stability of residues of tebufenozide in apple juice during frozen storage was investigated by Deakyne and Chen (1995b). Samples of control apple juice (20 g) were fortified with 20 ì g of tebufenozide (1 mg/kg) and stored frozen. Samples were removed from storage at intervals and analysed together with control samples stored under the same conditions and freshly fortified. After 6 months storage the mean recovery was 86.7%

Grapes and wine. The stability of tebufenozide in frozen grapes and wine stored for periods up to 12 months was investigated by Schuld and Holzwarth (1994b). Samples were fortified with tebufenozide at 0.2 mg/kg (grapes) or 0.1 mg/kg (wine) and stored at ≤-18ºC. Samples were removed for analysis after approximately 0, 1, 3, 6, 9, and 12 months. There was no loss during the 12 months.

	% recovery of tebufenozide after period, months					
	0	1	3	6	9	12
Grapes		91	82.5	88	84	89
Wine	97	93	118	74	92	94.5

Walnuts. Walnut kernels were fortified with 1 mg/kg of tebufenozide and stored frozen at -10ºC (Cui and Deakyne, 1994). Controls, freshly fortified controls, and stored samples were analysed after 0, 1, 2, and 3 months storage. No decrease was observed during this period and the study was continued for a total of 18 months. The final residue was 76.6% of the initial level, showing that residues of tebufenozide are adequately stable for at least 18 months of frozen storage.

Lettuce. Control samples of lettuce were homogenized and sub-samples of 20 g each were fortified with 20 ì g of tebufenozide in methanol. After evaporation of the methanol the samples were sealed and stored frozen. Samples were removed from storage and analysed at intervals (Chen *et al.,* 1994b). After 128 days the mean recovery was 90%.

Rice. Samples of milled rice spiked with tebufenozide at 2 mg/kg were stored at -20ºC for 20-21 days until analysis. Recoveries ranged from 53 to 80% (Komatsu andYabusaki, 1992c).

The Meeting was informed that the studies on apples, walnuts and lettuce would be continued for a full 3 years of storage.

Residue definition

Tebufenozide was the predominant residue found in all the plant metabolism studies, which included apples, grapes, sugar beet and rice. Although metatbolism occurs in plants no single metabolite in any plant metabolism study exceeded 10% of the total residue.

In some residue trials samples were analysed for residues of the metabolites RH-6595 and RH-1788. The levels of these were very low, and in most cases below the limit of determination. Some samples in supervised trials on vegetables in the USA (1991-93) were analysed for the terminal metabolite RH-0897 (Chen *et al.,* 1994c,d). All residues were below the LOD.

The Meeting concluded that the residue should be defined as tebufenozide. The residue is fat-soluble.

USE PATTERN

Tebufenozide is an insectside which acts as an agonist of the critically important insect-moulting hormone, 20-hydroxyecdysone. Treatment with tebufenozide results in an accelerated, incomplete moult and an inmediate cessation of feeding in lepidopteran larvae.

Tebufenozide is used to control caterpillar pests in fruits, vegetables and other crops. The registered uses are shown in Table 20, and proposed uses in Table 21.

Table 20. Registered uses of tebufenozide.

Crop	Country	Form.	Application			PHI, days
			No.	Rate, kg ai/ha	Spray concn., kg ai/hl	
Apples	Canada	2SC	1-4	0.12-0.24		14
Apples	France	2SC	3	0.144	0.0144	21
Apples	Japan	20SC	2	0.4	0.0133	45
Apples	Korea	20SC	3	0.6		30
Apples	Switzerland	2SC	2-3	0.144-0.24	0.012	21
Cabbage species	Switzerland	2SC	1-2	0.096	0.0096	14
Chinese Kale	Thailand	20SC	3-5	0.1875	0.0333	14
Cotton	Colombia	2SC		0.036-0.060		
Grapes	France	2SC	3	0.144	0.0144	21
Grapes	Italy	2SC	1-3	0.144	0.0144	30
Grapes	Portugal	2SC	2-3	0.144	0.0144	Pending
Grapes	Spain	2SC	1-4	0.12-0.144	0.012-0.0144	21
Grapes	Switzerland	2SC	3-4	0.072-0.230	0.012-0.014	
Grapes	Thailand	20SC	2-3	0.1	0.00533	14

Crop	Country	Form.	Application			PHI, days
			No.	Rate, kg ai/ha	Spray concn., kg ai/hl	
Grapes	Turkey	2SC	1-3	0.096	0.0096	21
Kiwifruit	New Zealand	70WP	2-4	>0.12	0.006	21
Legumes	Vietnam	20SC	2-4	0.1		
Onions	Vietnam	20SC	2-4	0.1		
Onions, Welsh	Taiwan	20SC	3	0.1		9
Ornamental & Forestry Trees	Italy	2SC	2-3		0.0048-0.0096	
Peppers, Chilli	Indonesia	10WP	2-3	0.05-0.10		15
Peppers, Chilli	Indonesia	2SC	2-3	0.05-0.10		15
Pome fruit	Belgium	2SC	3	0.12	0.012	28
Pome fruit	Chile	2SC	1-4	0.24-0.36	0.0096	28
Pome fruit	Italy	2SC	1-4	0.216-0.288	0.0144-0.0192	14
Pome fruit	New Zealand	70WP	4-9	>0.18	0.006	14
Pome fruit	Portugal	2SC	2-3	0.144	0.0144	Pending
Rice	Colombia	2SC		0.036-0.060		
Rice	Japan	0.75DL	2	0.3		14
Rice	Japan	10WP	2	0.1-0.25		21
Rice	Korea	0.1G	3	0.3-0.5		3
Rice	Korea	8WP	6	0.08-0.12		3
Rice	Malasya	20SC	2	0.1		21
Rice	Philippines	20SC	1-2	0.1		14
Rice	Thailand	20SC	1-2	0.075	0.015	14
Shallots	Indonesia	20SC	4-6	0.075-0.15		15
Shallots	Indonesia	10WP	4-6	0.075-0.15		15
Soya beans	Indenesia	20SC	1-2	0.05-0.10		15
Soya beans	Indonesia	10WP	1-2	0.05-0.10		15
Sugar beet	Japan	20SC	2-4	0.1	0.01	14
Tea	Japan	20SC	2	0.4	0.02	14
Tea	Sri Lanka	20SC	1-2	0.13-0.15		7
Walnuts	France	2SC	3	0.144	0.0288	30
Walnuts	USA	2SC	4	0.28	0.0265	30

Table 21. Anticipated registered uses of tebufenozide.

Country	Crop	Form.	Application			PHI, days
			No	Rate, kg ai/ha	Spray conc., kg ai/hl	
Australia	Pome fruit	70WP, 2SC	4-8	0.18	0.006	28
	Grapes	70WP, 2SC	1-3	0.06	0.006	28
Austria	Pome fruit	2SC	2-3	0.09-0.18	0.012	14
	Grapes	2SC	3-4	0.072-0.192	0.012	28
Cyprus	Pome fruit	2SC	2	0.12	0.012	14
	Citrus fruit	2SC	1-2	0.12-0.24	0.012-0.024	14
France	Pears	2SC	3	0.144	0.0144	21
Germany	Pome fruit	2SC	2-3	0.09-0.18	0.012	14
	Grapes	2SC	3-4	0.072-0.192	0.012	28
Greece	Pome fruit	2SC	4	0.288	0.0144	14
	Grapes	2SC	4	0.144	0.0144	21
Slovenia	Apples	2SC	1-3	0.216	0.0144	21
	Grapes	2SC	1-3	0.12	0.012	21

tebufenozide CLICK HERE to continue

Country	Crop	Form.	Application			PHI, days
			No	Rate, kg ai/ha	Spray conc., kg ai/hl	
Spain	Pome fruit	2SC	1-3	0.15-0.18	0.012-0.014	21
	Rice	240LV	2-3	0.12		21
Switzerland	Lettuce spinach	2SC	1-2	0.15-0.18	0.0096	14
USA	Pome fruit	2SC, 70WP	6	0.35		14
	Grapes	2SC		not for registration	filed import tolerance	
	Kiwifruit	70WP	(2-4)	(0.134)	(>0.12)	90
	Pecans	2SC	5	0.28	0.0347	14
	Cole crops	2SC, 70WP	7	0.067-0.135		7
	Leafy vegetables	2SC, 70WP	7	0.067-0.135		7
	Cotton	2SC	4	0.28		14

RESIDUES RESULTING FROM SUPERVISED TRIALS

Trials were carried out in Australia, Canada, Europe, Japan, New Zealand, the USA, and other countries. All trials in Germany and the USA were conducted according to Good Laboratory Practice (GLP). Some trials were designed to determine efficacious use rates and the effects of the timing of applications as well to assess the residues in the crop at various pre-harvest intervals. The field parts of these trials were not conducted according to GLP.

The results were not corrected for analytical recoveries unless noted. Analytical recoveries were mostly high (>80%), so using uncorrected or corrected results should not significantly influence the interpretations.

Pome Fruits

Apples. Eight trials were conducted from November 1992 to March 1993 in Australia (Rouch, 1994) as a randomized block of four duplicates. Four different application rates were used and eight applications were made at 2-week intervals. The spray concentration used were 1.6-6.4 times the proposed GAP rate. The residue values in Table 21 are the averages of samples from duplicate plots.

Six small-plot field trials (one tree per plot) were conducted from November 1994 to March 1995 in apples following season-long applications of tebufenozide 70WP and 200 SC in Australia (Armstrong, 1995b) as a randomized block of three replicates. Treatments commenced just at petal fall and continued every two weeks until commercial harvest. A motorized hand sprayer was used. The spray concentrations were 0.012 kg ai/hl and 0.024 kg ai/hl. All above-ground parts of each tree were sprayed to near the point of run-off each time. Fruit samples were collected at various PHIs.

These trials were at higher rates than the proposed GAP (4-8 applications at 0.18 kg ai/ha, 0.006 kg ai/hl, 28 days PHI). The average residue was 1.3 mg/kg on day 28.

A number of trials were conducted in Canada. In 1993 (Bruns, 1994) three to five applications were made either to three trees per plot or to single tree plots replicated three times in a randomized complete block design. The trees were sprayed to the point of runoff using a handgun. In six trials according to GAP in 1995 (Bruns, 1995) four applications were made at 0.24 kg ai/ha each and the spray intervals ranged from 2 weeks to 3 months. Samples were analysed by a modified HPLC method (Bruns, 1994). The limit of determination was 0.02 mg/kg and the average recovery 77%.

In other apple trials in Canada in 1994 (Tillman, 1995a) plots consisted of either four trees or single tree plots replicated three times in a randomized block. The trees were sprayed to runoff with a handgun. Samples were analysed by HPLC according to GLP.

Residues of tebufenozide in the trials according to Canadian GAP ranged from 0.06 to 1.1 mg/kg.

A series of efficacy trials was carried out in Chile to assess the bioactivity of tebufenozide in controlling the major pome fruit pest, codling moth (Gonzalez, 1995). In one trial samples were analysed for tebufenozide to establish the dissipation rate.

In a series of field trials with several different apple varieties in geographically representative apple orchards in France (Tillman, 1994) five to seven applications were usually made at intervals of about 2

weeks. The analyses were according to GLP but the field parts of the trials were not. In the four trials according to GAP the average residue was 0.1 mg/kg (PHI 18-21 days).

In GLP trials in Germany in 1992 in Germany according to proposed GAP (Raquet *et al.*, 1993) three applications at 0.122-0.202 kg ai/ha (0.013 kg ai/hl) were made with a motorized knapsack sprayer. The apples were analysed by a validated method (Schuld and Holzworth, 1994a).

In three other GLP trials in Germany in 1993 (Brusche and Holzwarth, 1995) tebufenozide was applied three times at the maximum proposed rate using a knapsack mist blower to 7-14 trees per plot.

The residues in whole fruit in the German trials according to proposed GAP ranged from 0.02 to 0.35 mg/kg.

A single trial in 1993 in Greece (AgrEvo, 1995) was not according to actual or proposed GAP. Eight applications were made to a single tree at 2-week intervals. The residues in treated samples were 0.4-0.66 mg/kg and in control samples <0.02 mg/kg.

Three of six trials (Tillman, 1994) in Italy in 1992 complied with GAP. Three applications were made at intervals of about 30 days to 6 trees per plot. Apple samples were analysed by GLC with an NPD. Residues in the trials according to GAP ranged from 0.28 to 0.55 mg/kg.

Two trials were conducted in Japan according to GAP: 2 applications, 10 days apart, at 0.400 kg ai/ha (Komatsu and Yabusaki, 1994). The dates of the applications were varied in order to harvest the mature samples with different PHIs. Samples were analysed for the parent compound, RH-1788 and RH-6595. RH-6595 was reduced to RH-1788 and both it and tebufenozide were methylated and analysed by GLC (Schuld and Holzwarth, 1994a). The residues of tebufenozide were 0.02 and 0.05 mg/kg. Residues of RH-1788 and RH-6595 were all below the LOD of 0.01 mg/kg.

Six residue trials during the 1992/1993 growing season with Gala apples (Tillman, 1995c) and ten during the 1994/95 growing season (Tillman, 1995b) in New Zealand were designed to assess the residues on apples from different use rates at different frequencies and timings. Applications were made by applying tebufenozide 70W to run-off with a hand-held lance. The application rates changed after the first three applications owing to the higher spray volumes. Typical volumes are 2500 to 3000 l/ha, increasing as the foliage becomes denser toward harvest.

The trials were with single trees with five replicates. All apple samples were analysed by HPLC (Deakyne *et al.*, 1994b). The LOD of the method was 0.03 mg/kg and recoveries ranged from 68 to 85% in 1992/1993 and from 77 to 120% in 1994/1995.

Two trials were conducted in Spain according to proposed GAP (Tillman, 1994). Two trees in each plot were sprayed three times with an atomizer spray. The samples were analysed in France by the method of Mellet (1993a). The residues were 0.37 and 0.54 mg/kg.

A series of field trials on apples in a number of States of the USA from 1991 to 1993 (Burnett *et al.*, 1994) were under GLP but not according to proposed GAP. Most trials were with 9 applications of 0.336 kg ai/ha and a few with 10 applications of 0.280 kg ai/ha. The plot sizes ranged from 0.54 to 6.19 ha with a minimum of 4 and a maximum of 37 trees in a plot. Samples were analysed by HPLC. The number of applications was greater than that recommended in the proposed GAP.

Four trials according to the proposed GAP (6 applications at 0.336 kg ai/ha) in 1994 at two locations in the USA (Dong, 1995d) were designed to compare the 2SC and 70WP formulations. The plot sizes ranged from 2.56 to 2.79 ha. Samples were analysed by HPLC. Residues from proposed GAP ranged from 0.36 to 0.6 mg/kg. The residues from the two formulations were similar.

The results of the trials are shown in Table 22.

Table 22. Residues of tebufenozide in apples. The underlined residues are from treatments according to GAP.

Country, location, year	Form.	Application			PHI, days	Residue, mg/kg	Reference, comments
		No.	kg ai/ha	kg ai/hl			
Australia	SC	8		0.010	7	1.1[1]	Rouch, 1994
1992-1993					14	1.2[1]	
					27	1.2[1]	
Australia	SC	8		0.014	7	2.2[1]	Rouch, 1994
1992-1993					14	1.2[1]	
					27	1.9[1]	
Australia	SC	8		0.019	7	2.7[1]	Rouch, 1994
1992-1993					14	2.4[1]	
					27	2.9[1]	
Australia	SC	8		0.038	7	5.2[1]	Rouch, 1994
1992-1993					14	5.5[1]	
					27	5.7[1]	
Australia	WP	8	0.24	0.012	0	1.4	Armstrong, 1995b
1994-1995					7	1.6	
					14	1.3	
					21	0.9	
					28	0.6	
Australia	WP	8	0.48	0.024	0	2.7	Armstrong, 1995b
1994-1995					7	4.3	
					14	2.3	
					21	2.0	
					28	2.4	
Australia	SC	8	0.24	0.012	0	2.5	Armstrong, 1995b
1994-1995					7	2.1	
					14	1.4	
					21	1.5	
					28	1.0	
Australia	SC	8	0.48	0.024	0	2.5	Armstrong, 1995b
1994-1995					7	3.3	
					14	2.3	
					21	2.0	
					28	2.0	

Country, location, year	Form.	Application			PHI, days	Residue, mg/kg	Reference, comments
		No.	kg ai/ha	kg ai/hl			
Australia	WP	8	0.24-0.36	0.012	0	1.02	Armstrong, 1995b
1994-1995					7	0.97	
					14	0.85	
					28	0.43	
Australia	WP	8	0.48-0.72	0.024	0	1.6	Armstrong, 1995b
1994-1995					7	2.7	
					14	2.2	
					28	1.4	
Canada	SC	3	0.24	0.010	0	0.24	Bruns, 1994
1993					7	0.15	
					14	0.12	
					21	0.09	
Canada	SC	5	0.24	0.010	0	0.23	Bruns, 1994
1993					7	0.17	
					14	0.19	
					21	0.19	
Canada	SC	3	0.12	0.005	0	0.07	Bruns, 1994
1993					7	0.07	
					14	0.06	
					21	<0.05	
Canada 1993	SC	4	0.24	0.010	50	0.13	Bruns, 1994
Canada 1993	SC	3	0.12	0.022	63	<0.05	Bruns, 1994
Canada 1993	SC	3	0.24	0.045	63	0.15	Bruns, 1994
Canada 1994	SC	4	0.107	0.008	14	0.03	Tillman, 1995a
Canada 1994	SC	3	0.16	0.012	14	0.14	Tillman, 1995a
Canada 1994	SC	3	0.115	0.012	14	0.17	Tillman, 1995a
Canada 1994	SC	4	0.077	0.008	14	0.06	Tillman, 1995a
Canada 1994	SC	3	0.107	0.008	50	0.03	Tillman, 1995a
Canada 1994	SC	2	0.16	0.012	50	<0.02	Tillman, 1995a
Canada 1994	SC	4	0.12-0.24	0.005-0.010	49	<0.02	Tillman, 1995a
Canada 1994	SC	3	0.24	0.010	88	<0.028	Tillman, 1995a
Canada 1994	SC	5	0.24	0.010	14	0.11	Tillman, 1995a
Canada 1994	SC	4	0.24	0.081	14	0.077	Tillman, 1995a
Canada 1994	SC	4	0.24	0.010	14	0.16	Tillman, 1995a
Canada 1994	SC	4	0.24	0.027	14	0.27	Tillman, 1995a
Canada 1995	SC	4	0.24	0.036	14	0.75	Bruns, 1995
Canada 1995	SC	4	0.24	0.036	14	0.84	Bruns 1995
Canada 1995	SC	4	0.24	0.036	14	1.1	Bruns, 1995
Canada 1995	SC	4	0.24	0.021	14	0.37	Bruns, 1995
Canada 1995	SC	5	0.24	0.021	14	0.43	Bruns, 1995
Canada 1995	SC	5	0.24	0.021	14	0.52	Bruns, 1995
Chile	SC	1	0.144	0.007	0	0.45	Gonzalez, 1995

Country, location, year	Form.	Application			PHI, days	Residue, mg/kg	Reference, comments
		No.	kg ai/ha	kg ai/hl			
1995					8	0.26	LOD 0.02 mg/kg
					14	0.2	
					20	0.19	
					28	0.2	
					35	0.15	
France	SC	7	0.10	0.010	16	0.14	Tillman, 1994
Agde, 1990	SC	7	0.15	0.015	16	0.09	control 0.04 mg/kg
France	SC	7	0.134	0.019	18	0.88	Tillman, 1994
Grisolles, 1991	SC	7	0.10	0.014	18	0.35	control 0.15
France	SC	7	0.144	0.036	42	0.11	Tillman, 1994
Bouldon, 1991	SC	7	0.192	0.046	42	0.11	control 0.07
France	SC	7	0.144	0.036	42	0.13	Tillman, 1994
Barbentane, 1991	SC	7	0.192	0.046	42	0.4	control <0.05
France	SC	1	0.144	0.014	0	0.13	Tillman, 1994
Meynes, 1991	SC	1	0.144	0.014	3	0.16	control <0.05
	SC	1	0.144	0.014	7	0.12	
	SC	1	0.144	0.014	14	0.13	
	SC	1	0.144	0.014	21	0.07	
France	SC	7	0.075	0.008	45	0.08	Tillman, 1994
Mauguio, 1991	SC	7	0.075	0.008	45	<0.05	control <0.05
France	SC	7	0.15	0.015	45	0.33	Tillman, 1994
Mauguio, 1991	SC	7	0.15	0.015	45	0.26	control <0.05
France	SC	5	0.12	0.012	21	0.14	Tillman, 1994
Garonne, 1992	SC	5	0.144	0.014	21	0.18	control <0.05
France	SC	7	0.084	0.012	21	0.06	Tillman, 1994
Pompignac, 1992	SC	7	0.10	0.014	21	0.09	control <0.01
France	SC	1	0.144	0.014	0	0.06	Tillman, 1994
St. Pardon, 1992	SC	1	0.144	0.048	3	0.04	control <0.01
	SC	1	0.144	0.048	7	0.04	
	SC	1	0.144	0.048	14	0.02	
	SC	1	0.144	0.048	21	0.01	
	SC	1	0.144	0.048	30	0.04	
France	SC	5	0.036	0.012	23	0.18	Tillman, 1994
Barbentane, 1992	SC	5	0.043	0.014	23	0.21	control <0.01
France	SC	5	0.042	0.012	21	0.08	Tillman, 1994
Boulbon, 1992	SC	5	0.05	0.014	21	0.15	control <0.01
Germany	SC	3	0.19	0.013	0	0.29	Raquet, 1993
Drage-Elbstorf,1992					7	0.24	
					14	0.24	
					28	0.16	(Belgian GAP)
Germany	SC	3	0.16	0.013	0	0.05	Raquet, 1993

Country, location, year	Form.	Application			PHI, days	Residue, mg/kg	Reference, comments
		No.	kg ai/ha	kg ai/hl			
Bornheim, 1992					7	0.03	
					14	0.02	
					28	<0.02	(Belgian GAP)
Germany	SC	3	0.20	0.013	0	0.39	Raquet, 1993
Bodenegg, 1992					7	0.28	
					14	0.35	
					28	0.20	(Belgian GAP)
Germany	SC	3	0.122	0.013	0	0.27	Raquet, 1993
Hoechst, 1992					7	0.2	
					14	0.11	
					28	0.08	
Germany	SC	1	0.122	0.013			Raquet, 1993
Niederdorfelden, 1992	SC	2	0.13	0.013	0	0.13	
					7	0.15	
					14	0.06	
					28	0.11	
Germany	SC	3	0.18	0.012	12	0.15	Brusche, 1995
Drage-Elbe, 1993					25	0.16	
Germany	SC	3	0.18	0.012	13	0.15	Brusche, 1995
Drage-Elbe, 1993					27	0.09	
Germany	SC	3	0.18	0.012	12	0.21	Brusche, 1995
Kippenhausen, 1993					27	0.23	
Greece	SC	8	0.288	0.014	10	0.66	AgrEvo, 1995
1993					20	0.4	LOD 0.02 mg/kg
Italy	SC	3	0.144	0.014	0	0.33	Tillman, 1994
Baricella BO, 1992					7	0.29	
					14	0.22	
Italy	SC	3	0.288	0.029	0	0.51	Tillman, 1994
Baricella BO, 1992					7	0.18	
					14	0.28	
Italy	SC	3	0.144	0.014	14	0.25	Tillman, 1994
Gallo FE, 1992	SC	3	0.288	0.029	14	0.55	
Italy	SC	3	0.144	0.014	14	0.29	Tillman, 1994
Baricella BO, 1992	SC	3	0.288	0.029	14	0.52	
Japan	FL	2	0.40	0.013	45	0.02	Komatsu, 1994
Nagano, 1993					60	0.02	
					90	<0.01	
Japan	FL	2	0.40	0.013	45	0.05	Komatsu, 1994
Iwate, 1993					60	0.08	
					90	<0.01	
New Zealand	WP	9	3x0.18-6x0.24	0.012	7	0.53	Tillman, 1995c
Hastings, 1992/93					14	0.48	

Country, location, year	Form.	Application			PHI, days	Residue, mg/kg	Reference, comments
		No.	kg ai/ha	kg ai/hl			
					21	0.20	
					28	0.49	
					35	0.31	
					42	0.36	
New Zealand	WP	9	3x0.09-6x0.12	0.006	7	0.24	Tillman, 1995c
Hastings, 1992/93					14	0.15	
					21	0.32	
					28	0.09	
					35	0.14	
					42	0.09	
New Zealand	WP	6	3x0.18-3x0.24	0.012	7	0.30	Tillman, 1995c
Hastings, 1992/93					14	0.19	
					21	0.23	
					28	0.28	
					35	0.27	
					42	0.24	
New Zealand	WP	6	3x0.09-3x0.12	0.006	7	0.13	Tillman, 1995c
Hastings, 1992/93	WP				14	0.10	
					21	0.09	
					28	0.06	
					35	0.06	
					42	0.08	
New Zealand	WP	2	0.18	0.012	11	0.32	Tillman, 1995c
Hastings, 1992/93					11	0.18	
New Zealand	WP	2	0.09	0.006	27	0.09	Tillman, 1995c
Hastings, 1992/93					27	0.1	
New Zealand	WP	8	0.096-0.15	0.006	7	0.09	Tillman, 1995b
Hawke's Bay, 1994/95					14	0.08	
					21	0.08	
					28	0.07	
					35	0.08	
					42	0.15	
New Zealand	WP	8	0.19-0.30	0.012	7	0.23	Tillman, 1995b
Hawke's Bay, 1994/95					14	0.18	
					21	0.14	
					28	0.15	
					35	0.18	
					42	0.13	
New Zealand	WP	4	0.096-0.15	0.012	7	0.25	Tillman, 1995b
Hawke's Bay, 1994/95					14	0.23	
					21	0.25	
					28	0.24	

Country, location, year	Form.	Application			PHI, days	Residue, mg/kg	Reference, comments
		No.	kg ai/ha	kg ai/hl			
					35	0.24	
					42	0.15	
New Zealand	WP	4	0.19-0.30	0.012	7	0.49	Tillman, 1995b
Hawke's Bay, 1994/95					14	0.45	
					21	0.34	
					28	0.44	
					35	0.38	
					42	0.33	
New Zealand Hawke's Bay, 1994/95	WP	2	0.19-0.13	0.006	140	<0.03	Tillman, 1995b
New Zealand Hawke's Bay, 1994/95	WP	2	0.19-0.25	0.012	140	<0.03	Tillman, 1995b
New Zealand	WP	10	0.15-0.18	0.012	0	0.13	Tillman, 1995b
Nelson, 1994/95					7	0.2	
					14	0.11	
					21	0.12	
					28	0.22	
New Zealand	WP	10	0.30-0.36	0.012	0	0.32	Tillman, 1995b
Nelson, 1994/1995					7	0.27	
					14	0.27	
					21	0.23	
					28	0.25	
New Zealand	WP	11	0.12-0.144	0.006	1	0.32	Tillman, 1995b
Hawke's Bay, 1994/95					8	0.30	
					15	0.24	
					22	0.26	
					29	0.12	
New Zealand	WP	11	0.24-0.29	0.012	1	0.47	Tillman, 1995b
Hawke's Bay, 1994/95					8	0.38	
					15	0.42	
					22	0.42	
					29	0.25	
Spain Epila (Z), 1992	SC	3	0.18	0.014	22	0.54	Tillman, 1994
Spain Epila (Z), 1992	SC	3	0.15	0.012	22	0.37	Tillman, 1994
USA	SC	10	0.28	0.023-0.024	0	0.47	Burnett, 1994
NY, 1991					9	0.43	
					23	0.29	
USA	SC	10	0.28	0.054	0	0.68	Burnett, 1994
PA, 1991					7	0.57	
					29	0.6	
USA WA, 1991	SC	10	0.28	0.030	11	1	Burnett, 1994
USA WA, 1992	SC	9	0.336	0.036	0	1.1	Burnett, 1994

Country, location, year	Form.	Application			PHI, days	Residue, mg/kg	Reference, comments
		No.	kg ai/ha	kg ai/hl			
					14	0.77	
USA NC, 1992	SC	9	0.336	0.057-0.080	16	1.31	Burnett, 1994
USA CA, 1992	SC	9	0.336	0.033-0.036	0	0.66	Burnett, 1994
					14	0.46	
					28	0.62	
USA OH, 1992	SC	9	0.336	0.039-0.043	15	0.32	Burnett, 1994
USA OR, 1992	SC	9	0.336	0.036	0	1.2	Burnett, 1994
					14	1.1	
USA VA, 1992	SC	9	0.336	0.043-0.054	0	0.6	Burnett, 1994
					15	0.47	
USA MI, 1992	SC	9	0.336	0.072-0.075	14	1.07	Burnett, 1994
USA WV, 1992	SC	9	0.336	0.039	15	0.46	Burnett, 1994
USA	SC	9	0.336	0.036	0	0.82	Burnett, 1994
WA, 1993					7	0.68	
					14	0.86	
					27	0.5	
USA	SC	9	0.336	0.040-0.043	0	1.1	Burnett, 1994
MI, 1993					7	1.06	
					14	0.84	
					28	0.82	
USA	SC	6	0.336	0.036	7	0.77	Dong, 1995d
WA, 1994					14	0.61	
USA	WP	6	0.336	0.036	7	0.58	Dong, 1995d
WA, 1994					14	0.46	
USA	SC	6	0.336	0.072	7	0.57	Dong, 1995d
PA, 1994					14	0.42	
USA	WP	6	0.336	0.072	7	0.57	Dong, 1995d
PA, 1994					14	0.36	
USA PA, 1991	SC	10	0.28	0.054	7	0.57	Deakyne, 1994c for processing
USA PA, 1994	WP	6	0.336	0.072	7	0.56	Deakyne, 1995a average of 2 analyses for processing

[1] Mean of two plots

Pears. In a trial on pears in Australia (Amstrong, 1995) a single tree per plot was used with three replicates. Treatments were with a motorized hand sprayer and each tree was sprayed near to the point of run-off. The application rates were higher than proposed GAP. Samples were analysed by GC-MS.

Two trials were carried out in Italy in 1992 with 6 trees per plot. Three applications were made at intervals of 30 days. One trial complied with GAP and showed a residue of 0.23 mg/kg.

Several trials in New Zealand were designed to assess the decline of residues at different

application rates. Single trees were treated, with four replicates. Samples were analysed by HPLC and the reported LOD was 0.03 mg/kg. The residues from treatments closest to GAP ranged from 0.09 to 0.29 mg/kg (Tillman, 1995b).

The results are shown in Table 23.

Table 23. Residues of tebufenozide in pears. Underlined residues are from trials according to GAP.

Country, location, year	Form.	No.	Application		PHI, days	Residue, mg/kg	Reference
			kg ai/ha	kg ai/hl			
Australia	WP	8	0.24	0.012	0	1.3	Armstrong, 1995c
1994-1995					7	1.2	
					14	1.0	
					21	1.0	
					28	1.4	
Australia	WP	8	0.48	0.024	0	2.1	Armstrong, 1995c
1994-1995					7	2.0	
					14	2.0	
					21	2.2	
					28	1.7	
Australia	SC	8	0.24	0.012	0	1.9	Armstrong, 1995c
1994-1995					7	2.0	
					14	1.8	
					21	1.4	
					28	1.5	
Australia	SC	8	0.48	0.024	0	2.4	Armstrong, 1995c
1994-1995					7	2.8	
					14	1.5	
					21	1.8	
					28	2.7	
Australia	WP	7	0.24	0.012	0	0.9	Armstrong, 1995c
1994-1995					7	0.7	
					14	0.7	
					28	0.3	
Australia	WP	7	0.48	0.024	0	1.5	Armstrong, 1995c
1994-1995					7	1.0	
					14	1.0	
					28	0.7	
Italy	SC	3	0.216	0.014	14	<u>0.23</u>	Mellet, 1994
1992	SC	3	0.432	0.029	14	0.58	
New Zealand	WP	8	0.048-0.054	0.003	1	0.15	Tillman, 1995b
Hawke's Bay, 1994/95					7	0.09	
					14	0.06	
					21	0.10	
New Zealand	WP	8	0.096-0.108	0.006	1	0.17	Tillman, 1995b
Hawkes Bay, 1994/95					7	0.14	
					14	<u>0.1</u>	
					21	0.09	

Country, location, year	Form.	No.	Application		PHI, days	Residue, mg/kg	Reference
			kg ai/ha	kg ai/hl			
New Zealand	WP	8	0.192-0.216	0.012	1	0.04	Tillman, 1995b
Hawke's Bay, 1994/95					7	0.21	
					14	0.29	
					21	0.21	
New Zealand	WP	10	0.057-0.062	0.003	0	0.09	Tillman, 1995b
Nelson, 1994/1995					7	0.10	
					14	0.08	
					21	0.09	
					28	<0.03	
New Zealand	WP	10	0.114-0.123	0.012	0	0.15	Tillman, 1995b
Nelson, 1994/1995					7	0.14	
					14	0.16	
					21	0.28	
					28	0.1	
New Zealand	WP	10	0.228-0.246	0.012	0	0.4	Tillman, 1995b
Nelson, 1994/1995					7	0.34	
					14	0.29	
					21	0.18	
					28	0.24	
New Zealand	WP	7	0.09	0.003	0	0.08	Tillman, 1995b
Nelson, 1994/1995					7	0.07	
					14	0.04	
					21	0.05	
					28	0.1	
New Zealand	WP	7	0.180	0.006	0	0.16	Tillman, 1995b
Nelson, 1994/1995					7	0.09	
					14	0.09	
					21	0.07	
					28	0.07	
New Zealand	WP	7	0.360	0.012	0	0.23	Tillman, 1995b
Nelson, 1994/1995					7	0.37	
					14	0.16	
					21	0.25	
					28	0.24	

Grapes. Several grape trials were carried out in Australia during the years 1992 to 1995, but in all of them the application rates or the number of applications or both were higher than those specified in proposed GAP.

The trials in 1992-1993 were with high volume sprays to the point of run-off. The reported residues are the averages of samples from duplicate trials at the same rates (Anderson, 1993). The 1994-1995 trials were also with high-volume sprays applied to the foliage (Armstrong, 1995).

In a large number of trials with red and white grapes at different locations in France between 1990 and 1992 (Gocha, 1995a) the plot sizes ranged from 11 m^2 to 48 m^2 with 5 to 22 vines in each plot. Most trials were with 4 replicates while some others were single or in duplicate. One or two applications were

made. Samples were harvested at intervals of 0 to 89 days after the (last) application. Only a few of these trials were according to GAP. The original GLC method used in 1990 measured tebufenozide residues with an LOD of 0.04 mg/kg. The extraction and methylation steps were slightly modified for the 1991 analyses to reach an LOD of 0.025 mg/kg. In 1992, using different chromatographic and methylation conditions, the laboratory was able to reach an LOD of 0.01 mg/kg in both solid and liquid substrates. This method was validated (Mellet, 1993a). Grape samples from two trials were processed to wine or spirit.

Three trials in France in 1992 for processing studies (DeWilde *et al.*, 1995a) were conducted with treated and untreated plots without replicates. The plot sizes were chosen according to the quantity of sample to be harvested but were not less than 1 ha. Two trials in 1993 in France for processing studies (DeWilde *et al.*, 1995b) complied with GAP. The plot sizes were not less than 1.4 ha. Applications were made with a motorized knapsack sprayer. Samples were harvested when the grapes reached the maturity required for commercial wine production.

The residues in the trials which accorded with French GAP ranged from 0.05 to 0.28 mg/kg.

Five trials in Germany were according to proposed GAP (Ulrich *et al.*, 1994). The plot sizes were not less than 0.58 ha. The applications were carried out with a motorized knapsack sprayer. At the last sampling about 200 kg of grapes were taken from at least 30 vines from two trials for processing to must and wine. Samples were analysed by GLC. Four similar trials, again according to proposed GAP, were carried out by Kaiser (1994).

Residues in the grapes from the German trials at the proposed 28-day PHI ranged from 0.21 to 0.5 mg/kg.

Four trials at two different locations in Italy (Kaiser and Holzwarth, 1994) were at excessive rates and the grapes were vinified. The limit of determination was 0.02 mg/kg for grapes, and 0.01 mg/kg for must, new wine and potable wine.

Two trials in Thailand (Ishii and Higuchi, 1995) were not according to GAP or GLP.

The results of the supervised trials on grapes are shown in Table 24.

Table 24. Residues of tebufenozide in grapes. Underlined residues are from treatments according to GAP.

Country, year	Form.	Application			PHI, days	Residue, mg/kg	Ref., comments
		No.	kg ai/ha	kg ai/hl			
Australia	SC	5	0.044-0.12	0.006	0	1.3[1]	Anderson, 1993
1992-1993					7	1.0[1]	
					14	0.82[1]	
					28	0.57[1]	
Australia	SC	5	0.129-0.192	0.010	0	1.9[1]	Anderson, 1993
1992-1993					7	1.5[1]	
					14	1.2[1]	
					28	0.87[1]	

Country, year	Form.	Application			PHI, days	Residue, mg/kg	Ref., comments
		No.	kg ai/ha	kg ai/hl			
Australia	WP	5	0.036	0.006	0	1.0	Armstrong, 1995a
1994-1995					7	0.98	
					14	0.7	
					28	0.32	
Australia	WP	5	0.058	0.010	0	1.6	Armstrong, 1995a
1994-1995					7	2.0	
					14	1.7	
					28	1.12	
Australia	SC	5	0.036	0.006	0	1.1	Armstrong, 1995a
1994-1995					7	1.9	
					14	0.9	
					28	0.5	
Australia	SC	5	0.058	0.010	0	1.2	Armstrong, 1995a
1994-1995					7	0.63	
					14	0.9	
					28	0.42	
Australia	WP	5	0.09-0.15	0.006	0	0.61	Armstrong, 1995a
1994-1995					7	1.4	
					14	1.3	
					28	0.71	
Australia	WP	5	0.144-0.24	0.010	0	1.6	Armstrong, 1995a
1994-1995					7	2.0	
					14	1.2	
					28	1.8	
Australia	SC	5	0.09-0.15	0.006	0	1.4	Armstrong, 1995a
1994-1995					7	1.1	
					14	1.2	
					28	1.1	
Australia	SC	5	0.144-0.24	0.010	0	1.96	Armstrong, 1995a
1994-1995					7	2.4	
					14	1.3	
					28	1.67	
France	SC	1	0.10	0.10	65	0.08	Gocha, 1995a
1990	SC	2	0.05	0.05	53	0.13	LOD 0.04 mg/kg
France	SC	1	0.10	0.10	89	0.09	Gocha, 1995a
1990	SC	2	0.05	0.05	76	<0.04	

Country, year	Form.	Application			PHI, days	Residue, mg/kg	Ref., comments
		No.	kg ai/ha	kg ai/hl			
France	SC	1	0.10	0.10	78	0.29	Gocha, 1995a
1990	SC	2	0.050	0.050	64	0.21	
France	SC	1	0.10		71	0.05	Gocha, 1995a
1990	SC	1	0.150		71	0.09	
France	SC	2	0.15	0.05	29	0.43	Gocha, 1995a
1990	SC	2	0.20	0.07	29	0.25	
	SC	2	0.15	0.05	22	0.56	
France	SC	2	0.15	0.04	18	0.19	Gocha, 1995a
1990	SC	2	0.20	0.05	18	0.12	
	SC	2	0.15	0.04	12	0.16	
France	SC	1	0.15	0.05	74	<0.04	Gocha, 1995a
1990	SC	1	0.15	0.05	81	<0.04	
	SC	1	0.20	0.06	81	<0.04	
France	SC	1	0.10	0.022	77	0.05	Gocha, 1995a
1990	SC	1	0.15	0.033	77	0.07	
France	SC	1	0.10	0.10	48	0.17	Gocha, 1995a
1990	SC	1	0.20	0.20	48	0.20	
France 1990	SC	1	0.096	0.042	56	<0.04	Gocha, 1995a
France 1990	SC	1	0.096	0.046	56	<0.04	Gocha, 1995a
France 1990	SC	1	0.096	0.064	77	0.06	Gocha, 1995a
France 1990	SC	1	0.096	0.064	38	0.08	Gocha, 1995a
France	SC	2	0.100	0.100	0	0.33	Gocha, 1995a
1990					7	0.23	
					14	0.26	
					29	0.43	
					58	0.24	
France	SC	2	0.100	0.050	0	0.29	Gocha, 1995a
1990					7	0.53	
					14	0.49	
					30	0.21	
					45	0.19	
					60	0.18	
France	SC	1	0.20	0.10	80	0.19	Gocha, 1995a
1990	SC	1	0.40	0.20	80	0.32	LOD 0.025 mg/kg
France	SC	1	0.144	0.021	66	0.05	
1991	SC	1	0.12	0.018	59	0.06	
	SC	1	0.144	0.021	59	0.07	

Country, year	Form.	Application			PHI, days	Residue, mg/kg	Ref., comments
		No.	kg ai/ha	kg ai/hl			
France	SC	1	0.144	0.048	0	0.14	Gocha, 1995a
1991	SC	1	0.144	0.048	2	0.13	
	SC	1	0.144	0.048	7	0.05	
	SC	1	0.144	0.048	14	0.05	
	SC	1	0.144	0.048	21	0.05	
France	SC	1	0.144	0.041	58	0.03	Gocha, 1995a
1991	SC	1	0.12	0.034	50	0.05	
	SC	1	0.144	0.041	50	0.03	
France	SC	1	0.10	0.067	78	<0.025	Gocha, 1995a
1991	SC	1	0.10	0.067	78	0.72	
	SC	1	0.125	0.083	78	0.04	
	SC	1	0.125	0.083	78	0.07	
France	SC	1	0.144	0.032	72	0.14	Gocha, 1995a
1991	SC	1	0.12	0.027	61	0.13	
	SC	1	0.144	0.032	61	0.17	
France	SC	1	0.12	0.040	66	0.09	Gocha, 1995a
1991	SC	1	0.144	0.048	66	0.11	
France	SC	1	0.144	0.048	20	0.24	Gocha, 1995a
1992	SC	1	0.144	0.048	29	0.13	LOD 0.01 mg/kg
	SC	1	0.144	0.048	39	0.44	
France	SC	1	0.144	0.048	21	0.12	Gocha, 1995a
1992	SC	1	0.144	0.048	30	0.07	
	SC	1	0.144	0.048	56	0.08	
France	SC	1	0.144	0.044	21	0.06	Gocha, 1995a
1992	SC	1	0.144	0.044	30	0.04	
	SC	1	0.144	0.044	61	0.14	
France	SC	1	0.144	0.048	0	0.72	Gocha, 1995a
1992	SC	2	0.144	0.048	3	0.81	
					7	0.9	
	SC	2	0.144	0.048	14	0.29	
					21	0.07	
France 1992	SC	2	0.144	0.048	21	0.28	Gocha, 1995a
1992							
France	SC	2	0.144	0.048	14	0.68	Gocha, 1995a
1992						0.81	
France 1992	SC	2	0.144	0.048	7	1.07	Gocha, 1995a
France	SC	2	0.144	0.048	3	3.7	Gocha, 1995a

Country, year	Form.	Application			PHI, days	Residue, mg/kg	Ref., comments
		No.	kg ai/ha	kg ai/hl			
1992						2.7	
France 1992	SC	2	0.144	0.048	0	4.2	Gocha, 1995a
France 1992	SC	1	0.144	0.048	65	0.04	Gocha, 1995a
France 1992	SC	2	0.144	0.048	30	0.05	Gocha, 1995a
1992							
France	SC	2	0.144	0.048	21	0.26	Gocha, 1995a
1992							
France	SC	2	0.144	0.144	50	0.17	Gocha, 1995a
1992							
France	SC	3	0.144	0.096	40	0.18	De Wilde, 1995a
1992							average of 2 analyses
France	SC	3	0.144	0.048	41	0.06	DeWilde, 1995a
1992							average of 2 analyses
France	SC	3	0.144	0.048	25	0.08	DeWilde, 1995a
1992							average of 2 analyses
France	SC	3	0.144	0.048	19	0.18	DeWilde, 1995b
1993							average of 2 analyses
France 1993	SC	3	0.144	0.048	21	0.28	DeWilde, 1995b
Germany	SC	4	0.079-0.206	0.013-0.040	0	0.59	Ulrich, 1994
1992					7	0.48	LOD 0.02 mg/kg
					14	1.1	
					28	0.28	
Germany	SC	4	0.074-0.196	0.013-0.040	0	0.37	Ulrich, 1994
1992					7	0.20	LOD 0.02 mg/kg
					14	0.44	
					28	0.5	
Germany	SC	4	0.075-0.210	0.0130.040	0	0.5	Ulrich, 1994
1992					7	0.28	LOD 0.02 mg/kg
					14	0.79	
					28	0.24	
Germany	SC	4	0.074-0.204	0.013-0.040	0	0.33	Ulrich, 1994
1992					7	0.53	LOD 0.02 mg/kg
					14	0.47	
					28	0.27	
Germany	SC	4	0.075-0.207	0.013-0.040	0	0 .54	Ulrich, 1994
1992					7	0.57	
					14	0.4	

Country, year	Form.	Application			PHI, days	Residue, mg/kg	Ref., comments
		No.	kg ai/ha	kg ai/hl			
					28	0.40	
Germany	SC	4	0.070-0.200	0.012-0.036	0	0.52	Kaiser, 1994a
1993					7	0.95	LOD 0.02 mg/kg
					14	0.16	
					28	0.21	
Germany	SC	4	0.076-0.197	0.012-0.036	0	0.33	Kaiser, 1994a
1993					7	0.50	LOD 0.02 mg/kg
					14	0.15	
					28	0.26	
Germany	SC	4	0.070-0.193	0.012-0.036	0	0.46	Kaiser, 1994a
1993					7	0.42	LOD 0.02 mg/kg
					14	0.64	
					28	0.22	
Germany	SC	4	0.072-0.196	0.012-0.036	0	0.46	Kaiser, 1994a
1993					7	0.32	LOD 0.02 mg/kg
					14	0.28	
					28	0.42	
Italy	SC	2	0.238-0.240	0.012	0	0.5	Kaiser, 1994b
1992					21	0.56	
Italy	SC	2	0.48	0.024	0	0.8	Kaiser, 1994b
1992					21	0.86	
Italy	SC	2	0.216-0.240	0.012	0	0.37	Kaiser, 1994b
1992					21	0.26	
Italy	SC	2	0.48	0.024	0	0.72	Kaiser, 1994b
1992					21	0.84	
Thailand	SC	8	0.10	0.005	0	0.91	Ishii, 1995a
1994					7	0.52	LOD ± 0.01 mg/kg
					14	0.15	
					21	0.18	
					35	0.14	
Thailand	SC	8	0.10	0.005	0	0.53	Ishii, 1995a
1994					7	0.20	LOD 0.01 mg/kg
					14	0.10	
					21	0.18	

[1] Mean of duplicate trials

Kiwifruit. A large number of residue trials in New Zealand between 1990 and 1995 (Tillman, 1995d) were

all with single-vine plots with 5 replicates per treatment in a randomized block design. All applications were made with 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 being equivalent to 2000 l/ha. The rate of 0.12 kg ai/ha per application was calculated from the reported spray concentration of 0.006 kg ai/hl and the total spray volume.

The 1990-93 trials were conducted to determine the efficacious use rates, to study the effects of the timing of the applications and to assess the residues in the crop at various pre-harvest intervals. The whole fruit samples were analysed by HPLC with HPLC-MS for confirmation (Deakyne *et al.*, 1995). All results were corrected for the analytical recovery, which ranged from 88.8 to 96.6%.

The 1994-95 trials were with three alternative use patterns: (a) 4 applications at pre-bloom, 75-95% petal fall, first cover (21-day interval) and second cover (21-day interval); (b) 7 applications, four as (a) followed by three further applications at 21-day intervals; (c) 2 applications at pre-bloom and 75-95% petal fall. Whole kiwifruit were analysed as before.

In two trials in the USA in 1995 (Deakyne, 1996) four applications of the 70WP formulation were made at 0.15 or 0.30 kg ai/ha, giving total treatments of 0.60 or 1.20 kg ai/ha. The airblast applications were made at intervals of 6 to 14 days. The plot sizes were 0.50-0.54 ha.

Single samples were taken 90 days after the last applications. Whole fruit samples were analysed by the method of Deakyne *et al.* (1995). The residues in the treated samples were all less than 0.5 mg/kg, even from the higher rate.

The results are shown in Table 25.

Table 25. Residues of tebufenozide in kiwifruit.

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, 1995d
1990-1991					21	0.86	
					52	0.21	
					115	0.22	
New Zealand	SC	4	a	0.012	10	1.69	Tillman, 1995d
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, 1995d
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, 1995d
1992					7	0.82	

Country, location, year	Form.	Application			PHI, days	Residue, mg/kg	Reference
		No.	kg ai/ha	kg ai/hl			
					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, 1995d
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, 1995d
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, 1995d
1992					42	0.16	
					72	0.06	
					98	0.05	
					127	0.08	
					147	0.08	
New Zealand	WP	3	b	0.012	21	0.37	Tillman, 1995d
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, 1995d
1993					14	0.6	
					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, 1995d
1993					14	1.4	
					21	2.1	
					28	1.8	
					35	1.3	
					42	1.5	
New Zealand	WP	4	c	0.003	1	0.28	Tillman, 1995d
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, 1995d

Country, location, year	Form.	Application			PHI, days	Residue, mg/kg	Reference
		No.	kg ai/ha	kg ai/hl			
1994-1995					7	0.82	
					14	0.40	
					28	0.31	
					107	0.08	
New Zealand	WP	4	c	0.012	1	1.1	Tillman, 1995d
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, 1995d
1995					7	0.42	
					14	0.33	
					28	0.29	
New Zealand 1995	WP	7	c	0.012	31	0.62	Tillman, 1995d
New Zealand 1994	WP	2	c	0.006	154	0.05	Tillman, 1995d
New Zealand	WP	4	c	0.003	0	0.46	Tillman, 1995d
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, 1995d
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, 1995d
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, 1995d
1995					8	0.82	
					14	0.84	
					28	0.58	
New Zealand	WP	7	c	0.012	28	1.3	Tillman, 1995d
1995							
New Zealand 1994	WP	2	c	0.006	163	0.04	Tillman, 1995d
USA	WP	4	0.150	0.010-0.011	90	0.15	Deakyne, 1996
CA, 1995	WP	4	0.300	0.020-0.022	90	0.49	
USA	WP	4	0.150	0.010-0.011	90	0.09	Deakyne, 1996
CA, 1995	WP	4	0.300	0.019-0.021	90	0.19	

a Each vine received approximately 5-7 l of spray at each application, which was sprayed to the point of run-off
b Each vine received approximately 5 l of spray at each application
c Each vine received approximately 6 l of spray at each application

Vegetable Crops

Numerous field trials were conducted at various locations throughout the USA from 1991 to 1993 (Chen *et al.*, 1994c,d; Dong, 1995b,c). Ground applications were either by foliar spray or directly to the soil at intervals of 5 to 10 days. Single samples at each PHI were analysed. The proposed use pattern for leafy vegetables and cole crops in the USA is 7 applications at 0.067-0.135 kg ai/ha with a 7-day PHI.

Celery. In trials by Chen *et al.* (1994d) plot sizes were 0.15-0.74 ha. In two trials samples of both stalk and stalk with foliage were analysed for comparison, while in the others only stalk samples were analysed. Analyses were by HPLC with an LOD of 0.05 mg/kg. The interval from sampling to analysis (SAI) ranged from 150 to 525 days.

Dong (1995c) compared the 2SC and 70WP formulations in the USA in 1994-5 with 7 to 9 applications at a rate of 0.140 kg ai/ha. The plot sizes were 0.74 to 1.24 ha. The SAI was 71-350 days.

The residues of tebufenozide from trials approximating proposed GAP ranged from 0.1 to 0.6 mg/kg in the stalk and were 0.4 and 1.3 mg/kg in stalk with foliage. Two samples of stalk were analysed for RH-0897. Both contained <0.01 mg/kg.

Table 26. Residues of tebufenozide in celery, USA, 1992-1995.

State, year	Form.	Application			PHI, days	Residue, mg/kg	Reference
		No.	kg ai/ha	kg ai/hl			
MI	SC	7	0.14	0.045-0.047	0	1.4	Chen, 1994d
1992					6	0.47	
FL	SC	7	0.14	0.015	9	0.1	Chen, 1994d
1992-1993							
CA	SC	7	0.14	0.030	0	2.3*	Chen, 1994d
1993					7	1.3*	
					0	0.29	
					7	0.49	
MI	SC	8	0.14	0.05-0.055	0	1.2	Chen, 1994d
1993					6	1.2	
					13	0.64	
CA	SC	8	0.14	0.075	0	1.2*	Chen, 1994d
1993					7	0.41*	
					0	0.15	
					7	0.09	
MI	SC	8	0.28	0.100-0.111	0	4.5	Chen, 1994d
1993					6	3.2	
					13	1.5	
CA	SC	7	0.14	0.025	0	0.38	Dong, 1995c
1994					7	0.64	
CA	WP	7	0.14	0.025	0	0.47	Dong, 1995c

State, year	Form.	Application			PHI, days	Residue, mg/kg	Reference
		No.	kg ai/ha	kg ai/hl			
1994					7	0.6	
FL	SC	9	0.14	0.05	0	0.08	Dong, 1995c
1995					7	0.04	
FL	WP	9	0.14	0.05	0	0.08	Dong, 1995c
1995					7	0.05	

* stalk with foliage

Lettuce. Six field trials with head lettuce and four with leaf lettuce were carried out at different locations in the USA (Chen *et al.*, 1994d). The plot sizes ranged from 0.52 to 1.68 ha. In three of the head lettuce trials samples with and without wrapper leaves were analysed and in the other three only samples with wrapper leaves. The LOD was 0.01 mg/kg and the SAI ranged from 246 days to just over three years. In three samples analysed for RH-0897 the residues were all below the LOD of 0.01 mg/kg.

Two trials on head lettuce with wrapper leaves and two on leaf lettuce were conducted with tebufenozide formulated as the 2SC and 70WP at 0.14 kg ai/ha (Dong, 1995c). The SAI ranged from 178 to 344 days.

Residues from 7-8 applications at 0.14 kg ai/ha, at a 7-day PHI, in head lettuce ranged from <0.01 mg/kg without wrapper leaves to 6.6 mg/kg wrapper leaves, and in leaf lettuce from 1.1 to 3.2 mg/kg.

Table 27. Residues of tebufenozide in head lettuce, USA, 1991-1994.

State Year	Form.	Application			PHI, days	Residue, mg/kg	Reference
		No.	kg ai/ha	kg ai/hl			
NJ	SC	7	0.14	0.043	0	0.03[1]	Chen, 1994d
1991					7	0.009[1]	
					0	0.48	
					7	0.092	
CA	SC	5	0.14	0.075	30	<0.01	Chen, 1994d
1991							
CA	SC	8	0.14	0.075	0	1.7[1]	Chen, 1994d
1991					7	0.053[1]	
					0	5.1	
					7	0.83	
FL	SC	8	0.14	0.019-0.03	0	0.09[1]	Chen, 1994
1991					7	0.018[1]	
					14	0.006[1]	
					0	1.0	
					7	0.9	
					14	0.02	

State Year	Form.	Application			PHI, days	Residue, mg/kg	Reference
		No.	kg ai/ha	kg ai/hl			
TX	SC	9	0.14	0.044	0	1.5[1]	Chen, 1994d
1992					7	0.29	
CA	SC	7	0.14	0.042	0	0.41	Chen, 1994d
1993					7	0.14	
					14	0.009	
CA	SC	7	0.14	0.037	0	3	Dong, 1995c
1994					7	2.3	
CA	WP	7	0.14	0.037	0	3.8	Dong, 1995c
1994					7	6.6	
AZ	SC	7	0.14	0.05	0	3.5	Dong, 1995c
1994					7	3.2	
AZ	WP	7	0.14	0.050	0	4.4	Dong, 1995c
1994					7	2.7	

[1] Head without wrapper leaves

Table 28. Residues of tebufenozide in leaf lettuce, USA, 1991-1994.

State, year	Form.	Application			PHI, days	Residue, mg/kg	Reference
		No.	kg ai/ha	kg ai/hl			
NJ	SC	7	0.14	0.043	0	3.5	Chen, 1994d
1991					7	2.2	
CA	SC	8	0.14	0.042	0	5.7	Chen, 1994d
1991					6	1.7	
FL	SC	8	0.14	0.019	0	0.88	Chen, 1994d
1991					7	0.41	
TX	SC	9	0.14	0.044	0	2.7	Chen, 1994d
1991					7	0.69	
CA	SC	7	0.14	0.025	0	3.7	Dong, 1995c
1994					7	1.1	
CA	WP	7	0.14	0.025	0	3.5	Dong, 1995c
1994					7	2.5	
AZ	SC	7	0.14	0.050	0	3.3	Dong, 1995c
1994					7	3.2	
AZ	WP	7	0.14	0.05	0	3.6	Dong, 1995c
1994					7	2.6	

Spinach. Five field trials on spinach were conducted at different locations in the USA. Plot sizes ranged from 0.31 to 2.4 ha (Chen *et al.*, 1994d). Samples from three trials were analysed for RH-0897: all residues were below the LOD (0.01 mg/kg).

Other trials were carried out in the USA to compare the 2SC and 70WP formulations. Plot sizes

ranged from 0.37 to 1.24 ha (Dong, 1995c). The residues of tebufenozide from treatments approximating proposed GAP ranged from 1 to 4.2 mg/kg.

Table 29. Residues of tebufenozide in spinach, USA, 1991-1994.

State, year	Form.	Application			PHI, days	Residue, mg/kg	Reference
		No.	kg ai/ha	kg ai/hl			
VA	SC	6	0.14	0.05-0.058	0	10.3	Chen, 1994d
1991					7	7.1	
AZ	SC	7	0.14	0.074-0.076	0	4.4	Chen, 1994d
1993					7	0.99	
					14	0.13	
CA	SC	8	0.14	0.075	0	5.5	Chen, 1994d
1991					7	8.1	
OK	SC	7	0.14	0.050-0.052	0	5.1	Chen, 1994d
1993					7	1.3	
TX	SC	9	0.14	0.044	0	15	Chen, 1994d
1992					7	2.7	
CA	SC	7	0.14	0.025	0	7.0	Dong, 1995c
1994					7	3.9	
CA	WP	7	0.14	0.025	0	6.95	Dong, 1995c
1994					7	3.3	
TX	SC	7	0.14	0.05	0	8.3	Dong, 1995c
1994					7	3.8	
TX)	WP	7	0.14	0.05	0	7.1	Dong, 1995c
1994					7	4.2	

Broccoli, cabbage and mustard greens. Seven field trials on broccoli, ten on cabbage, and seven on mustard greens were conducted at various locations throughout the USA from 1991 to 1993 (Chen *et al.*, 1994c). The plot sizes were 0.28 to 2.5 ha for broccoli, 0.60 to 1.23 ha for cabbage, and 0.14 to 2.51 ha for mustard greens. Samples of cabbage with and without wrapper leaves were analysed in some of the trials. The residues in the samples without wrapper leaves were generally significantly lower.

Selected samples analysed for RH-0897 showed no residues above the limit of determination (0.01 mg/kg).

Dong, (1995b) compared the 2SC and 70WP formulations with 7 to 9 applications at a rate of 0.140 kg ai/ha. The plot sizes were 0.09 to 1.24 ha for broccoli, 0.60 to 3.01 ha for cabbage, and 1.24 to 1.48 ha for mustard greens. Samples were analysed by the method of Chen *et al.* (1994a).

The results of the trials are shown in Tables 30, 31 and 32.

The residues from tyreatments approximating proposed GAP in the USA were 0.1-0.34 mg/kg in broccoli, <0.01-0.5 mg/kg in cabbage with and without wrapper leaves, and 0.65-5.6 mg/kg in mustard greens.

Table 30. Residues of tebufenozide in broccoli, USA, 1991-1994.

State, year	Form.	Application			PHI, days	Residue, mg/kg	Reference
		No.	kg ai/ha	kg ai/hl			
OR	SC	8	0.14	0.044-0.047	0	0.67	Chen, 1994c
1991					7	0.24	
TX	SC	8	0.14	0.047	0	0.45	Chen, 1994c
1991					7	0.11	
CA	SC	8	0.14	0.042	0	0.33	Chen, 1994c
1991					7	0.09	
OR	SC	9	0.14	0.03	7	0.11	Chen, 1994c
1992							
VA	SC	7	0.14	0.038	7	0.330	Chen, 1994c
1992							
TX	SC	8	0.14	0.047-0.050	6	0.01	Chen, 1994c
1992							
CA	SC	8	0.14	0.050	0	0.46	Chen, 1994c
1993					7	0.07	
					14	0.05	
CA	SC	7	0.140	0.025	0	0.36	Dong, 1995b
1994					7	0.1	
CA	WP	7	0.14	0.025	0	0.32	Dong, 1995b
1994					7	0.11	
CA	SC	9	0.14	0.025	0	0.75	Dong, 1995b
1994					7	0.31	
CA	WP	9	0.14	0.025	0	0.94	Dong, 1995b
1994					7	0.34	

Table 31. Residues of tebufenozide in cabbage, USA, 1991-1994.

State, year	Form.	Application			PHI, days	Residue, mg/kg	Reference
		No.	kg ai/ha	kg ai/hl			
CA	SC	8	0.14	0.075	0	0.39	Chen, 1994c
1991					7	0.17	
NY	SC	8	0.14	0.026-0.028	0	1.45	Chen, 1994c
1991					7	0.09	
TX	SC	8	0.14	0.047	0	0.24[1]	Chen, 1994c
1991					7	0.01[1]	

State, year	Form.	Application			PHI, days	Residue, mg/kg	Reference
		No.	kg ai/ha	kg ai/hl			
					0	1.1	
					7	0.11	
GA	SC	9	0.14	0.075	0	0.07[1]	Chen, 1994c
1991					7	0.01[1]	
					0	1.3	
					7	0.38	
OH	SC	7	0.14	0.048-0.054	0	<0.01[1]	Chen, 1994c
1992					8	<0.01[1]	
					0	<0.01	
					8	0.004	
FL	SC	7	0.14	0.015	9	<0.01[1]	Chen, 1994c
1992					9	0.03	
CA	SC	8	0.14	0.05	0	1.5	Chen, 1994c
1993					7	1.0	
					14	0.91	
TX	SC	7	0.14	0.045-0.062	0	0.02[1]	Chen, 1994c
1993					7	<0.01[1]	
					14	<0.01[1]	
					0	0.05	
					7	0.30	
					14	0.06	
WI	SC	7	0.14	0.057-0.064	0	<0.01[1]	Chen, 1994c
1993					7	0.01[1]	
					0	0.01	
					7	0.04	
VA	SC	7	0.14	0.037	7	0.53	Chen, 1994c
1993							
TX	SC	9	0.14	0.05-0.075	0	0.8	Dong, 1995b
1994					7	0.78	
TX	WP	9	0.14	0.05-0.075	0	1.2	Dong, 1995b
1994					7	1.3	
FL	SC	9	0.14	0.05	0	3.6	Dong, 1995b
1994					7	4.6	
FL	WP	9	0.14	0.05	0	5.2	Dong, 1995b
1994					7	4.3	

[1] cabbage head without wrapper leaves

Table 32. Residues of tebufenozide in mustard greens, USA, 1991-1994.

State, year	Form.	Application			PHI, days	Residue, mg/kg	Reference
		No.	kg ai/ha	kg ai/hl			
CA	SC	8	0.14	0.075	0	8.2	Chen, 1994c
1991					7	3.9	
TX	SC	8	0.14	0.047	0	4.1	Chen, 1994c
1991							
CA	SC	8	0.14	0.075	0	5.5	Chen, 1994c
1991					7	6.9	
CA	SC	8	0.14	0.075	0	5.6	Chen, 1994c
1991					7	4.4	
NJ	SC	7	0.14	0.043	7	5.6	Chen, 1994c
1992							
NJ	SC	7	0.14	0.043	0	4.1	Chen, 1994c
1993					7	1.6	
AZ	SC	7	0.14	0.060	0	7.1	Chen, 1994c
1993-1994					7	2.6	
					14	1.6	
CA	SC	7	0.14	0.025	0	4.3	Dong, 1995b
1994					7	0.65	
CA	WP	7	0.14	0.025	0	2.5	Dong, 1995b
1994					7	0.93	
TX	SC	8	0.14	0.050-0.075	0	5.1	Dong, 1995b
1994					7	1.9	
TX	WP	8	0.14	0.050-0.075	0	6.8	Dong, 1995b
1994					7	2.4	

Chilli peppers. A single field trial was carried out in Thailand on Chilli peppers with the 20SC formulation at three different rates on a 0.68 ha plot (Ishii and Higuchi, 1994). Three were 5 applications at intervals of 10 days. The trial was not according to GAP or GLP. The results are shown in Table 33.

Table 33. Residues of tebufenozide in Chilli peppers, Thailand, 1993 (Ishii and Higuchi, 1994).

Form.	Application			PHI, days	Residue, mg/kg[1]
	No.	kg ai/ha	kg ai/hl		
SC	5	0.10	0.013	0	0.18
				3	0.08
				7	0.06
				14	0.04
SC	5	0.15	0.020	0	0.21
				3	0.14
				7	0.1
				14	0.04
SC	5	0.20	0.027	0	0.36

Form.	Application			PHI, days	Residue, mg/kg[1]
	No.	kg ai/ha	kg ai/hl		
				3	0.18
				7	0.14
				14	0.11
SC	5	0.30	0.040	0	0.8
				3	0.5
				7	0.26
				14	0.18

[1] The reported values are average from duplicate analyses

Chinese kale. In a similar trial on Chinese kale with the 20SC formulation at three rates in Thailand in 1993 (Ishii and Higuchi, 1993) the plot size was 0.94 ha. There were 5 sprays 4 days apart. The trial did not comply with GLP or GAP (Table 34).

Table 34. Residues of tebufenozide in Chinese kale, Thailand, 1993 (Ishii and Higuchi, 1994).

Form.	Application			PHI, days	Residue mg/kg
	No.	kg ai/ha	kg ai/hl		
SC	5	0.125	0.017-0.027	0	8.0
				3	3.8
				5	2.4
				10	0.73
				15	0.35
SC	5	0.150	0.020-0.032	0	11.0
				3	5.3
				5	3.2
				10	0.68
				15	0.54
SC	5	0.250	0.033-0.053	0	17.2
				3	8.8
				5	5.2
				10	1.5
				15	0.88

Rice. A total of eight trials, four of them official, with two different formulations, 0.75DL (dustless) and 10WP, were conducted in Japan (Komatsu and Yabusaki, 1992c). The results of duplicate anaylses were averaged. Rice samples were harvested at maturity: pre-harvest intervals were varied by changing the date of the last spray application rather than the date of harvest. After harvesting, samples were dried and rough rice was dehulled and sieved through a 1.7 mm sieve. Samples of grain and staw were analysed for the parent compound and in some cases for RH-1788 and RH-6595 by the methods of Komatsu and Yabusaki

(1992a,b). The reported LOD was 0.005 mg/kg.

Two trials were conducted in 1992 in Spain (Mellet, 1993b) with one aerial application at a rate of 0.240 kg ai/ha. The plot sizes were 5 and 10 ha. Rice was harvested 37 or 54 days after the treatment. Approximately 1 kg each of straw, brown rice and cleaned rice were chosen randomly from the test and control plots. Grain was cleaned in a small pilot plant and the whole straw sample was treated by cutting and mixing. Sub-samples of more than 300 g of grain were then crushed to provide the analytical samples. Only the parent compound was determined. The average recovery was 76.4% from straw and 72.8% from rice. The residues in the straw were much higher than in the rice.

The results from Spain and Japan are summarized in Table 35.

Table 35. Residues of tebufenozide in rice grain and straw. Underlined residues are from treatments according to GAP.

Country, Location, year	Form.	Application			PHI, days	Residue, mg/kg				Reference / comments
		No.	kg ai/ha	kg ai/hl		tebufenozide		Total RH-1788/RH-6595		
						rice	straw	rice	straw	
Japan, Chiba	DL	2	0.30		14	0.05	7.7	<0.005	0.32	Komatsu, 1992c*
1992					21	0.05	6.0	<0.005	0.42	
					30	0.02	4.4	<0.005	0.42	
					45	0.01	5.1	<0.005	0.38	
Japan, Akita	DL	2	0.30		14	0.03	2.2	<0.005	0.22	Komatsu, 1992c*
1992					21	0.02	3.9	<0.005	0.41	
					30	0.02	2.8	<0.005	0.32	
					45	0.006	0.7	<0.005	0.12	
Japan, Hyogo	WP	2	0.15	0.010	21	0.008	2.6	<0.005	0.2	Komatsu, 1992c*
1992					30	0.01	2.4	<0.005	0.08	
					45	0.006	2.9	<0.005	0.1	
Japan, Ohita	WP	2	0.10	0.010	21	0.07	6.2	0.006	0.2	Komatsu, 1992c*
1992					30	0.08	5.9	0.008	0.1	
					45	0.05	3.1	0.008	0.1	
Japan, Tokushima	WP	2	0.25	0.010	20	0.06				Komatsu, 1992c
1991					30	0.03				
					45	0.01				
Japan, Tochigi	WP	2	0.15	0.010	21	0.007				Komatsu, 1992c
1992					30	0.01				
					45	0.01				
Japan, Hiroshima	DL	2	0.30		14	0.02				Komatsu, 1992c
1991					21	0.02				
					30	0.01				
					45	0.01				
Japan, Hyogo	DL	2	0.30		14	<0.005				Komatsu, 1992c
1992					21	<0.005				
					30	<0.005				
					45	<0.005				
Spain 1992	SC	1	0.24	0.012	37	0.06	0.82			Mellet, 1993b
Spain 1992	SC	1	0.24	0.012	54	0.02 (Brown rice also 0.02)	1.23			Mellet, 1993b

DL: Dustless * Official trial

Nut crops

Pecans. Eight field trials on pecans were conducted in geographically representative States in the USA (Cui and Desai, 1995). Four of them were at the proposed GAP rate, but six applications were made in all the trials whereas five are proposed. The plot sizes ranged from 2.32 ha to 13.17 ha with 4 to 12 trees per plot. Pecan kernels were analysed for tebufenozide by the method described by Cui and Desai (1994). As shown

in Table 36, the residues of tebufenozide were below the LOD of 0.01 mg/kg in all the trials, and below the limit of detection of 0.003 mg/kg in most of them.

Table 36. Residues of tebufenozide in pecan kernels, USA, 1993 (Cui and Desai, 1995).

State	Form.	Application			PHI, days	Residue, mg/kg
		No.	kg ai/ha	kg ai/hl		
TX	SC	6	0.140	0.017	0	ND
					14	ND
					28	ND
TX	SC	6	0.280	0.035	0	<0.01
					14	ND
					28	ND
NM	SC	6	0.140	0.015	0	ND
					14	ND
					28	ND
NM	SC	6	0.280	0.303	0	ND
					14	ND
					28	<0.01
AL	SC	6	0.140	0.009-0.015	0	ND
					14	ND
					28	ND
AL	SC	6	0.280	0.019-0.031	0	<0.01
					14	ND
					28	ND
GA	SC	6	0.140	0.016	0	ND
					14	ND
					28	ND
GA	SC	6	0.280	0.033	0	ND
					14	ND
					28	ND

ND < limit of detection (0.003 mg/kg)

Walnuts. In six field trials on walnuts according to GAP at five sites in the USA in 1992 (Cui *et al*. 1993c) four applications were made to plots of 1.64-5.95 ha with four trees per plot at monthly intervals. Nut-meat samples were analysed for tebufenozide by the analytical method of Cui *et al*. (1993a) with an LOD of 0.01 mg/kg.

Two field trials in 1994 in the USA were each with both 2SC and 70WP formulations (Dong, 1995a). Four ground applications were made by airblast at the GAP rate of 0.28 kg ai/ha. The plot sizes ranged from 4.46 to 6.83 ha with 6 trees per plot. The LOD was 0.01 mg/kg and the limit of detection 0.003 mg/kg.

Two field trials were conducted in France in 1993 and 1994 (Benzekri, 1995), the 1993 trial with five applications from June to August at various intervals and the 1994 trial with four applications at 3-

week intervals.

The residues of tebufenozide in walnut kernels from applications according to GAP ranged from undetectable (0.003 mg/kg) to 0.02 mg/kg.

Table 37. Residues of tebufenozide in walnut kernels. Underlined residues are from treatments according to GAP.

Country (state), year	Form.	Application			PHI, days	Residue, mg/kg	Reference/Comments
		No.	kg ai/ha	kg ai/hl			
USA (CA)	SC	4	0.280	0.014	30	<0.01	Cui, 1993b
1992					43	ND	Limit of detection 0.003 mg/kg
USA (CA)	SC	4	0.560	0.029	30	0.02	Cui, 1993b
1992					43	ND	
USA (CA)	SC	4	0.235-0.280	0.013-0.017	30	<0.01	Cui, 1993b
1992					43	0.01	
USA (CA)	SC	4	0.560	0.031-0.034	30	0.02	Cui, 1993b
1992					43	ND	
USA (OR)	SC	4	0.280	0.015	29	ND	Cui, 1993b
1992					41	ND	
USA (OR)	SC	4	0.560	0.030	29	ND	Cui, 1993b
1992					41	ND	
USA (OR)	SC	4	0.280	0.015	29	0.02	Cui, 1993b
1992					41	ND	
USA (OR)	SC	4	0.560	0.030	29	0.05	Cui, 1993b
1992					41	<0.01	
USA (CA)	SC	4	0.280	0.010-0.029	30	ND	Cui, 1993b
1992					44	<0.01	
USA (CA)	SC	4	0.560	0.020-0.058	30	0.01	Cui, 1993b
1992					44	<0.01	
USA (CA)	SC	4	0.280	0.010-0.029	30	0.02	Cui, 1993b
1992					45	0.01	
USA (CA)	SC	4	0.560	0.020-0.058	30	0.03	Cui, 1993b
1992					45	<0.01	
USA (CA) 1994	SC	4	0.280	0.015	6	ND	Dong, 1995a.
USA (CA) 1994	WP	4	0.280	0.015	6	<0.01	Dong, 1995a.
USA (CA) 1994	SC	4	0.280	0.027	30	ND	Dong, 1995a.
USA (CA) 1994	WP	4	0.280	0.030	30	ND	Dong, 1995a.
France	SC	5	0.144	0.029	31	ND	Benzekri, 1995
1993							Limit of detection 0.003 mg/kg
France 1994	SC	4	0.144	0.029	33	ND	Benzekri, 1995

ND < Limit of detection; 0.003 mg/kg

Tea. Summaries of two field trials, one of them according to GAP, conducted in Sri Lanka in 1994 were reported (Ishii, 1995b). Critical data such as plot size and type of application were not provided. The analyses were carried out in Japan. The residue in the trial which complied with GAP was 14.6 mg/kg.

Two trials were conducted according to GAP at different locations in Japan in 1993 (Komatsu and Yabusaki, 1993). Samples were analysed for the parent compound, RH-6595 and RH-1788 by GLC. Residues of the parent compound at the GAP PHI were 11.5 and 11.6 mg/kg. The corresponding levels of RH-1788 + 6595 were 0.23 and 0.24 mg/kg.

The residues found in dry and brewed tea from the trials in Sri Lanka and Japan are shown in Table 38.

Table 38. Residues of tebufenozide in dry and brewed tea from SC applications. Underlined residues are from treatments according to GAP.

Country Year	Application		PHI, days	Residue, mg/kg		Reference
	No.	kg ai/ha		Dry tea	Brewed tea	
Sri Lanka	1	0.15	0	93.2	22.6	Ishii, 1995b
1994			4	22.3	4.1	
			7	<u>14.6</u>	2.66	
			10	3.9	0.8	
			21	0.49	0.08	
			28	0.13	0.02	
Sri Lanka	1	0.30	0	87.2	22.0	Ishii, 1995b
1994			4	22.0	4.4	
			7	16.0	3.0	
			10	4.3	0.8	
			21	0.6	0.1	
			28	0.2	0.03	
Japan	2	0.40	14	<u>11.5</u>	<u>3.5</u>	Komatsu, 1993
1993	2	0.40	21	6.2	0.9	
	2	0.40	30	2.4	0.3	
	2	0.40	14	<u>11.6</u>	<u>1.4</u>	
	2	0.40	21	2.0	0.2	
	2	0.40	30	0.2	0.01	

Animal feeding study

In a study in Japan two cows were dosed by capsule with either 40 or 400 mg tebufenozide bw day for 7 days (Inoue, 1993). From the average weight of the cows, these doses correspond to 0.074 and 0.74 mg/kg per day. Milk was collected on days 1, 3, 5, and 7. Milk samples were injected onto a C-18 Sep-Pak and the fraction containing tebufenozide was concentrated and analysed on a TSK-gel ODS-80 HPLC column eluted with acetonitrile/water (7:3) with UV detection at 254 nm. The limit of determination was 0.02 mg/kg. There were no detectable residues in any of the milk samples.

It was reported that a feeding study was in progress in the USA in which groups of four cows had been dosed with the equivalent of 0, 6, 18, and 60 ppm of tebufenozide in the diet. Residues of tebufenozide and the main metabolites identified in the goat metabolism study were being determined in the milk, liver, muscle, kidney and fat.

No poultry feeding study was reported. Most of the crops for which registration of tebufenozide has been approved or is expected do not produce feed components for poultry. An exception is cotton, from which cotton seed meal can be used, but no data from this crop were provided.

FATE OF RESIDUES IN STORAGE AND PROCESSING

In storage

No data were provided.

In processing

Processing studies have been conducted on apples, grapes and tea.

Apples. Washed apples from two trials in Germany at a two-fold rate were processed into juice and purée on a houshold scale (Raquet *et al*., 1993). Sample were analysed by GLC with an LOD of 0.01 mg/kg for juice and washings and 0.02 mg/kg for fruit.

The aqueous washings showed no residues above 0.02 mg/kg. The peel obtained during the production of purée was not analysed. Pomace from the production of juice showed slightly higher residues than those in the original fruit. The results are shown in Table 39.

Apples from other German trials (Brusche and Holzwarth, 1995) were processed to purée and juice. The procedures are shown in Figures 9 and 10. After the addition of 0.5 l water per kg fruit, the fruit was boiled for 20 minutes and sieved. The fractions obtained were washed fruit, washings, peels and pips, and purée (apple sauce). For the preparation of juice, cut apples were pressed in a juice extractor without prior heating. The crude juice was then filtered, poured into glass bottles, and pasteurized for 25 minutes at 75ºC. The low concentration of tebufenozide in the washings indicates that washing does not substantially reduce the residues in the fruit. About 40-60% of the residues in the harvested apples remained in the wet pomace or peels and pips.

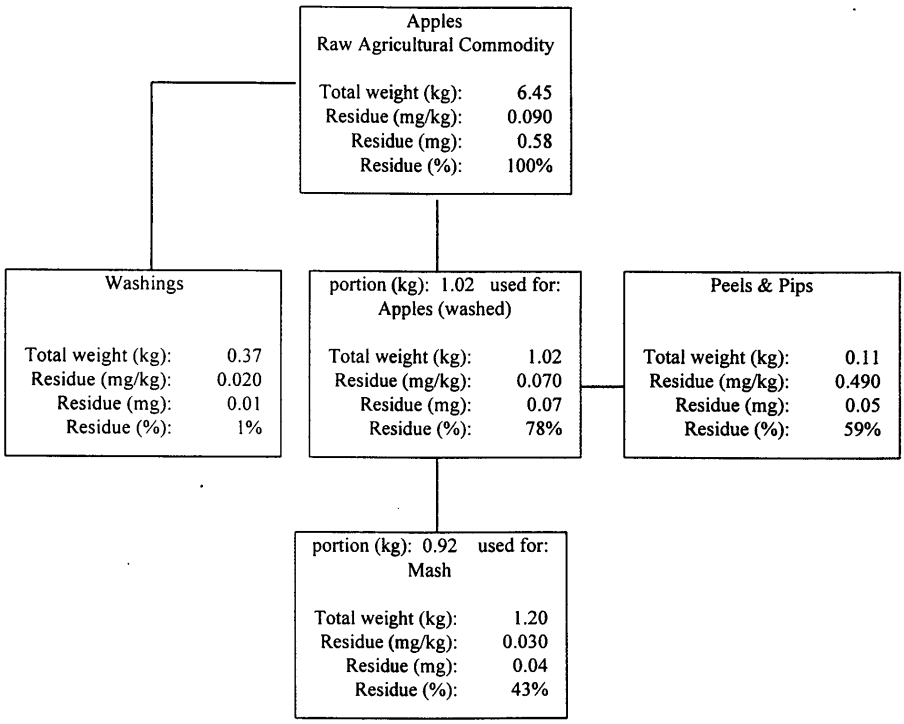
Table 39. Residues of tebufenozide in apples and processed fractions.

Country, year	kg ai/ha	PHI, days	Residue, mg/kg (Ratio to residue in unwashed fruit)						Reference
			Unwashed fruit	Washed fruit	Wet pomace	Purée	Washings	Juice	
Germany	0.366	28	0.08	0.05	0.11	0.02	<0.01	0.01	Raquet, 1993
1992				(0.63)	(1.4)	(0.25)		(0.13)	
Germany	0.382	28	0.11	0.06	0.13	0.02	<0.01	<0.01	Raquet, 1993

Country, year	kg ai/ha	PHI, days	Residue, mg/kg (Ratio to residue in unwashed fruit)						Reference
			Unwashed fruit	Washed fruit	Wet pomace	Purée	Washings	Juice	
Germany	0.366	28	0.08	0.05	0.11	0.02	<0.01	0.01	Raquet, 1993
1992				(0.55)	(1.2)	(0.18)		10* (0.09)	
Germany	0.54	25	0.1	0.15	0.22	0.02	0.02	0.03	Brusche, 1995
1993				(1.5)	(2.2)	(0.2)	(0.2)	(0.3)	
Germany	0.54	27	0.07		0.3	0.03	peel and pips 0.49	0.02	Brusche, 1995
1993					(4.3)	(0.43)	0.02 (0.29)	(0.29)	
Germany	0.54	27	0.18	0.18	0.26	0.04	0.02	0.04	Brusche, 1995
1993				(1.0)	(1.4)	(0.22)		(0.22)	
USA	2.80	7	0.57		0.85			0.54	Deakyne, 1994c
1991					(1.5)			1 (0.95)	
USA	2.02	7	0.56	0.44	2.4		pasteurized	0.025 (0.05)	Deakyne, 1995a
1994				(0.79)	(4.3)		unpasteurized	0.026 (0.05)	

Figure 9. Apple processing scheme 1 (Germany).

% values refer to absolute amount of tebufenozide (mg) in the raw agricultural commodity (unwashed apples)



N.B.: for the preparation of mash 0.5 litre water/kg peeled fruit was added

Red delicious apples were treated with tebufenozide (10 applications at 0.28 kg ai/ha each) in the USA (Deakyne, 1994). The treatment was not according to GAP but was designed to produce high residues for processing. Unwashed apples harvested 7 days after the final application were sliced with a Hobart slicer and then added to a Hobart pulp/juicer. The juice and wet pomace were collected in separate containers. The wet pomace was pressed in a small wine press to remove excess juice which was added to the juice previously collected. Half the wet pomace was further homogenized (wet pomace fraction). Samples were analysed by HPLC (Deakyne *et al.*, 1994b). The limit of determination was 0.02 mg/kg. The residue was slightly reduced in juice but slightly increased in wet pomace.

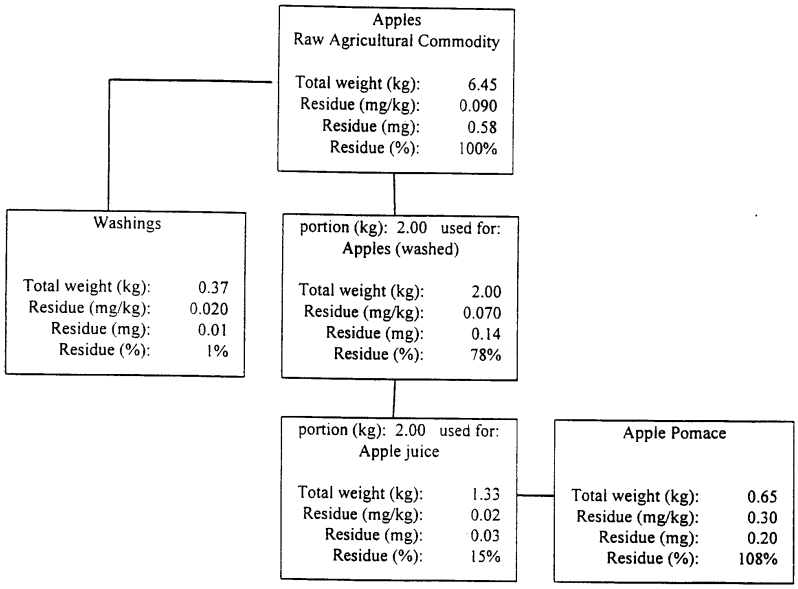
In a 1994 processing study in the USA (Deakyne, 1995a), six applications of the 70WP formulation of tebufenozide were made at 0.336 kg ai/ha to red delicious apples, a total of 2.0 kg ai/ha. Apples harvested 7 days after the final application were processed to produce pomace, unpasteurized juice and pasteurized juice by a procedure similar to the commercial process (Figure 11). The apples were lightly washed as is typical of commercial practice before grinding in a Fitzpatrick hammer mill with a no. 4 screen. A small press was used to press out the apple juice from the mash. The freshly squeezed juice was canned and some cans were subjected to pasteurization in a heat exchanger at a minimum temperature of 87.7ºC. The ground wet pomace taken directly from the press was split into sublots and stored frozen at -23.3ºC. All samples were kept frozen until analysis in duplicate by the method of Deakyne, *et al.* (1994b). The results are shown in Table 39. Washing the treated apples with water reduced the residues by about 20%. Pasteurized and unpasteurized juice contained essentially identical residues, 0.025 and 0.026 mg/kg respectively, about 5% of the level in the paplies. The residue in the pomace was about four times that in the whole fruit.

Processing apples to purée and juice reduced the residues considerably. In German apple processing studies about 40-60% of the residues on the harvested samples remained with the pomace or peel and pips, with an overall reduction of the residue level of 2.3-5.5-fold for the preparation of purée and 3-10-fold for the preparation of juice.

A 1-4-fold concentration of the residues in wet pomace was found in apple processing studies in Germany and the USA.

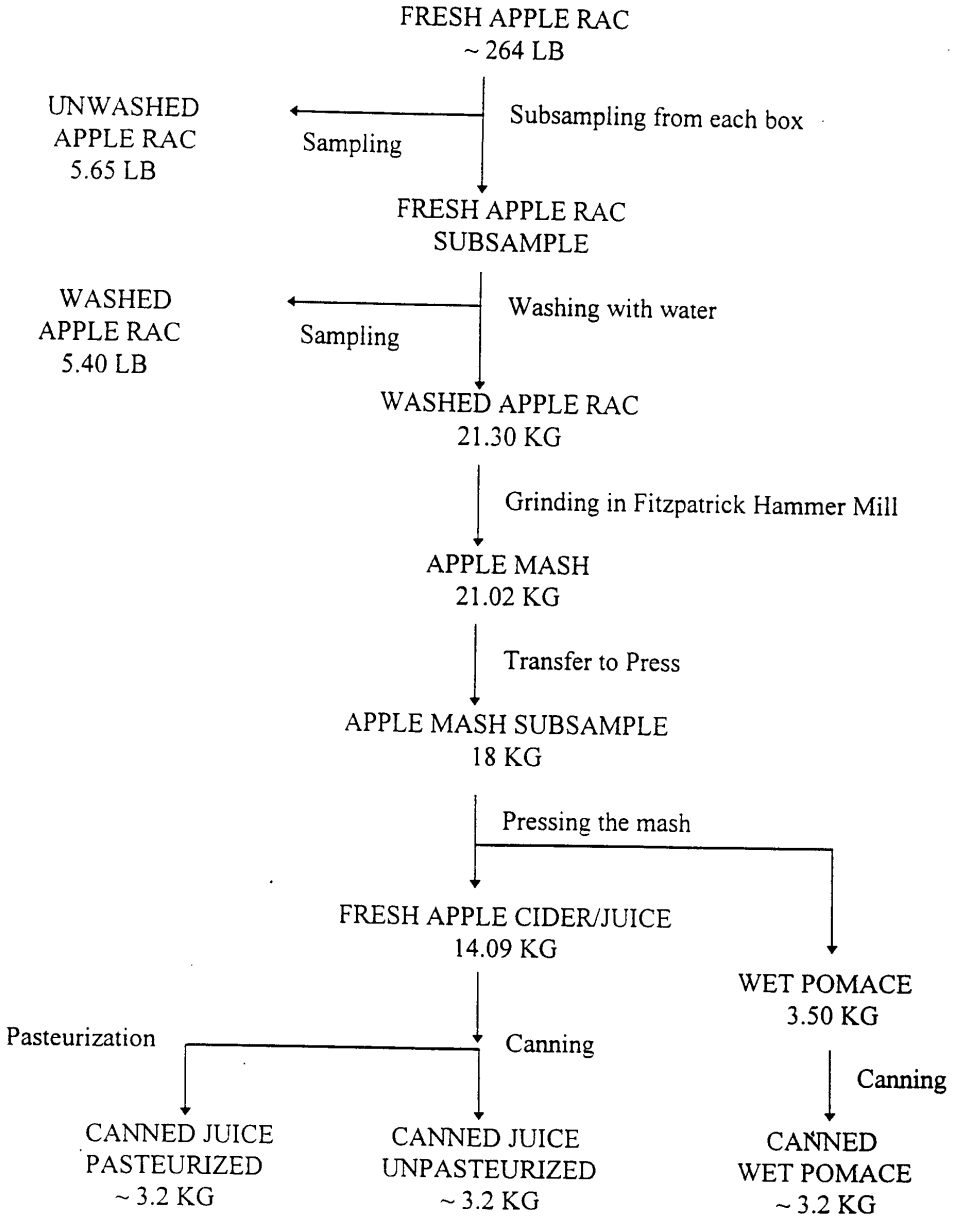
Figure 10. Apple processing scheme 2 (Germany).

% values refer to absolute amount of tebufenozide (mg) in the raw agricultural commodity (unwashed apples)



N.B.: for the preparation of mash 0.5 litre water/kg peeled fruit was added

Figure 11. Apple processing scheme (USA).



Grapes. Three trials in 1992 and two in 1993 were conducted in France for processing studies (DeWilde *et al.,* 1995a,b) with three applications at the recommended rate of 0.144 kg ai/ha. The grapes were harvested at the maturity stage required for wine production and processed to either red or white wine depending on the grape variety.

Processing was by the officially recommended method of the Ministry of Agriculture in France to establish the influence of plant protection products on the wine-making process (DeWilde *et al.,* 1995a). The vinification processes for red and white wines are shown in Figures 12 and 13. Normally white wine is fermented without the grape skins but the skins were included in some white fermentations to assess the residue distribution.

Samples were analysed by the GLC method of Mellet (1993a). Processing of grapes to must and wine reduced the residues in the wine to about 20-40% of those in the grapes and increased the residues in the must with skins by about 13-40%. A single sample of pomace showed a concentration of the residue.

The fate of tebufenozide during the distillation of wine spiked with it was studied in France (Gocha, 1995a). A solution containing 2.5 mg of tebufenozide was added to one of two 5 l jars of wine to give a concentration of 0.5 mg/l. The second jar was used as a control. The wine was kept at room temperature overnight before distilling 4800 ml each of control and treated samples under conditions simulating those used commercially to produce cognac. Four fractions were analysed: wine before distillation, the fraction distilling at increasing temperatures up to 89ºC, the fraction distilling at a constant 90ºC, and the undistilled residue. Analyses were by the method of Mellet (1993a). No tebufenozide was detected in any of the distilled fractions.

Processing trials were conducted in Germany in 1992 (Ulrich *et al.,* 1994) and 1993 (Kaiser, 1994). The plot size in 1992 was governed by the number of samples to be taken but was not less than 0.58 ha. At the last sampling (28-day PHI), about 200 kg grapes were taken from at least 30 vines at 2 locations for processing to must and wine. The processing scheme is shown in Figure 14. The first wine sample was taken for residue analysis at the time of bottling at the filter outlet and a second about 6 months after bottling (the normal maturation period). Processing the grapes to must (without skins) and wine reduced the residues. In the 1993 trials the residues in must (without skins) were lower and those in pomace higher by factors of about 3 to 4 than those in the corresponding grapes. The residues in the must were 0.06 and 0.12 mg/kg.

In trials in Italy (Kaiser and Holzwarth, 1994) harvested white grapes were crushed and destalked, then pressed to give must (without skins) accounting for 60 to 65% of the total mass of the grapes. For red wine, the destalked mash was fermented for 4 to 6 days before pressing and further fermentation and run-off as for white wine. After storage of the young wine in stainless steel containers for about 5 months, wine samples were taken for residue analysis. The residues in the must and wine were again significantly lower than in the fresh grapes. The results of all the trials are shown in Table 40. Residues inmature wine are compared with those in the original grapes in Table 41, which shows that residues in the wine were on average only 36% of those in the grapes.

Table 40. Residues of tebufenozide in processed products of grapes.

Country, year, red/white graphes	Total applicn., kg ai/ha	PHI, days	Residue (mg/kg)					Comments	Reference
			Grapes	Must	Pomace	Wine			
						young	matured		
France 1992 r	0.432	40	0.18	0.25[1]			0.08	duplicate analyses	DeWilde, 1995a
France 1992 r	0.432	41	0.06	0.08[1]			0.03	duplicate analyses	DeWilde, 1995a
France 1992 w	0.432	25	0.08[1]	0.09[1]			0.03	duplicate analyses	DeWilde, 1995a
France 1993 w	0.432	19	0.17	0.06[2]	0.266		0.04	triplicate analyses of grapes mud=0.129 lees=0.168	DeWilde, 1995b
France 1993 r	0.432	21	0.28	0.34[1]		0.09	0.06		DeWilde, 1995b
France 1991 r	0.144	66	0.11				0.05	triplicate analyses PHI=70 days for wine	Gocha, 1995a
France 1992 w	0.288	50	0.17	<0.01				spirit <0.01	Gocha, 1995a
Germany 1992 w	0.557	28	0.27	0.03[2]		0.02	0.02		Ulrich, 1994
Germany 1992 r	0.555	28	0.4	0.12[2]		0.18	0.26		Ulrich, 1994
Germany 1993 w	0.519	28	0.22	0.06[2]	0.620		0.08		Kaiser, 1994
Germany 1993 r	0.535	28	0.42	0.12[2]	1.570		0.14		Kaiser, 1994
Italy 1992 r	0.478	21	0.56	0.07[2]		0.18[3] (0.11)[4]	0.1		Kaiser and Holzwarth, 1994
Italy 1992 r	0.96	21	0.86	0.07[2]		0.23[3] (0.17)[4]	0.17		Kaiser and Holzwarth, 1994
Italy 1992 w	0.456	21	0.26	0.03[2]		0.1[3] (0.14)[4]	0.18		Kaiser and Holzwarth, 1994
Italy 1992 w	0.96	21	0.84	0.09[2]		0.27[3] (0.37)[4]	0.33		Kaiser and Holzwarth, 1994

[1] Fermentation with skins
[2] Fermentation without skins
[3] Mid-fermentation
[4] End of fermentation

Figure 12. Red wine vinification process (France).

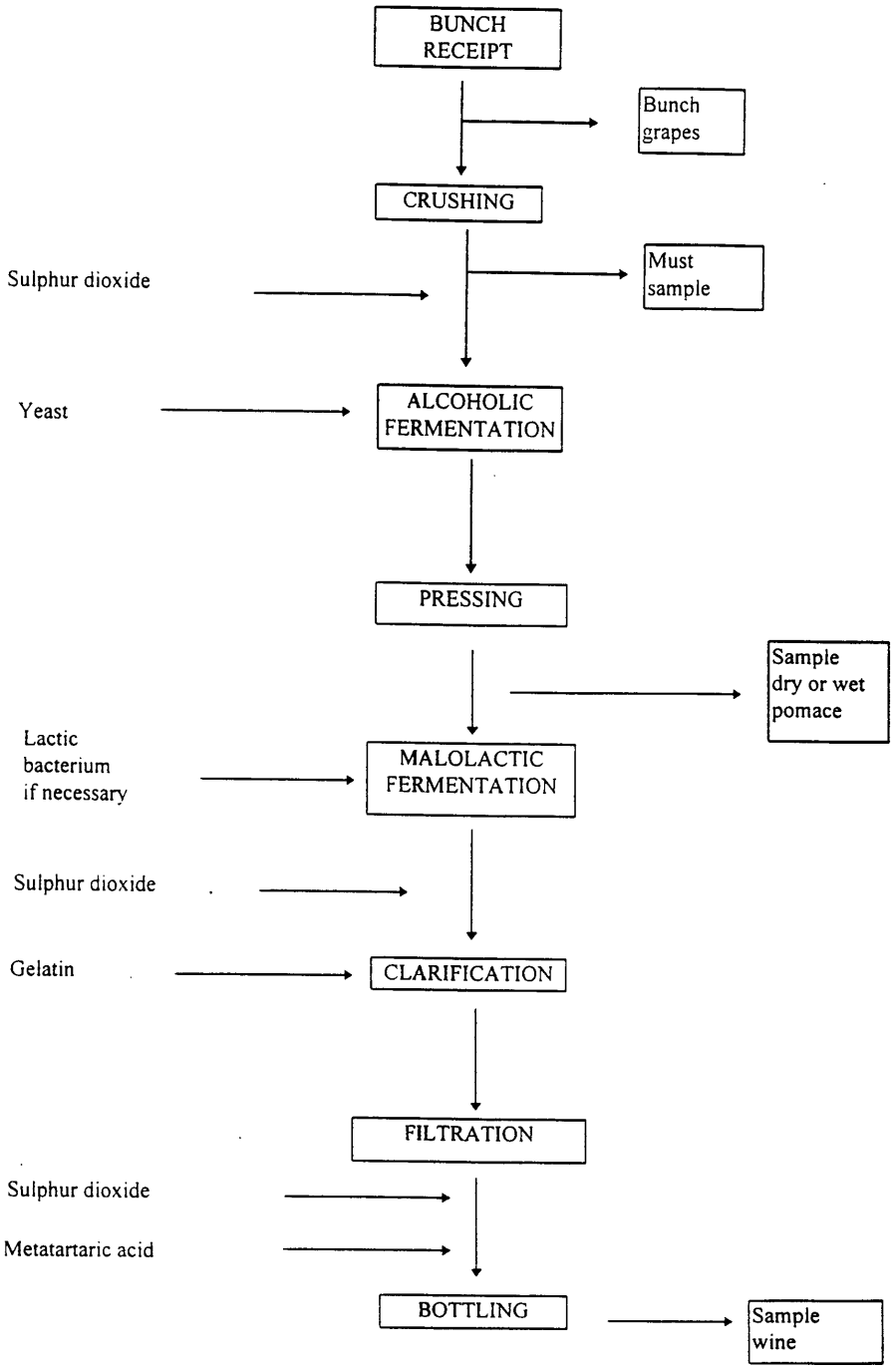


Figure 13. White wine vinification process (France).

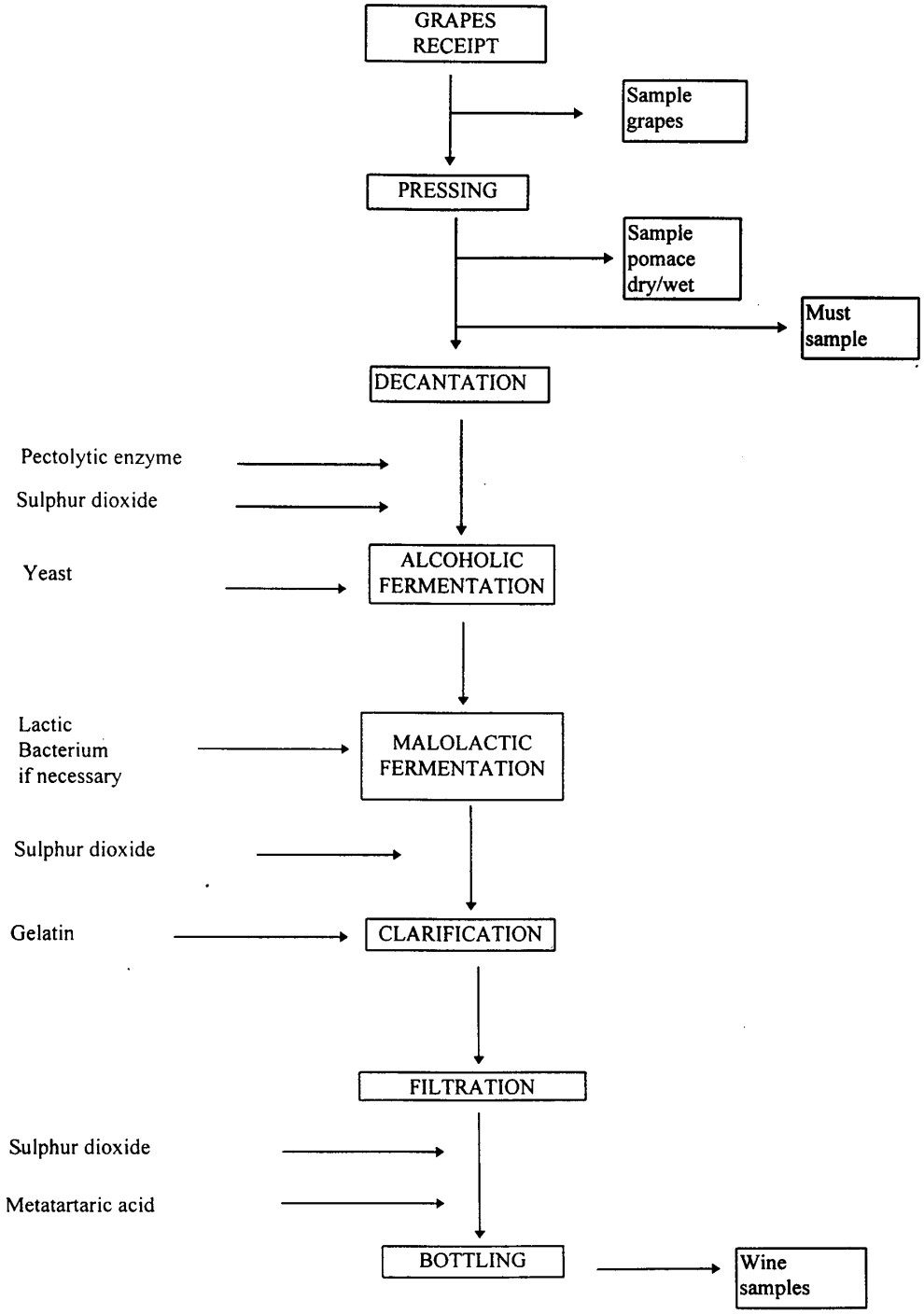


Figure 14. Wine vinification process (Germany, Italy).

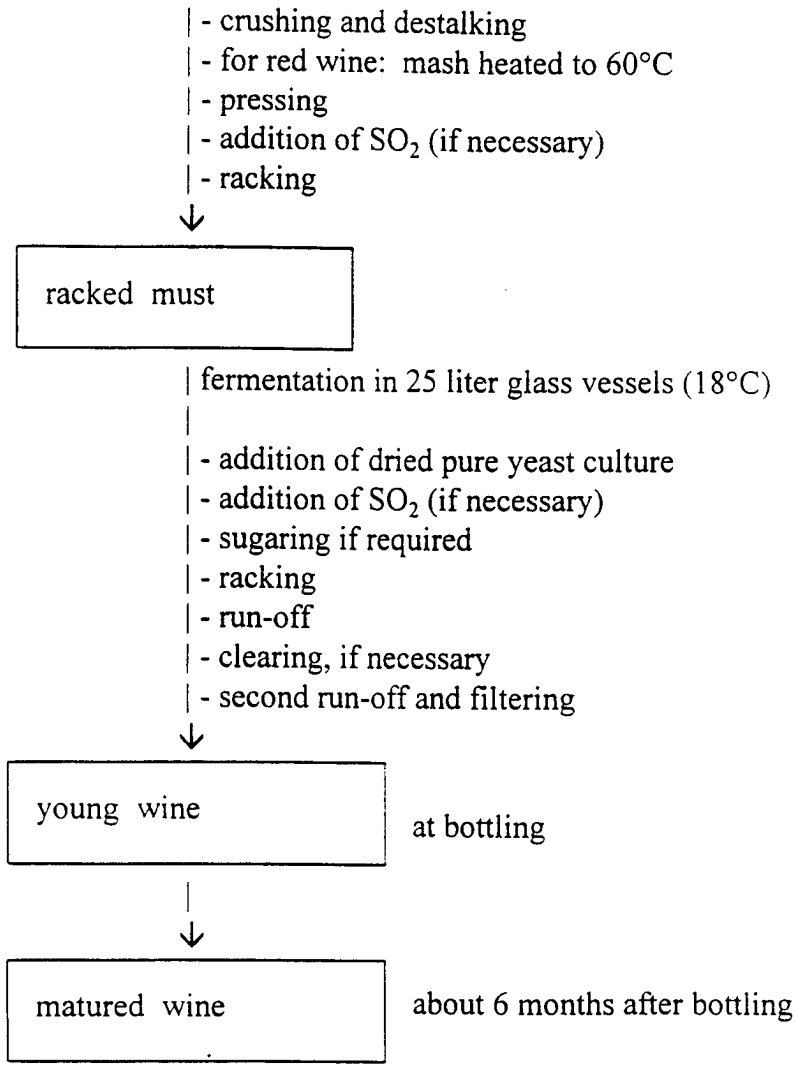


Table 41. Residues of tebufenozide in wine and grapes.

Country	Total treatment kg ai/ha	Grape type	Residue mg/kg		Reference	mg/kg in wine as percentage of mg/kg in grapes
			Grapes	mature wine		
France	0.432	red	0.179	0.083	DeWilde, 1995a	46
	0.432	red	0.065	0.029		45
	0.432	white	0.080	0.025		31
France	0.432	white	0.166*	0.038	DeWilde, 1995b	23
	0.432	red	0.282	0.063		22
France	0.144	red	0.110	0.048	Gocha, 1995a	44
Germany	0.557	white	0.27	0.02	Ulrich, 1994	7
	0.555	red	0.40	0.26		65
Germany	0.519	white	0.220	0.080	Kaiser, 1994a	36
	0.535	red	0.420	0.140		33
Italy	0.478	red	0.560	0.100	Kaiser, 1994b	18
	0.96	red	0.860	0.170		20
	0.456	white	0.260	0.180		69
	0.96	white	0.840	0.330		39
						Average 36

Tea. Tea was brewed from samples of dry tea obtained from the field trials in Sri Lanka and Japan (Ishii, 1995; Komatsu and Yabusaki, 1993) by adding boiling water to the dry tea (60 ml/g) and allowing the solution to stand for 5 minutes. Samples of filtered brewed tea were analysed after partition and clean-up steps. The final residue in the brewed tea was calculated on the basis of the weight of the dry tea.

Although the residues of tebufenozide are relatively high in dry tea, they are much lower in brewed tea. As shown in Table 42, residues in the infusion, expressed on a dry tea basis, were only 5-31% of those in the original dry tea, with a mean of 17%.

Table 42. Residues of tebufenozide in dry tea and brewed tea.

Country	Application rate kg ai/ha	PHI, days	Residue, mg/kg		Residue in brewed tea as % of that in dry tea
			Dry tea	Brewed tea[1]	
Sri Lanka	0.15	0	93.2	22.6	24
		4	22.3	4.1	18
		7	14.6	2.6	18
		10	3.98	0.78	20
Sri Lanka	0.15	21	0.49	0.08	16
		28	0.13	0.02	15
	0.30	0	87.2	22	25

Country	Application rate kg ai/ha	PHI, days	Residue, mg/kg		Residue in brewed tea as % of that in dry tea
			Dry tea	Brewed tea[1]	
		4	22	4.4	20
		7	16	3	19
		10	4.3	0.76	18
		21	0.59	0.1	17
		28	0.2	0.03	15
Japan	0.80	14	11.5	3.5	31
		21	6.2	0.92	15
		30	2.4	0.29	12
	0.80	14	11.6	1.4	12
		21	2.0	0.18	9
		30	0.22	0.01	5
					Average 17

[1] Expressed on dry tea basis

Residues in the edible portions of food commodities

No data were provided on residues in the edible portions of food commodities except those included in the processing trials described above. These showed overall reduction factors of 2.3-5.5 for preparing apple purée, 3.5 to 22 for preparing apple juice, 2-13 for vinifying grapes and about 3-20 for brewing tea.

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

No information was provided.

NATIONAL MAXIMUM RESIDUE LIMITS

The following national MRLs were reported.

Country	Commodity	MRL, mg/kg
Belgium	Pome fruit	0.2
Canada	Apples	1.0
France	Apples	0.5
	Grapes	0.5
	Walnut	0.05
Israel	Grapes	0.5
Italy	Pome fruit	0.5
	Grapes	0.5
Japan	Apples	0.5
	Rice	0.5

Country	Commodity	MRL, mg/kg
	Sugar beet	0.1
	Tea	25
Korea	Apples	0.5
	Rice	0.5
New Zealand	Pome fruit	0.5
	Kiwifruit	1.0
Philippines	Rice	0.5
Portugal	Pome fruit	0.5
	Grapes	0.5
Spain	Grapes	0.5
Switzerland	Pome fruit	0.3
	Grapes	0.3 (0.1 in wine)
	Cabbage species	0.5
	Lettuce, spinach	1.0
Taiwan	Welsh 0nion	5
Thailand	Rice	0.5
USA	Apples	1.0 (Import tolerance)
	Walnut	0.1

The Meeting was also informed that the following MRLs for tebufenozide were likely to be adopted.

Country	Commodity	MRL, mg/kg
Austria	Grapes	0.5
Austria	Pome fruit	0.5
France	Pears	0.5
Germany	Grapes	0.5
Germany	Pome fruit	0.5
Greece	Pome fruit	0.5
New Zealand	Grapes	0.5
Slovenia	Apple	0.5
Slovenia	Grapes	0.5
Spain	Pome fruit	0.5
Switzerland	Lettuce	1
Switzerland	Spinach	1
USA	Broccoli	2
USA	Celery	10
USA	Kiwifruit	0.3
USA	Pecans	0.05
USA	Vegetables, leafy	2

APPRAISAL

Tebufenozide is an insecticide used to control caterpillar pests in fruits, vegetables, and other crops. It is an agonist of an insect moulting hormone, which is selectively toxic to lepidoptera and a few diptera. It is registered in many countries around the world.

Tebufenozide is marketed in several formulations, the most important being two suspension concentrates and a wettable powder.

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

The fate of tebufenozide has been studied in rats, goats, poultry, fish, apples, grapes, rice, sugar beet, soil and water, with the compound labelled with ^{14}C (a) uniformly in the ethylbenzoyl ring (A-ring label), (b) uniformly in the dimethylbenzoyl ring (B-ring label) and (c) at the central carbon of the *tert*-butyl group (t-butyl label).

The principal metabolites and degradation products identified are as follows.

RH-6595: *N-tert*-butyl-*N′*-(4-acetylbenzoyl)-3,5-dimethylbenzohydrazide
RH-1788: *N-tert*-butyl-*N′*-[4-(1-hydroxyethyl)benzoyl]-3,5-dimethylbenzohydrazide
RH-2703: *N-tert*-butyl-*N′*-(4-carboxymethylbenzyol)-3,5-dimethylbenzohydrazide
RH-9886: *N-tert*-butyl-*N′*-(4-ethylbenzoyl-3-hydroxymethyl-5-methylbenzohydrazide
RH-0282: *N-tert*-butyl-*N′*-[4-(1-hydroxyethyl)benzoyl]-3-hydroxymethyl-5-methylbenzohydrazide
RH-9871: *N-tert*-butyl-*N′*-(4-acetylbenzoyl)-3-hydroxymethyl-5-methylbenzohydrazide
RH-2778: *N-tert*-butyl-*N′*-[4-(1-hydroxyethyl)benzoyl]-3,5-di(hydroxymethyl)benzohydrazide

The metabolic degradation of tebufenozide has been found to proceed generally via oxidation at the alkyl groups located on the two rings, and sometimes conjugation of the oxidation products. Eventually the amide bonds are broken and the structural sub-units are oxidized to CO_2. The parent compound has been a significant component in most metabolism studies together with its oxidation products such as monoalcohols, the RH-6595 ketone and the corresponding acid. More extensively oxidized products such as diacids, diols, and alcohol-acids have also been found in smaller quantities.

Male and female rats were dosed orally with two or three radiolabelled versions of tebufenozide at 3 mg/kg or 250 mg/kg bw and another group received a dose of 3 mg/kg of labelled tebufenozide after being dosed for two weeks with the unlabelled material.

Most of the radioactivity (94-104%) was excreted in the faeces, with low levels in the urine (0.5-8%) in the first 24 hours. The main routes of metabolism were via oxidation of the alkyl groups on the phenyl rings. The main component of the excreted activity was the parent compound, but seventeen metabolites were identified in the faeces. The extent of the metabolism was found to be highly dependent on the amount of the dose. A high dose produced a low proportion of metabolites, while about 50% of a low dose was metabolized.

In goats, most of the radioactivity was excreted in the faeces and urine, less than 0.3% was excreted in the milk and less than 0.4% of the dose remained in the tissues. Levels of ^{14}C in the milk

remained fairly constant through the dosing period. Tebufenozide was extensively metabolized by multiple oxidative transformation. The highest residues in the tissues were found in liver and the next highest in fat.

Milk contained only a low concentration of the parent compound (13.7% of the ^{14}C) together with the alcohl metabolites RH-0282, RH-9871 and RH 9886 and its conjugates. In fat the parent compound and three fatty acid conjugates of RH-9886 were identified. Liver was unique in that no residue of the parent compound was found; the major component was the carboxylic acid RH-2703, together with an almost equal amount of 2-propanol. Tebufenozide was detected in the kidneys and muscle at low levels.

In poultry the highest total residues occurred in the liver, kidney and fat when laying hens were dosed with different radiolabelled versions of tebufenozide for 7 days at a level equivalent to 30 ppm in the feed. Residues in eggs increased during during the feeding period and were still increasing on day 7, although they represented a low percentage of the dose. In eggs the principal residues were tebufenozide and RH-9871 (the alcohol-ketone). The parent compound and RH-0282 (dialcohol) were the major components in the fat, and acetaldehyde and 2-propanol were the major metabolites identified in liver. Excreta wree the main source of metabolites in the study.

Fish. Bluegill sunfish were continuously exposed to a nominal concentration of 50 ì g/l of [^{14}C]tebufenozide for 29 days to study the kinetics of its uptake and elimination. Some bioconcentration of tebufenozide was observed in the fish, but depuration of the residue was rapid: its half-life was less than three days and by the last (15th) day of depuration at least 90% of the radioactivity had been eliminated from the fish.

The main residue in the fish was the parent compound, but eight metabolites were also isolated and identified. Metabolism occurs via oxidation of the ethyl group to an alcohol or ketone and of the two equivalent methyl groups successively to monoalcohol, dialcohol and acid-alcohol.

The fate of residues in plants was studied in apples, grapes, rice and sugar beet using the three radiolabelled versions of tebufenozide. The compound was metabolized by oxidation of the alkyl substituents of the aromatic rings to combinations of alcohols, the ketone and acids. Only grapes were not metabolized.

The parent compound was the main component of the residue in all four crops, constituting essentially 100% of the ^{14}C in grapes, 77% in apples, 72% in rice grain, 67% in sugar beet root and 41% in sugar beet tops. No single metabolite occurred at more than 10% of the total residue or more than 15-20% of the tebufenozide residue in any studied crop.

The extent of metabolism appeared to be related to the length of time in the plant. In grapes where the exposure was 30 days, the residue was 100% parent compound. In rice after 64 days 72-76% of the residue was tebufenozide, and in sugar beet after 120 days 41-67% was the parent compound.

The degree of oxidation also appears to be a function of exposure. In rice two monoalcohols were produced with further oxidation to form the corresponding ketone and aldehyde. In apples the same monoalcohols were again the first metabolites, followed by oxidation of substituents on the second ring to produce the diol RH-0282 and triol RH-2778. In sugar beet, with the longest exposure, there was further oxidation to carboxylic acids. Sugar beet after foliar treatment with tebufenozide showed a moderate translocation of the parent compound and its metabolites from the foliage to the roots. All the metabolites isolated in plants were also observed as metabolites in the rat.

Metabolites were identified by HPLC, TLC, LSC, and MS.

In summary, the plant metabolism studies indicated that the major residual compound in crops is unchanged tebufenozide. Metabolites were not observed in grapes at a 30-day PHI, showing that metabolism of tebufenozide in plants is slow. The principal metabolites found in other crops were RH-6595, RH-1788 and RH-9886. The proportion of metabolites in the residue increased with time.

Studies of the fate of tebufenozide in soil (high organic sand, low organic sand , loamy sand, and sandy loam) under aerobic condition showed that it is degraded by oxidation of the ethyl group to produce the ketone and two carboxylic acids, followed by mineralization to CO_2. The half-life of tebufenozide ranged from 7 to 105 days. Carbon dioxide and unextractable material associated with humic and fulvic acid fractions were the major terminal residues.

A study of the adsorption and desorption of [^{14}C]tebufenozide in five different soils showed that it could be classified as having low mobility. The study suggested that some of the tebufenozide may be irreversibly bound to soil.

The leaching of tebufenozide and its aged residues was studied in different soils. At the end of the leaching period (48 h) only small amounts of tebufenozide had been leached. The study with aged residues, in clay loam, sandy loam, loam and sand, showed that while tebufenozide had some mobility in the sand, the two carboxylic acid products were mobile in all four soils. In another study with aged residues applied to a sandy soil with low organic matter, tebufenozide and its ketone derivative remained in the soil whereas both carboxylic acids were found in the leachates.

Tebufenozide in sterile buffered solutions at pH 5, 7 and 9 at 25ºC showed no hydrolytic degradation.

The photolysis of tebufenozide was studied in a treated soil irradiated with a xenon lamp for 30 days with a 12-hour light/dark cycle. The parent compound was degraded slowly to yield a total of 7 detected products of which the ketone and aldehyde were the major compounds identified. The half-life under these conditions was 98 days. Photolysis was also studied in natural pond water, again with irradiation by a xenon lamp. Tebufenozide was degraded with a half-life of 67 days. The major product was the ketone RH-6595 (5.3% of the radioactivity). Eight other compounds were also detected but none exceeded 3.5% of applied activity. Almost no degradation occurred in the dark control.

Field dissipation studies conducted at different sites in various countries according to the worst-case scenario (a high rate applied directly to bare ground) showed that tebufenozide and its soil degradation products decline at a moderate to a fairly fast rate. When the ground was covered with grass tebufenozide was translocated to the soil, reaching a maximum concentration in soil on day 122. These studies also demonstrated that neither tebufenozide nor its ketone degradation product showed any downward mobility. Residues of tebufenozide were principally found in the top (7.5-10 cm depth) of the soils. It was also shown that the parent compound is degraded more slowly in soils with a low pH (3.9-5.1). Degradation products of tebufenozide were only occasionally observed above the limit of determination (0.01-0.02 mg/kg).

The Meeting was informed that a study on the uptake of tebufenozide from soil by crops was in progress and the full report would be available to a future Meeting.

Environmental fate in water/sediment systems

Tebufenozide has a low solubility in water and a relatively high affinity for soils and sediments. It is not readily degraded by either hydrolysis or photolysis. The fate of tebufenozide was studied in two aerobic water/sediment systems and a single anaerobic system. The half-lives were 99-101 days in the aerobic systems and 179 days in the anaerobic.

These studies also confirmed the degradation pathways found in soil, with the ketone and two carboxylic acids as the main products, with further breakdown to carbon dioxide.

The octanol/water partition coefficient of tebufenozide indicates that some bioconcentration in aquatic organisms might occur. In a bioconcentration study with bluegill sunfish it was observed that the residues which accumulated in fish exposed to tebufenozide were depurated with half-lives of less than three days when they were removed from the system.

The analytical methods used in the reported studies involved extraction with methanol/acid or acetone, successive partitioning with hexane and methylene chloride or acetonitrile, and clean-up by column chromatography on basic alumina, Florisil or silica gel. Quantification is by HPLC with UV detection or by GLC with an NPD after methylation of the residues. The GLC method determines tebufenozide and HPLC determines tebufenozide and RH 6595 in the same extract.

The methods were validated with many crops. Recoveries of tebufenozide from fruits were above 80% with an LOD of 0.02 mg/kg by the GLC method, and about 80% with LODs of 0.01-0.02 mg/kg in most crops and 0.05 mg/kg in celery by HPLC. Confirmation by GC-MS or HPLC-MS was reported in some studies.

The storage stability of residues in frozen analytical samples has been studied with apples, apple juice, grapes and wine, rice, walnuts and lettuce. Tebufenozide, RH-6595, and RH-1788 were found to be stable at -20°C in apples for at least 200 days, nuts for 18 months, grapes for at least 12 months, lettuce for 6 months and dry tea for 65 days.

It was reported that studies on apples, walnuts and lettuce would be continued until data from the full 3 years of storage had been obtained.

The results of metabolism studies showed that tebufenozide is the major residue in plants to be found in supervised trials, and the Meeting concluded that the residue should be defined as tebufenozide. The residue is fat-soluble.

Tebufenozide is available as 200 and 240 g/l suspension concentrates, 70 and 10% wettable powders and also as a granulate formulation.

Residue data obtained from trials on about 12 crops in several countries, submitted to national registration authorities to support registered uses or uses pending registration, were evaluated.

Apples. The results of many trials from several countries around the world were reported. Trials in Australia and Greece were not according to GAP and were not considered for the estimation of maximum residue levels. In four US trials according to proposed GAP, residues ranged from 0.36 to 0.61 mg/kg. Residues from treatments complying with GAP were 0.08-1.1 mg/kg from 3-4 applications at 0.24 kg

ai/ha, 14 days PHI in Canada, 0.01-0.18 mg/kg in 4 trials in France and 0.08-0.32 mg/kg in 4 trials in New Zealand (4-8 applications at 0.18 kg ai/ha, 14 days PHI). In two of these trials the residues at days 21 and 22 were taken because they were higher than at days 14 and 15. Residues from Italian GAP (3 applications at 0.288 kg ai/ha, 14 days PHI), were 0.28 to 0.55 mg/kg, and from Japanese GAP (two trials) 0.02 and 0.05 mg/kg. Eight trials in Germany (3 applications at 0.158-0.190 kg ai/ha, 28-day PHI) were evaluated against Belgian GAP and one trial in Spain against French GAP. Metabolites were not determined in any of the trials.

In summary, tebufenozide residues in apples from trials according to GAP were 0.08, 0.11, 0.12, 0.14, 0.16, 0.19, 0.265, 0.37, 0.43, 0.52, 0.75, 0.84 and 1.1 mg/kg in Canada; 0.01, 0.07, 0.14 and 0.18 mg/kg in France; <0.02, 0.08, 0.09, 0.11, 0.16, 0.16, 0.20 and 0.23 mg/kg in Germany; 0.28, 0.52 and 0.55 mg/kg in Italy; 0.02 and 0.05 mg/kg in Japan, and 0.08, 0.10, 0.26 and 0.32 mg/kg in New Zaland. The Meeting estimated a maximum residue level of 1 mg/kg for apples.

Pears. Supervised trials on pears were carried out according to GAP in Italy and New Zealand. The residues in two trials in New Zealand (4-9 applications at >0.18 kg ai/ha, 14 days PHI) were 0.09 and 0.10 mg/kg, and in one trial in Italy (3 applications at 0.216 kg ai/ha, 14 days PHI) 0.23 mg/kg.

Taking into account the similarity of the use patterns in apples and pears, the Meeting agreed to evaluate the combined data on apples and pears as applying to pome fruit.

This gave tebufenozide residues in pome fruit in rank order (median underlined) of 0.01, <0.02, 0.02, 0.05, 0.07, 0.077, 0.08, 0.08, 0.09, 0.09, 0.10, 0.10, 0.11, 0.11, 0.12, 0.14, 0.14, 0.16, 0.16, 0.16, 0.18, 0.19, 0.20, 0.23, 0.23, 0.26, 0.27, 0.28, 0.32, 0.37, 0.37, 0.43, 0.52, 0.52, 0.55, 0.75, 0.84 and 1.1 mg/kg.

The Meeting estimated a maximum residue level of 1 mg/kg and an STMR of 0.16 mg/kg for pome fruits.

Grapes. Numerous field trials on vines have been conducted in Australia, France, Germany, Italy and Thailand. Those in Australia, Italy and Thailand, and several from France, were not according to GAP. Residues in nine trials in Germany in accordance with proposed GAP ranged from 0.21 to 0.42 mg/kg. Nine trials in France complied with GAP (3 applications at 0.144 kg ai/ha, 21 days PHI) and the residues in these in rank order were 0.05, 0.06, 0.07, 0.08, 0.12, 0.18, 0.26, 0.28 and 0.28 mg/kg.

The Meeting estimated a maximum residue level of 0.5 mg/kg and an STMR of 0.12 mg/kg for tebufenozide in grapes.

Kiwifruit. Several field trials were carried out in New Zealand and the USA, but the Meeting could not relate these trials to GAP in New Zealand and was unable to estimate a maximum residue level for kiwifruit.

Leafy vegetables. Several field trials on lettuce were carried out at different locations in the USA according to proposed GAP. Residues in head lettuce ranged from 0.01 to 6.6 mg/kg and in leaf lettuce from 0.4 to 3.2 mg/kg. Head lettuce samples were stored before analysis for 264 to 1186 days.

Six of nine field trials on spinach at different locations in the USA were according to proposed GAP (7 applications at 0.140 kg ai/ha, 7 days PHI). The residues from them ranged from 1 to 4.2 mg/kg.

The storage period before analysis was about 750 days.

Several supervised trials on mustard greens were carried out in the USA. According to the proposed GAP of 7 applications at 0.067-0.135 kg ai/ha with a 7-day PHI. The reported storage period before analysis ranged from 139 to 930 days. The residues were 0.65 to 5.6 mg/kg.

The evaluation of data on leafy vegetables can be reconsidered when GAP has been established and studies on the storage stability of residues in lettuce have been completed.

Celery. Several field trials were conducted in the USA with 7, 8 or 9 applications at 0.14 kg ai/ha. In trials where both stalk and stalk with foliage were analysed, the residues in the stalk with foliage were significantly higher than those in the stalk. The residues ranged from 0.1 to 1.3 mg/kg (1.3 mg/kg in stalk with foliage).

Since there is no existing GAP for celery, the Meeting could not estimate a maximum residue level.

Several supervised trials on cabbage and broccoli were carried out in the USA according to proposed GAP: 7 applications at 0.067-0.135 kg ai/ha and a 7-day PHI. The reported storage period before analysis ranged from 110 to 938 days. The residues in broccoli were 0.1-0.33 mg/kg (3 samples) and in cabbage <0.01-0.53 mg/kg (5 samples). The residues in head cabbage without wrapper leaves were ≤0.01 mg/kg.

The evaluation of data on these crops can be reconsidered when GAP has been established and studies on the storage stability of residues in vegetables have been completed.

Field trials on Chinese kale and chilli peppers in Thailand were reported but they were not according to GAP. The Meeting could not estimate a maximum residue level.

Rice. Eight supervised trials were carried out in Japan according to GAP, with two different formulations (0.75 DL and 10 WP). The residues in the husked grain at 14-21 days PHI ranged from <0.005 to 0.07 mg/kg and in the straw from 2.2 to 7.7 mg/kg. Metabolite residues (sum of alcohol and ketone) were at or below the LOD in the grain and 0.2-0.3 mg/kg in the straw, but increased at longer PHIs to maxima of 0.008 mg/kg in grain and 0.42 mg/kg in straw.

Two trials on rice in Spain were not according to GAP.

The residues in the Japanese trials in rank order were <0.005, 0.007, 0.008, 0.02, 0.03, 0.05, 0.06 and 0.07 mg/kg. The Meeting estimated a maximum residue level of 0.1 mg/kg and an STMR of 0.03 mg/kg for rice, husked.

Pecans. Eight field trials in the USA were all with more applications than the number in the proposed GAP, but four of them were at the proposed application rate. All the residues were below the LOD of 0.01 mg/kg and only one was above the limit of detection (0.003 mg/kg). The Meeting estimated a maximum residue level of 0.01* mg/kg but could not recommend it for use as an MRL because there is no existing GAP.

Walnuts. The highest residue found in nut-meat was 0.05 mg/kg, when the compound was applied at twofold rate (0.560 kg ai/ha). In six field trials in the USA according to GAP (4 applications at 0.28 kg ai/ha, 30 days PHI), the residues ranged from <0.003 to 0.02 mg/kg. In two trials in France considered to

comply with GAP the residues were undetectable (<0.003 mg/kg).

Tebufenozide residues in walnuts in rank order (median underlined) were <0.003, <0.003, <0.003, <0.003, <0.003, 0.006, 0.007, 0.02 and 0.02 mg/kg. The Meeting estimated a maximum residue level of 0.05 mg/kg and an STMR of 0.003 mg/kg for walnuts. A maximum residue level of 0.05 mg/kg, rather than 0.02 mg/kg was estimated, because in two trials the residues were determinable at 0.02 mg/kg.

Tea. Trials were conducted in Japan and Sri Lanka but those in Sri Lanka were not adequately reported. The residues in dry tea from the two Japanese trials according to GAP were 11.5 and 11.6 mg/kg at a 14-day PHI. Residues of the combined alcohol and ketone metabolites determined as the alcohol were 0.23-0.24 mg/kg.

The Meeting concluded that the data were insufficient to estimate a maximum residue level for dry tea.

Processing studies

Apples. Apples were processed to juice, wet pomace and purée according to household procedures. In trials in Germany, apples treated at 2 or 3 times the recommended rate were harvested at 25-28 days and processed to purée. The residue levels of tebufenozide in the purée, washings, and peels and pips were 43%, 1%, and 59% of those in the apples.

In another study, when apples were processed to obtain juice and pomace, the residue levels in the juice were 15% and in the wet pomace 108% of those in the apples.

In a trial in the USA, apples were processed to wet pomace, unpasteurized juice and pasteurized juice in simulated commercial processing. Pasteurized and unpasteurized juice contained almost identical residues of 5% of the residue level in the unwashed fruit. Residues in wet apple pomace were concentrated about fourfold. Washing the treated fruit reduced the residues by about 20%.

The results indicated an apparent concentration of the residues from whole apples to wet pomace of 1-4 times, with an average of 2.5 times, and a reduction of residues in purée and juice by factors of 4 and 8 respectively. Pasteurization did not change the residue level in the juice.

The Meeting estimated STMR-Ps of 0.4 mg/kg for wet apple pomace and 0.02 mg/kg for apple juice.

Grapes. Trials were carried out in France, Germany and Italy to study the fate of tebufenozide in processed products. Residues in wine decreased 2-13-fold, with an average factor of 4, while pomace showed a concentration of the residue by factors of 1.6-3.7, with an average of 2.7, although only three results were available.

The Meeting estimated STMR-Ps of 0.36 mg/kg for wet grape pomace and 0.03 mg/kg for wine.

Tea. Twelve to 30% of the tebufenozide found in dry tea was transfered to the tea infusion.

Animal feeding studies. In a study in Japan two cows were dosed by capsule with 40 or 400 mg/day of tebufenozide for 7 days. The dose levels corresponded to 0.07 and 0.74 mg/kg bw/day. Milk samples

showed no detectable residues of tebufenozide. The LOD was 0.02 mg/kg.

Because of the short duration of the study and because the dose levels were not confirmed by analysis only milk was analysed, the Meeting could not estimate a maximum residue level.

No poultry feeding studies were reported to the Meeting, but the results of the metabolism study in hens demonstrated that transfer of the residues from the diet to the tissues or eggs is low. As the study was for only 7 days and plateau level could not be reached, the Meeting concluded that it was not an inadequate basis for the estimation of maximum residue levels.

Of the studied crops, the main animal feed component contributing to the diet of beef or dairy cattle is apple pomace. On the basis of the maximum residue level estimated for pome fruit (1 mg/kg), and the mean concentration factor from fruit to pomace of 2.5, the maximum theoretical dietary intake of tebufenozide can be estimated as 2.5 mg/kg for beef cattle and 1.3 mg/kg for dairy cows.

No data were provided on the residues in the edible portions of food commodities apart from those included in the supervised trials or processing studies.

No information was provided on the residues of tebufenozide occurring in commerce or at consumption.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the maximum residue levels listed below are suitable for establishing MRLs. The supervised trials median residues (STMRs) are suitable for use in estimations of dietary intake.

Definition of the residue for compliance with MRLs and for the estimation of dietary intake: tebufenozide

The residue is fat-soluble.

COMMODITY		MRL, mg/kg	PHI	STMR/ STMR-P, mg/kg
CCN	NAME			
FP 0009	Pome fruit	1	14	0.16
FB 0269	Grapes	0.5	21	0.12
TN 0678	Walnut	0.05	30	0.003
CM 0649	Rice, husked	0.1	14-21	0.03
	Apple pomace, wet			0.4
	Apple juice			0.02
	Apple purée			0.04
	Grape pomace, wet			0.36
	Wine			0.03

FURTHER WORK OR INFORMATION

Desirable

1. Information on tebufenozide residues in raisins, raisin culls and rice hulls.

2. Information on residues of tebufenozide in foods in commerce or at consumption.

3. A transfer study on poultry.

4. The results of a cow-feeding study which the Meeting was informed was in progress.

5. Data on residues in paddy rice and on the stability of residues in analytical samples of rice stored for longer periods than the 20-21 days already reported.

6. A detailed report of the completed study of uptake by rotational crops that the Meeting was informed was available.

7. Representative data on the storage stability of residues on leafy vegetables for the full duration of the studies that the Meeting was informed are in progress.

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Yajima, K. 1992. Dissipation (and Metabolism) of RH-5992 in Upland Soil. Rohm and Haas Report No. 34-92-81. Unpublished.

TEFLUBENZURON (190)

IDENTITY

ISO common name: teflubenzuron

Chemical name:

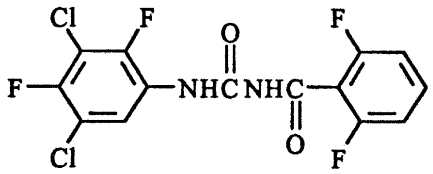
IUPAC: 1-(3,5-dichloro-2,4-difluorophenyl)-3-(2,6-difluorobenzoyl)urea

CA: *N*-[[(3,5-dichloro-2,4-difluorophenyl)amino]carbonyl]-2,6-difluorobenzamide

CAS No: 83121-18-0

Synonyms: DART; DIARACT; NEMOLT; NOMOLT; CL 291,898; SAG 134

Structural formula:



Empirical formula: $C_{14}H_6Cl_2F_4N_2O_2$

Molecular weight: 381.1

Physical and chemical properties

Technical material

Purity: 97 ± 0.72% (w/w)

State: crystalline solid

Colour: white to yellowish

Odour: practically odourless

Vapour pressure: 8×10^{-10} Pa (20°C)
3×10^{-10} Pa (40°C) (Celamerck, 1985a)
1.3×10^{-8} Pa (25°C) (Harteveld and Jager, 1988)

Melting Point: 222.5°C (Pesticide Manual, 1994)

Octanol/water partition coefficient: log P_{OW} = 4.56 (Darskus, 1982)

Solubility (g/l at 20°C):

hexane	0.05	ethanol	1.4
methanol	0.6	dichloromethane	1.8
n-octanol	0.7	acetone	10
2-propanol	0.8	dioxane	19
toluene	0.9	cyclohexanone	24
acetonitrile	1.1	dimethylformamide	170
water	2×10^{-5}		(Cardinaals, 1989; Pesticide Manual, 1994)

Hydrolytic stability: half-life at pH 5-7: 30 days (25°C), 5 days (50°C)
half-life at pH 9: 10 days (25°C), 4 h (50°C)

Photolytic stability: in aqueous solution teflubenzuron shows a half-life of approximately 10 days

Storage stability : minimum 2 years under normal conditions (Pesticide Manual, 1994)

Formulations

Teflubenzuron is available as suspension concentrates containing 150 g ai/l (NOMOLT, NEMOLT, and DART SC 15) or 50 g ai/1 (NOMOLT and DART SC 5).

METABOLISM AND ENVIRONMENTAL FATE

Animal metabolism

The biokinetics and metabolism of teflubenzuron were studied in rats, lactating goats and laying hens.

Rats. The Absorption, distribution, excretion and metabolism of teflubenzuron in the rat were studied by Schlüter (1984, 1985a, 1986a). [^{14}C]teflubenzuron labelled in the aniline ring was administered by oral gavage.

Experiments were performed at dose levels of 25 and 750 mg ai/kg body weight (Schlüter, 1984). The test substance was applied as a suspension in a 1:1 mixture of 1% Tylose C 30 (methylcellulose) and 1% Tween 80 (polyoxyethylene sorbitan mono-oleate) ensuring a high concentration and stable suspension of teflubenzuron. The low dose corresponded approximately to a no-effect level and the high dose was the highest that could be applied without gavage difficulties. The test substance was administered to 6 groups of rats. The general design of the study is shown in Table 1.

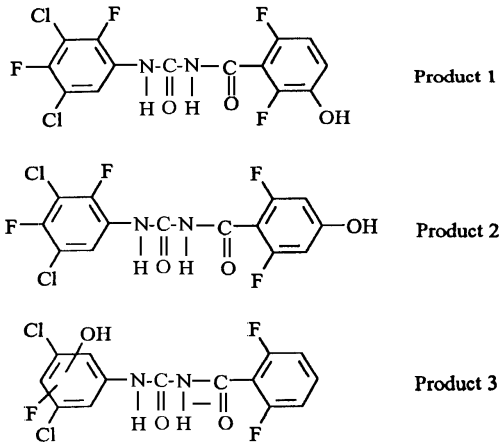
Table 1. Metabolism of teflubenzuron in rats, study design (Schlüter, 1984).

Group	Number of rats	Administration and type of study
A	1 male, 1 female	single low dose, analysis of expired air
B	5 males, 5 females	single low dose, excretion and metabolism
C	5 males, 5 females	repeated low dose, excretion and metabolism
D	5 males, 5 females	single high dose, excretion and metabolism
E	5 males, 5 females	single low dose, blood level
F	5 males, 5 females	single high dose, blood level

The results revealed that teflubenzuron was excreted completely within a short time with no differences between the sexes and no differences due to the type of exposure. The main route of elimination was in the faeces with more than 85% of the dose being eliminated within 24 h. The main component in the faeces was the unchanged parent compound (90 and 96% of the faecal residue from the low and high dose respectively), indicating low absorption by the gastrointestinal tract. Traces of metabolites (up to at least 15, mostly polar) were also found. One of them was identified as 3,5-dichloro-2,4-difluorophenylurea. The remainder of the total dose (10% and 4% of the low and high doses respectively) was absorbed and subsequently small quantities were excreted in the bile and urine. The amounts in the urine were very low: 0.5-0.9% and 0.1-0.2% of the low and high doses respectively, consisting of several polar products.

Another study (Schlüter, 1985a) revealed three metabolites in the urine. Two compounds (products 1 and 2) were structural isomers formed by hydroxylation of the benzoyl ring at positions 3 and 4 (Figure 1). The third compound was formed by replacement of one fluorine atom of the aniline ring by hydroxyl.

Figure 1. Metabolites of teflubenzuron in rat urine (Schlüter, 1985a).



The low absorption of teflubenzuron from the gastrointestinal tract of the rat was confirmed by its low levels in the plasma. The administration of 25 mg/kg body weight gave less than 0.5 µg equivalents of parent compound/ml plasma. After treatment with 750 mg/kg body weight the plasma concentrations amounted to 1-3 µg/ml (Schlüter, 1986a).

The examination of organs and tissues showed that even after 7 days administration by gavage the compound is rapidly and completely eliminated. Two days after the last treatment, no residues exceeding 0.05% of the applied radioactivity were found in any organ or tissue except the liver (0.1-0.2%). Five days after treatment the ^{14}C residues in all tissues and organs (including the gastrointestinal tract) were below 0.01 %, except in the liver which contained 0.05%.

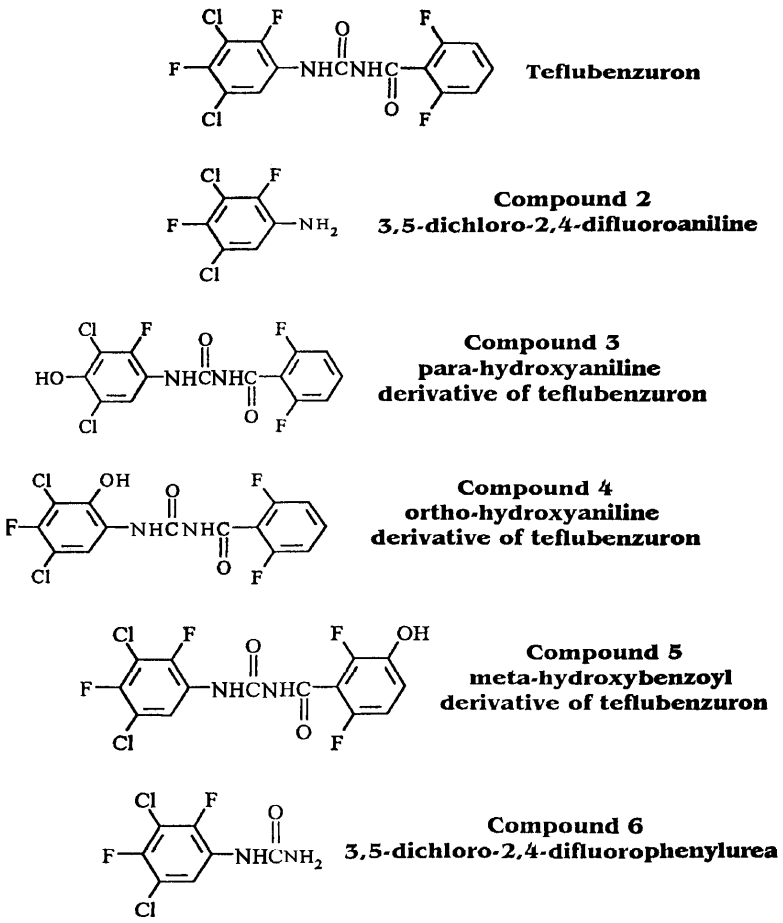
No ^{14}C was detected in expired air, indicating that the aniline ring was metabolically stable. Less than 1% of the radioactivity was detected in the carcases of the animals at the end of the study.

The metabolism and biokinetics of teflubenzuron can be characterized as showing poor absorption from the gastrointestinal tract and rapid elimination, mainly in the faeces and largely as the unchanged parent molecule with no accumulation in any organ or tissue, the sum of all metabolites accounting for less than 1% of the total radioactivity (Schlüter, 1984, 1985a, 1986a).

Nendza (1991) considered that phenylurea derivatives, including teflubenzuron, are rapidly metabolized, e.g. by demethylation and hydroxylation, and hence do not accumulate in mammalian tissues.

The biliary excretion and metabolism of aniline ring-labelled [^{14}C]teflubenzuron was studied in two groups of 6 rats (3 males and 3 females) with bile-duct canulae by Hawkins and Mayo (1988a). After the oral administration of [^{14}C]teflubenzuron at 25 mg/kg, about 16% and 1% of the dose was excreted in 48 h in the bile and urine respectively. After dosing with 750 mg/kg the corresponding figures were about 2% and 0.4%. Summation of the radioactivity in the urine, bile and liver indicated a total absorption of about 18% and 2% of the dose after administration at the 25 and 750 mg/kg levels respectively. It is evident that absorption is dose-dependent. Virtually all the absorbed teflubenzuron has been shown to be transformed. A radioactive component which yielded material which co-chromatographed with compound 5 (Figure 2) after hydrolysis indicated that one biotransformation pathway was meta-hydroxylation of the benzoyl ring and conjugation. Only very small amounts of radioactivity were co-eluted with the hydroxylated aniline derivatives of teflubenzuron (compounds 3 and 4). A radioactive component associated with 3,5-dichloro-2,4-difluorophenylurea (compound 6) confirmed that scission of the benzoyl-urea bond was an additional degradation pathway. Hydrolytic treatments of bile indicated that this phenylurea may also be present as a sulfate conjugate.

The Figure 2. Reference compounds used in the teflubenzuron bile-duct cannulation study (Hawkins and Mayo, 1988a).



biotransformation products in bile and urine included much unidentified polar material. The proportion of this material decreased only slightly after various enzyme treatments, to produce the *m*-hydroxybenzoyl derivative of teflubenzuron and the dichlorodifluorophenylurea. Acid and alkaline hydrolysis decreased the proportion of this polar material further to produce some unidentified products. There was no appreciable difference between the high-level and low-level doses in the proportions of radioactive components produced in bile or the effects of various hydrolytic treatments.

Lactating goats. Cameron *et al.*(1987a) carried out a study to determine the rates and routes of excretion of orally administered [^{14}C]teflubenzuron uniformly labelled in the aniline ring in two lactating goats and to quantify and identify the radioactive metabolites in the milk, plasma, urine, faeces, bile, organs and tissues. The nature of the radioactivity in the faeces and bile was also investigated. The goats were dosed orally twice daily for 7.5 days at a level of 7 mg/kg body weight/day.

The main route of elimination of radioactivity was in the faeces, accounting for 99% of the total administered dose (including intestinal contents at post-mortem). The major radioactive component in goat faeces had identical HPLC and TLC retention characteristics to teflubenzuron and accounted for 76.9% of the radioactivity. A minor radioactive component with similar retention characteristics to compound 5 in Figure 2 accounted for 3.6% of the radioactivity, and a second unknown minor component for 5.9%.

The levels of total radioactivity in the plasma following the first dose remained at or close to the limit of detection. During the dosing period they increased to a maximum of 8-10 ng teflubenzuron equivalent/ml by day 4.

The levels of total radioactivity in the milk were similar to those in the plasma at the same times. The highest levels were found in the day 5 evening milk (10-15 ng equivalent/ml) and represented 0.002-0.005% of the cumulative administered dose up to that time. The radioactivity in the milk accounted for 0.03% of the total administered dose. Cameron *et al.* (1989) showed that the

radioactive residues had the chromatographic characteristics of teflubenzuron.

The radioactive residues in all organs, tissues and body fluids examined post mortem were low in relation to the total dose. The highest mean levels in organs were in the liver and lung with 486 ng equivalent/g and 136 ng equivalent/g respectively, which corresponded to 0.14% and 0.02% of the total administered dose in the whole organs. Relatively high levels were also detected in bile (mean level 1306 ng equivalent/ml, 0.002% of the total administered dose). The levels of radioactivity in the liver and bile indicate biliary excretion as being important in the elimination of the absorbed fraction of an orally administered dose. The absence of similar levels in the plasma suggests that much of the absorbed radioactivity is removed by 'first-pass metabolism' in the liver.

The radioactivity in the bile was mainly in ß-glucuronide (or possibly sulfate) conjugates. When these were hydrolysed the main product had similar chromatographic characteristics to 1-(3,5-dichloro-2,4-difluorophenyl)-3-(2,6-difluoro-3-hydroxybenzoyl)urea (compound 5 in Figure 2). No unchanged teflubenzuron was found in bile either before or after enzymic hydrolysis.

The levels of radioactivity in all other organs, tissues and body fluids were generally less than 100 ng equivalent/g. Teflubenzuron was, therefore, shown to be poorly absorbed after oral administration: the absorbed fraction appears to be metabolized in the liver and conjugated before elimination, mainly in the bile.

Cameron *et al.* (1989) examined the nature of the radioactivity in extracts of the liver. The major component was a polar compound which was not identical to any of the reference compounds. Traces of material co-chromatographing with 1-(3,5-dichloro-2,4-difluorphenyl)-3-(2,6-difluoro-3-hydroxybenzoyl)urea (compound 5 in Figure 2) were also detected. The distribution of the radioactivity was unaffected by treatment with deconjugating enzymes, indicating that the polar material was not a glucuronide or sulfate conjugate.

None of the extracts contained any radioactive components with similar characteristics to either 3,5-dichloro-2,4-difluoroaniline or 3,5-dichloro-2,4-difluorophenylurea (compounds 2 and 6 in Figure 2).

Laying hens. Cameron *et al.* (1987b) investigated the disposition of teflubenzuron in laying hens and the levels and identity of the radioactive compounds in the plasma, bile, organs, tissues and eggs. [^{14}C]teflubenzuron uniformly labelled in the aniline ring was administered orally to 3 groups of 6 laying hens twice daily for 7.5 days at a level of 1.25 mg/kg/day.

The administered radioactivity was almost quantitatively recovered from the excreta (mean recovery 95.6%). The low levels of radioactivity found in plasma, eggs and post-mortem tissue samples suggest that teflubenzuron is only poorly absorbed after oral administration to hens. The absorbed radioactivity was readily eliminated from the eggs and plasma when dosing stopped (half-life of elimination 1.5-2 days).

The main radioactive component in extracts of the excreta had identical HPLC retention characteristics to teflubenzuron.

The levels of radioactivity detected in the liver and bile indicate biliary excretion as being important in the elimination of the absorbed fraction of orally administered doses. The radioactivity in the bile was again mainly in â-glucuronide (or possibly sulfate) conjugates which yielded a product with the chromatographic characteristics of 1-(3,5-dichloro-2,4-difluoropheny1)-3-(2,6-difluoro-3-

hydroxybenzoyl)urea (compound 5 in Figure 2) on hydrolysis. No unchanged teflubenzuron was found in bile either before or after enzymic deconjugation.

A radioactive component with identical retention characteristics to teflubenzuron was found in the plasma, fat, liver and egg yolk. One or possibly two very polar radioactive components were also observed in the plasma and liver which had similar retention characteristics to the main constituent of untreated bile.

In summary teflubenzuron was found to be poorly absorbed after oral administration with the absorbed fraction passing into body tissues, especially fatty tissues and egg yolk. It was readily eliminated when dosing was stopped. The absorbed fraction is apparently metabolized in the liver and conjugated before elimination, mainly in the bile. The main route of metabolism of teflubenzuron in the liver seems to be by hydroxylation of the difluorobenzoyl ring followed by conjugation of the hydroxyl group to form a â-glucuronide.

An additional study with similarly labelled teflubenzuron was carried out to identify the radioactive components in hen excreta, liver, kidney, egg yolk, and fat by HPLC and TLC (Cameron *et al*., 1988).

Liver extracts were shown to contain two components, one with characteristics identical to teflubenzuron and one very polar compound which could not be identified. No significant differences were noted following treatment with deconjugating enzymes, indicating that this polar species was not a conjugate. No compounds were found with the chromatographic characteristics of 3,5-dichloro-2,4-difluoroaniline or 3,5-dichloro-2,4-difluorphenylurea (compounds 2 and 6 in Figure 2).

Kidney extracts were shown to contain both species detected in liver extracts and also traces of material which co-chromatographed with 3,5-dichloro-2,4-difluorphenylurea.

Most of the radioactivity in yolk extracts and all of it in fat extracts co-chromatographed with teflubenzuron.

Plant metabolism

The metabolism of teflubenzuron was investigated in apples, potatoes, cotton and spinach.

Apples. Schlüter (1987a) treated selected parts of young apple trees three times with formulated [U-*aniline*-^{14}C]teflubenzuron according to the spraying schedule to be applied in agricultural practice: treatments at 3-week intervals with a 3-6 week PHI. The surfaces of the fruits and leaves were covered with droplets of the application mixture. The concentration of the active ingredient was 0.2 mg/ml (two to four times that used in agricultural practice, 0.05-0.1 mg/ml). Treated apples were sampled at intervals as well as at normal harvest, and treated and untreated leaves and untreated apples only at harvest. The radioactive residues in treated leaves and apples consisted almost exclusively of unchanged teflubenzuron: 99% and 98% respectively.

It was concluded that teflubenzuron does not penetrate into the fruits or leaves if it is sprayed on apple trees, with no systemic transport and no metabolism within the plants.

Potatoes. Schlüter (l987b) studied metabolism and kinetics of [^{14}C]teflubenzuron (uniformly labelled in the aniline ring) in potato plants. The tops of the plants as well as the soil surfaces of a potato plot were separately treated four times according to the spraying schedule to be used in agricultural

practice (treatments at about 2-week intervals, the last treatment at the beginning of flowering). Some of the plants were treated by spraying the leaves only (the soil was covered with plastic foil and cellulose tissues), and in part of the plot the application mixture was applied to the soil with no leaf treatment. The application rate of the active ingredient was 90 g ai/ha (3 times the highest rate recommended in practice of 10-30 g ai/ha). Treated and untreated tubers and tops were sampled at the growing stage and at normal harvest.

At the end of the test period (63 days after the first treatment), 99.8% of the total radioactivity was extractable from the treated leaves and could be identified as the unchanged parent compound. No significant amounts of radioactive residues were found in the tubers of leaf-treated plants, but trace amounts were detectable in the tubers sampled after soil treatment. After peeling the tubers, mean residues of 0.009 mg/kg were found in the peel, which corresponded to 0.03% of the total radioactivity applied. A total residue of 0.002 mg/kg in unpeeled tubers can be calculated.

It was concluded that teflubenzuron does not penetrate into leaves or stems if it is sprayed on to the aerial parts of potato plants. No translocation or metabolism occurs in the plants.

Cotton. Cotton plants (*Gossypium hirsutum*) were repeatedly treated with formulated teflubenzuron according to the recommendations. Six unlabelled treatments at 81 g ai/ha were followed by two applications of 156 g ai/ha of formulated [U-*aniline*-^{14}C]teflubenzuron (Schwalbe-Fehl *et al.*, 1986). Various plant parts and soil were sampled periodically until harvest. The concentrations of ^{14}C in the samples at harvest were as follows.

Soil: 0.05 µg ai equivalent/g air dried soil (0-5 cm)
Leaves: 6.4 µg ai equivalent/g fresh sample
Stems: 0.5 µg ai equivalent/g fresh sample
Capsule walls: 0.4-0.9 µg ai equivalent/g fresh sample
Seed hairs: 0.02-1.5 µg ai equivalent/g fresh sample
Seeds: 0.005-0.011 µg ai equivalent/g fresh sample

Significant residues were found only in those plant parts which were directly hit by the radiolabelled spray. The test substance was not translocated to closed cotton fruits or seeds. The radioactive residues were almost quantitatively extractable (93-98% of the total radioactivity). More than 99% of the extractable radioactivity was from the unchanged parent compound.

Spinach. Spinach plants were subjected to one run-off spray treatment with 20 ml of a 0.18% spray-wash containing 247 mg/kg [^{14}C]teflubenzuron, which corresponds to an application rate of 60 g ai/ha (Schlüter, 1985b).

The treated plants showed 6.9, 1.01, and 0.7 mg/kg total radioactive residues at 0, 8 and 15 days after treatment respectively. Almost all the residue could be removed from the plants by a surface rinse at all sampling times (at day 0 99%, day 8 99.1%, and day 15 99.2% of the corresponding total radioactive residue).

The remaining 0.8-1.0% was extractable after homogenization of the plants. TLC showed that most of the radioactive residue consisted of unchanged teflubenzuron, which amounted to 94.8% of the total radioactive residue on day 0, 91.7% on day 8 and 77.1% on day 15.

The sum of the degradation products reached 22.9% at day 15. No major product was found. The fact that the radioactivity was almost completely removed by surface rinsing indicates the

absence of metabolic degradation. It is more likely that teflubenzuron is photolytically degraded when applied to the plant surface.

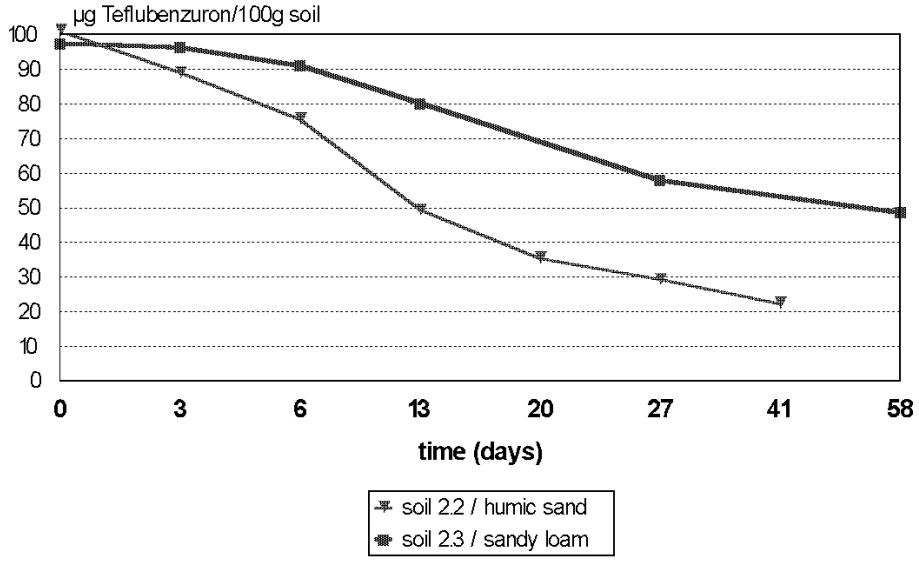
Environmental fate in soil

Laboratory studies.

Degradation. The degradation of unlabelled teflubenzuron in two different types of soil was studied by Heupt (1984). Analytical grade teflubenzuron was added at a starting concentration of 100 µg per 100 g soil (1 mg/kg).

The results showed a big difference in the rates of degradation in the different soil types. In humic soil (humic sand) degradation was more rapid than in sandy loam soil which showed less microbiological activity. The results emphasize that the microbiological factor is of primary importance. The course of the degradation curves (Figure 3) confirm this microbial metabolism. Both curves after an initial linear course show a distinct break after 3 weeks in humic soil and 4 weeks in loam soil. Thereafter they are approximately linear again with a reduced gradient. This effect is probably determined by a change of microbial activity, owing to a partial 'intoxication' of some micro-organisms involved in the degradation by the metabolites formed. The half-life of teflubenzuron in humic sand soil was 2 weeks and in sandy loam soil 6 weeks.

Figure 3. Degradation of teflubenzuron in soil (Heupt, 1984).



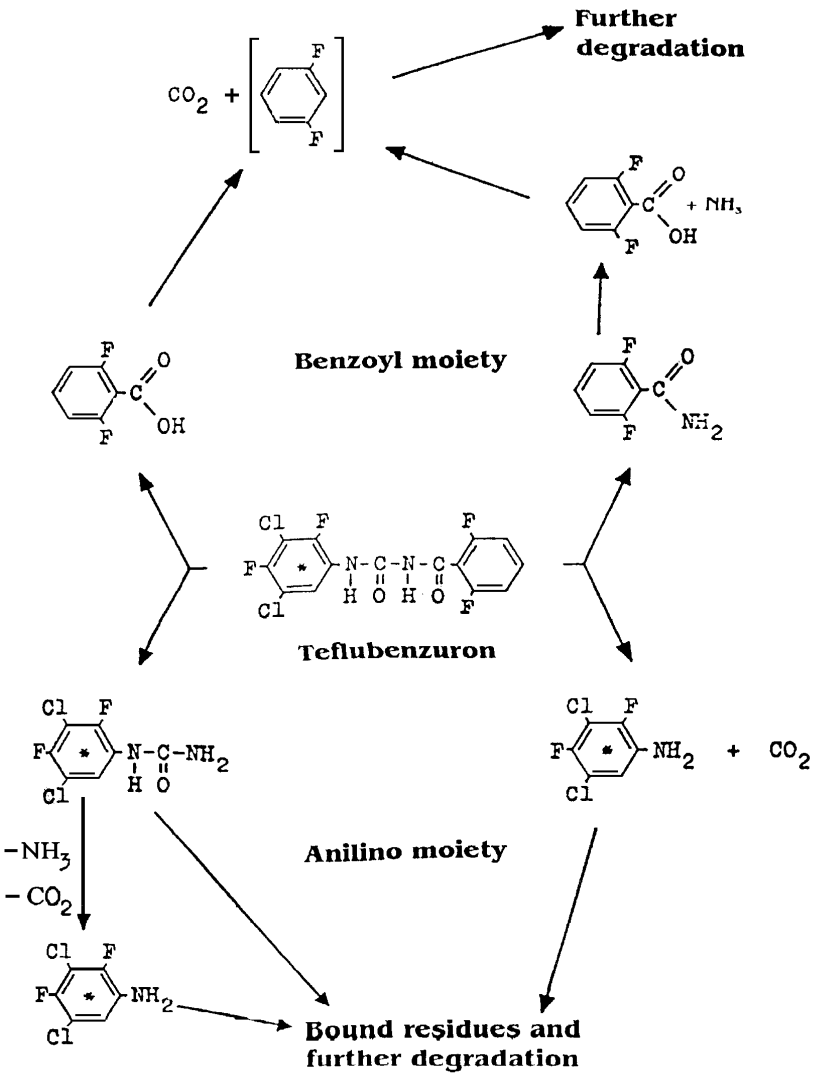
The aerobic and anaerobic degradation of teflubenzuron in a sandy loam soil (5 mg/kg) was studied by Schlüter (1985c). The compound was uniformly labelled with ^{14}C in the aniline ring. The results show that teflubenzuron is degraded in a sandy loam soil under anaerobic conditions about six

times as rapidly as under aerobic. This is confirmed by the fact that the sample taken about 1 h after treatment in the anaerobic test already showed considerable degradation (about 14%) of the parent compound whereas the aerobic test showed about 3% degradation at day 0. Under practical conditions in the field, degradation is likely to be considerably higher than under aerobic conditions in the laboratory because anaerobic degradation is involved.

There is no fundamental difference in the initial degradation pathway under anaerobic and aerobic conditions (cleavage of the parent compound and formation of unextractable residues). In both cases 3,5-dichloro-2,4-difluorophenylurea and 3,5-dichloro-2,4-difluoroaniline were found, indicating that the initial steps in the degradation route were not affected by the difference in conditions. This is consistent with the previous results which demonstrated that 3,5-dichloro-2,4-difluorophenylurea was the major product under both aerobic and anaerobic conditions.

A proposed pathway for the degradation of teflubenzuron in soil under aerobic and anaerobic conditions is shown in Figure 4.

Figure4. Proposed pathway of teflubenzuron degradation in soil under aerobic and anaerobic conditions (Schlüter, 1985c).



In an additional 150-day study the aerobic and anaerobic degradation of teflubenzuron in silty clay loam (0.5 mg/kg) was investigated by Croucher and Edwards (1990), using the benzoyl-^{14}C compound. Soil samples were incubated under aerobic conditions for 30 days, when conditions in some of the samples were made anaerobic by flooding with water and purging with nitrogen. Soils incubated in both aerobic and anaerobic conditions were analysed 60 days and 90 days after treatment.

The degradation of teflubenzuron in aerobic soil occurred at a moderately rapid rate with a half-life (DT-50) of 29 days and a DT-90 of 108 days. After the change to anaerobic conditions the rate of $^{14}CO_2$ evolution slowed considerably but the overall rate of degradation did not change, indicating that the initial steps in the degradation route were not affected by the change in conditions.

The proportions of the radioactive products recovered after 90 days from the anaerobic and aerobic soils were different. Only 3% of the applied radioactivity was evolved as $^{14}CO_2$ in the anaerobic phase of the experiment compared with 24% from the aerobic soils over the same time period. The bound residue was higher in the anaerobic than the aerobic soil.

The major extractable radioactive component at all sampling times under both aerobic and anaerobic conditions was teflubenzuron, but at least 6 other components were observed in extracts of 60-day anaerobic soil accounting for a total of 7% of the applied radioactivity. The radioactive

product expected from the initial cleavage of the [*benzoyl*-^{14}C]teflubenzuron was 2,6-difluorobenzoic acid. It was not observed under aerobic conditions, which indicated that its rate of depletion exceeded its rate of formation, but trace amounts were identified in the anaerobic soil where the rate of mineralization of the benzoyl ring was slower. Another minor degradation product identified under anaerobic conditions was formed by replacement of fluorine by hydroxyl in the 4-position of the aniline ring. This may be an aerobic transformation which was detected because of the slower rate of subsequent catabolism under anaerobic conditions. Alternatively reductive defluorination is a possible first step, followed by replacement of hydrogen by hydroxyl. Decarboxylation of 2,6-difluorobenzoic acid would also be expected. This provides good evidence for the mineralization of the bound residues under aerobic conditions.

Hawkins *et al.* (l987) studied the photodegradation of [^{14}C]teflubenzuron, uniformly labelled in the aniline ring, applied to thin layers of soil on glass plates at a rate of 30 mg/m^2 and irradiated with artificial sunlight from a xenon arc lamp for periods up to a maximum of 15 days. The compound was also applied to control plates which were maintained in the dark under similar conditions of temperature and ventilation.

On exposed plates, teflubenzuron was degraded with a half-life of about 104 days. The major degradation product was volatile and accounted for about 7.2% of the applied radioactivity after 15 days exposure; it was probably $^{14}CO_2$. Most of the radioactivity remaining in the soil was extractable into acetone and chromatographed with teflubenzuron, although after 15 days exposure 7.5-11.9% of the applied radioactivity remained bound to the soil. 3,5-Dichloro-2,4-difluorophenylurea was a minor degradation product (about 2% of the applied radioactivity) after 15 days exposure. No volatile labelled products were formed on the control plates. Apparently photodegradation has relatively little significance in the degradation of teflubenzuron in soil.

Leaching. The fate and behaviour of compounds in soil depends on the extent to which they are leached. The leaching of unlabelled teflubenzuron was studied in three types of soil: (i) sand with a low humus content, (ii) loam sand with a high humus content, and (iii) sandy loam with low humus (Celamerck, 1980). The application rate was 0.6 kg ai/ha (0.12 mg/column) and the columns were leached with a simulated rainfall of about 200 mm during two days. The leachates were extracted with benzene and analysed by HPLC. The LOD was 5 µg/l. Teflubenzuron was not detectable in the drainage water of any of the three soils.

In a more recent study (Schlüter, 1986b) a sandy loam soil was treated with [^{14}C]teflubenzuron uniformly labelled in the aniline ring at 0.5 mg/kg and aged for 30 days under aerobic conditions. After ageing 90.7% of the applied radioactivity could be extracted with solvents of increasing polarity. The radiolabelled material consisted of teflubenzuron (75.5% of the applied activity), 3,5-dichloro-2,4-difluoroaniline (2.1%), 3,5-dichloro-2,4-difluorophenylurea (5.6%), and traces of various unidentified products (7.45% in total).

Aged soil samples each equivalent to 100 g of air-dried soil were placed on top of a 30 cm segmented leaching column of untreated soil to which 11 ml distilled water was added each day for 45 days. After this simulated irrigation most of the applied radioactivity was found in the originally treated soil (80.5% of the applied activity) and the first 5 cm column segment (13.3%). In these segments teflubenzuron was still the main residue and was accompanied by the two compounds identified in the aged soil before the start of the irrigation period.

From the first 5 cm segment only 0.03% of the applied radioactivity could be found in the

aqueous effluent obtained during the 45 days period. Neither teflubenzuron nor its identified degradation products could be found in the effluent, and all three compounds show almost no tendency to migrate into deeper soil layers. The contamination of ground water by these compounds is extremely improbable in agricultural practice.

The lack of leaching is related to high adsorption of teflubenzuron by soil as is evident from the next study.

Adsorption/desorption in soil. The adsorption/desorption of teflubenzuron in soil was studied by Schlüter (1986c) in sand, sandy loam, silt loam and clay loam using a solution of the compound in 0.01 M $CaCl_2$ at a level of about one-half saturation (9.46 µg/l). After 6 hours the concentration in the supernatant solutions in contact with sand, sandy loam, silt loam and clay loam had decreased to 0.24, 0.09, 0.07, and 0.05 µg/l respectively. The concentration of the solution without soil was still 7.94 µg/l.

From these results it was calculated that the proportions of teflubenzuron adsorbed after the 6-h treatment period were 96.9% from sand, 98.8% from sandy loam, 99.1% from silt loam and 99.4% from clay loam, showing very strong adsorption of the compound to all the soils tested.

Desorption tests with the treated soil samples revealed that 6.1%, 3.7%, 1.2%, and 1.3% of the adsorbed radioactivity could be desorbed again from sand, sandy loam, silt loam and clay loam soils respectively during two desorption periods of 24 h each. It is very improbable that these trace amounts still consist only of the unchanged parent compound, since the results of the degradation studies under aerobic and anaerobic conditions indicate appreciable degradation after a corresponding period of time.

It was concluded that teflubenzuron itself shows practically no tendency to migrate once it is applied to soil. This is attributable to the very low solubility of the compound in water, and strong adsorption with very little leaching in all types of soil tested.

Rotational crops

In a study by Schlüter (1989) on rotational crops with [^{14}C]teflubenzuron uniformly labelled in the aniline ring, soil was treated with 0.5 kg ai/ha and aged under anaerobic conditions. At 30, 120 and 360 days after the soil treatment, head lettuce, carrots and wheat were planted or sown as rotational crops and grown to maturity. The ^{14}C residues, as mg/kg teflubenzuron equivalents, are shown in Table 2.

Table 2. ^{14}C levels in rotational crops after treatment with [^{14}C]teflubenzuron, expressed as mg/kg teflubenzuron equivalents (Schlüter, 1989).

Crop	^{14}C as teflubenzuron, mg/kg		
	Soil aged 30 days	Soil aged 120 days	Soil aged 360 days
head lettuce	0.007	0.006	0.002
carrots, peeled	0.013	0.006	0.002
peel	0.08	0.053	0.017
whole root	0.026	0.013	0.005
wheat straw	0.24	0.088	0.035
wheat grain	0.005	0.003	0.002

The comparatively high residue in wheat straw is partly due to the high degree of dryness of the material at harvest. Relatively high values were also found in carrots; the cause in this case is the direct contact with the treated soil. Peeled carrots, and all other fresh plant material, contained equivalents of ≤0.01 mg/kg. The results further show that, in all samples, the radioactive residues decrease significantly with increasing soil ageing periods. This decrease corresponds to the observed decrease in extractable radioactivity in soil on increasing the ageing period. Unextractable soil residues, on the other hand, increase markedly throughout the entire experimental period (up to 70% of the radioactivity originally applied). These observations indicate that the unextractable residues in soil are very probably not taken up by the rotational crops and that the radioactive residues found in the plants originate only from the extractable soil residues. Since at all times the concentration of the unchanged parent compound in the extractable soil residues is much higher than those of the individual degradation products it is probable that teflubenzuron itself is taken up by the plants.

In so far as the low residues made analysis possible, it could be shown that the plants contained numerous, mainly polar, compounds in very small amounts (≤0.05 mg/kg). Neither teflubenzuron nor its known soil degradation products (3,5-dichloro-2,4-difluorophenylurea and 3,5-dichloro-2,4-difluoroaniline) could be detected in the plants (<0.01 mg/kg).

Environmental fate in water and water/sediment systems

Laboratory studies

Hydrolysis. Teflubenzuron (unlabelled) was incubated with buffers at pH 5, 7 and 9 at room temperature. It was stable at pH 5 but was hydrolysed at pH 7 and 9 with half-lives of 8 months and 8 days respectively. In additional studies with suspensions the products identified by HPLC were 2,6-difluorobenzoic acid, 2,6-difluorobenzamide, and 3,5-dichloro-2,4-difluoroaniline, showing that all three N-CO bonds are cleaved when teflubenzuron is hydrolysed (Hawkins *et al.*, 1988b; Heupt, 1983).

Photolysis. The photodegradation of [^{14}C]teflubenzuron in aqueous solution has been investigated by Hawkins *et al.* (1988c). [^{14}C]teflubenzuron (uniformly labelled in the aniline ring) in ethanol was added to acetate buffer (0.1 M, pH 5) to a concentration 0.1 mg/l (10% ethanol) and irradiated with artificial sunlight from a xenon arc lamp for periods up 15 days. Control samples were maintained in the dark under similar conditions of temperature and ventilation.

In the exposed solutions, teflubenzuron was degraded with a half-life of about 10 days. Only one breakdown product exceeded 10% of the applied radioactivity, accounting for about 32% after 15 days, when teflubenzuron accounted for about 45%. About 5% of the applied radioactivity was recorded as volatile breakdown products. No volatile products were formed in the control incubations and after 15 days most of the applied radioactivity chromatographed with teflubenzuron.

The main product of photolysis chromatographed with the reference compound 1-(3,5-dichloro-2,4-difluorophenyl)-5-fluoro-3*H*-dihydroquinazoline-2,4-dione. The degradation product was isolated and its identification confirmed by mass spectrometry.

Sunlight has little effect on the aqueous stability of teflubenzuron. It may be adsorbed onto organic matter, thus reducing its concentration in field water. In highly polluted water, the chemical appeared to be lost because of microbial action as well as adsorption onto organic matter (Schaefer *et*

al., 1988).

Water/sediment systems. The biodegradation of [^{14}C]teflubenzuron labelled in the aniline ring was determined in two water/sediment systems in The Netherlands (Muttzall, 1987). Ditch water and one sediment sample were collected from an unpolluted ditch surrounding the premises of "TNO-Zuidpolder", Delft. A second sediment sample was collected from the river "Kromme Rijn" near Odijk (Province of Utrecht). This river has been contaminated with biocides for many years and is considered to be polluted. The concentrations of the test substance were 1 and 0.02 mg/l of labelled and unlabelled teflubenzuron.

In the "Kromme Rijn" sediment system, very little of the teflubenzuron was biodegraded to CO_2: after 12 weeks (t_{12}) only 0.9% of the initial radioactivity was detected as $^{14}CO_2$. The radioactivity in the aqueous phase increased from 12% at t_0 to 37% at t_{12} with the test concentration of 0.02 mg/l. In the test with 1 mg/l, the aqueous radioactivity reached only about 20% at the end of the test. While bound residues increased steadily from 4% at t_0 to 18% at t_{12} the radioactivity in the extracts of the solids decreased from 80% to 33%.

In the "TNO" sediment system only 0.6% of the initial ^{14}C was detected as $^{14}CO_2$ after 12 weeks. The biodegradation was similar to that in the "Kromme Rijn" system: the radioactivity in the aqueous phase rose from 8% at the beginning to 12% at the end of the test. Bound residues increased from 4% to 32% after 12 weeks, during which time the radioactivity in the extracts of the solids fell from 85% to 48%.

Analysis of the aqueous phase and the extracts of the solids by thin-layer chromatography showed that the amount of teflubenzuron decreased from 87% to 35% after 12 weeks in the Kromme Rijn sediment system: its half-life was calculated to be 6 weeks. Two major degradation products were found, one with an Rf value of 0.23, which accounted for 6% of the initial radioactivity, and one with an Rf value of 0.08, which was identified as 3,5-dichloro-2,4-difluorophenylurea. The amount of this compound increased from 3% at the beginning of the test to 14% at t_{12}.

In the TNO sediment system, the amount of teflubenzuron fell from 86% at t_0 to 40% at t_{12}, giving a half-life of 7 weeks. In this system the same 2 major products were found, 5% of the radioactivity being present as the compound with an Rf value of 0.23. The product with the Rf of 0.08 (the phenylurea) increased from 1% at t_0 to 23% at t_{12}.

METHODS OF RESIDUE ANALYSIS

Analytical methods

This type of compound is particularly suited to determination by HPLC with ultraviolet detection because of its strong absorbance near 254 mm. Teflubenzuron could not be determined by GLC on packed columns, but when GLC with capillary columns became available this procedure was also employed.

Plant material and soil. The HPLC procedure was used for alfalfa, apples, blackberries, broccoli, cabbage, citrus, cotton, cucumbers, grapes, grass, maize, mushrooms, peaches, pears, peppers, potatoes, soya beans and tomatoes, as well as for soils (Celamerk, 1982, 1985b, 1986; Cyanamid, 1995; Shell, 1988a). Teflubenzuron is extracted with acetone or from soil also with an acetone/water

mixture. Clean-up is by solvent partition followed by gel-permeation chromatography and/or silica gel column chromatography. The residue is determined by reversed-phase HPLC with ultraviolet detection at 254 nm. Recoveries determined at levels ranging from 0.01 to 2 mg/kg were 70-110%.

Some years ago, an alternative method using capillary gas chromatography was introduced and applied to cherries and plums (Shell, 1992). As in the other methods, the acetone extracts are cleaned up by solvent partition and gel-permeation chromatography. Determination is by both reversed-phase HPLC and gas chromatography with mass-selective detection. Thus GC-MS can be used as a confirmatory method. Validation was at fortification levels between 0.01 and 0.1 mg/kg. Recoveries were 85-96% and the limit of determination (LOD) was 0.01 mg/kg for both procedures.

Animal products. An HPLC method was developed for the residue determination of teflubenzuron in products of cattle and hens (Shell, 1988b). The residue is extracted with methanol or acetonitrile. In the case of high-fat material the fat is separated in a cooling bath. Further clean-up and quantification by HPLC is as in the crop method. The procedure was validated for muscle, liver, kidney, fat, skin, milk and eggs. Fortification levels were 0.01 to 0.2 mg/kg. Recoveries from the various types of sample were in the range 73-110% with an LOD of 0.01 mg/kg.

Water. A modification of the HPLC method was developed for the determination of residues in water (Cyanamid, 1988) to meet the requirements of the EU drinking water directive. Teflubenzuron is extracted from the water sample on a C_{18} "Bondelut" solid-phase column. After elution, further clean-up on a silica gel column follows if necessary. The compound is determined by reversed-phase HPLC with UV detection at 254 nm. Analysis of water samples spiked at 0.0001 - 0.002 mg/l gave recoveries of 78-100%. The LOD was 0.0001 mg/l.

Air. Air is sucked through a Tenax or XAD column and teflubenzuron is adsorbed. The compound is then eluted and determined by reverse-phase HPLC with UV detection or by GLC with a mass-selective detector as a confirmatory method. The LOD was 56 $\mu g/m^3$ air. The method was later validated in a separate study and the LOD was lowered to 10 $\mu g/m^3$ air (Weitzel, 1995). The range of recoveries was 83-110%.

Stability of pesticide residues in stored analytical samples

The stability of teflubenzuron in various crops held under frozen conditions up to 36 months was tested by Thorstenson (1990). Untreated apple, pear, potato and cabbage samples were fortified at 0.2 mg/kg with teflubenzuron and placed in a freezer maintained at -20°C. The frozen samples were analyzed after 3, 6, 12, and 36 months of storage. The results are shown in Table 3.

Table 3. Stability of teflubenzuron in various crops stored under frozen conditions (Thorstenson, 1990).

Crop	Teflubenzuron (%)[1] remaining after			
	3 months	6 months	12 months	36 months
Apple	115.0	101.3	95.0	74.3
Pear	90.7	113.7	90.0	82.3
Potato	77.0	114.3	91.3	84.7
Cabbage	103.0	102.3	95.0	94.0

[1] Average values

After one and three years storage, 91-95% and 74-94% of the original teflubenzuron remained respectively, showing that teflubenzuron is stable in the crops investigated when stored under deep-frozen conditions.

USE PATTERN

Teflubenzuron is an insecticide which inhibits the biochemical synthesis of chitin. It controls all immature stages of the insects; the youngest larvae are the most susceptible and less teflubenzuron needs to be applied to control young larvae than older ones. The insecticidal activity of teflubenzuron results primarily from the ingestion of treated foliage; contact activity is less significant. The compound is also ovicidal by topical application.

Teflubenzuron controls a wide range of insect pests (*lepidopterous* and *coleopterous* larvae being the most sensitive), and some mites e.g. citrus rust mites - *Phyllocoptruta spp.*

The Meeting received information on GAP from the manufacturer, Germany (Anon., 1995a), Poland (Anon., 1995b), The Netherlands (Anon., 1996) and Spain (Anon., 1993). The information submitted by Spain referred to 1993 and was not taken into consideration because current Spanish GAP (March 1996) was included in the information from the manufacturer.

Table 4 shows the registered uses of teflubenzuron on all important crops and the countries where they apply.

Table 4. Registered uses of teflubenzuron.

Crop	Country	Product	Application			PHI, days
			No.	Max. rate kg ai/ha (kg ai/hl)	F/G	
Apple	Argentina	150 SC	2	0.15	F	21
	France	150 SC	1	0.05	F	14
	Greece	150 SC	2-3	0.21	F	30
	Italy	150 SC 53 SC	1-3 1-3	0.15 0.16	F F	14 14
	Jordan	150 SC	2	(0.0075)	F	15
	Netherlands	150 SC	1-3	0.11-0.16 (0.015)	F	28
	Portugal	150 SC	3	0.07	F	14
	Saudi Arabia	150 SC	2	(0.011)	F	21
	Spain	150 SC	1-2	0.09	F	28
	Switzerland	150 SC	2-3	0.3	F	21
	United Arab Emirates	150 SC	2	(0.0038)	F	7
Broccoli	Netherlands	150 SC	2-4	0.06	F	14
Brussels sprouts	Netherlands	150 SC	6-8	0.09	F	14
Head cabbages	Indonesia	50 EC		0.025	F	
	Italy	150 SC 53 SC	1 1	0.03 0.03	F F	7 7
	Jordan	150 SC	2	(0.0075)	F	14
	Poland	150 SC	1	0.03	F	14
	Switzerland	150 SC		0.045	F	14
Cabbage, Red	Germany	150 SC	1	0.06	F	14
	Netherlands	150 SC	2-4	0.06	F	14
Cabbage, Savoy	Germany	150 SC	1	0.06	F	14
	Switzerland	150 SC		0.045	F	14
Cabbage, White	Germany	150 SC	1	0.06	F	14
	Netherlands	150 SC	2-4	0.06	F	14
Cauliflower	Netherlands	150 SC	2-4	0.06	F	14
Cereals	Switzerland	150 SC		0.06	F	42
Chinese cabbage	Netherlands	150 SC	2-4	0.06	F	14
Citrus fruits	Saudi Arabia	150 SC	2	(0.011)	F	21
	South Africa	150 SC	1-2	(0.003)	F	30
	United Arab Emirates	150 SC	2	(0.0038)	F	7
Coffee	Brazil	150 SC		0.038	F	30
	Kenya	150 SC	1-2	0.11	F	30
Cotton	Argentina	150 SC	2	0.011	F	21
	Brazil	150 SC		0.0075-0.1	F	30
	Colombia	150 SC	2	0.019	F	
	Ecuador	150 SC	2	0.019-0.045	F	

	Guatemala	150 SC	2-3	0.075	F	
	Paraguay	150 SC	2-3	0.0075	F	30
Cucumber	Jordan	150 SC	2	(0.0075)	F	3
	Netherlands	150 SC	3-5	0.23 (0.015)	G	3
	Saudi Arabia	150 SC	2	(0.011)	F	
Cucurbits	Spain	150 SC	2-3	0.18	F,G	3
Egg plant	Italy	150 SC 53 SC	1-2 1-2	0.022 0.024	F F	10 10
	Jordan	150 SC	2	(0.0075)	F	
	Netherlands	150 SC	3-5	0.23 (0.015)	G	3
	Saudi Arabia	150 SC	2	(0.011)	F	
	Spain	150 SC 150 SC	2-3 2-3	0.18 0.23	F G	3 3
Gherkin	Jordan	150 SC	2	(0.0075)	F	
	Netherlands	150 SC	3-5	0.23 (0.015) 0.06 (0.0075)	G F	3 3
Grapes	Italy	150 SC 53 SC	2 2	0.09 0.096	F F	28 28
	Saudi Arabia	150 SC	2	(0.011)	F	21
	Spain	150 SC	2	0.09	F	28
	Switzerland	150 SC	2	0.09	F	21
Maize	Colombia	150 SC	2	0.045	F	
	Ecuador	150 SC	2	0.045	F	21
	Italy	150 SC 53 SC	2 2	0.15 0.16	F F	28 28
Melon	Netherlands	150 SC	3-5	0.23 (0.015)	G	3
Mushroom	Belgium	150 SC		3.0	G	14
	Italy	150 SC 53 SC	1 1	6.0 4.8	F F	45 45
Nectarine	Italy	150 SC 53 SC	1-3 1-3	0.11 0.12	F F	21 21
Nuts	France	150 SC	1	0.045	F	
Olives	Greece	150 SC	2-3	0.29	F	
Orchards and small trees	Poland	150 SC	1	0.11	F	28
Peach	Italy	150 SC 53 SC	1-3 1-3	0.11 0.12	F F	21 21
	Saudi Arabia	150 SC	2	(0.011)	F	21
Pear	France	150 SC	1	0.05	F	14
	Greece	150 SC	2-3	0.21	F	60
	Italy	53 SC	1-3	0.16	F	14
	Jordan	150 SC	2	(0.0075)	F	15
	Netherlands	150 SC	1-4	0.11-0.16 (0.011)	F	28

	Portugal	150 SC	3	0.07	F	14
	Spain	150 SC	1-2	0.06	F	28
	Switzerland	150 SC	2-3	0.3	F	21
	United Arab Emirates	150 SC	2	(0.0038)	F	7
Peppers, Sweet	Italy	150 SC 53 SC	1-2 1-2	0.075 0.08	F F	10 10
	Netherlands	150 SC	3-5	0.23 (0.015)	G	3
	Spain	150 SC 150 SC	2-3 2-3	0.18 0.23	F G	3 3
	Jordan	150 SC	2	(0.0075)	F	
	Saudi Arabia	150 SC	2	(0.011)	F	
Peppers, Chilli	Indonesia	50 SC		0.1	F	
Potato	Germany	150 SC	1	0.045	F	14
	Italy	150 SC 53 SC	1-2 1-2	0.024 0.024	F F	28 28
	Poland	150 SC	1-2	0.038	F	14
	Saudi Arabia	150 SC	2	(0.011)	F	
	Spain	150 SC	1-2	0.022	F	28
	Switzerland	150 SC		0.038	F	21
Quince	France	150 SC	1	0.05	F	14
Sorghum	Colombia	150 SC	2	0.045	F	
	Ecuador	150 SC	2	0.045	F	21
Soya bean	Brazil	150 SC		0.0075-0.023	F	30
	Paraguay	150 SC	2-3	0.0075	F	30
Squash	Saudi Arabia	150 SC	2	(0.011)	F	
Squash, Summer	Netherlands	150 SC	3-5	0.23 (0.015) 0.06 (0.0075)	G F	3 3
Stone fruits	Switzerland	150 SC	2-3	0.12	F	21
Tomato	Argentina	150 SC	3-8	0.075	F	7
	Brazil	150 SC	5-8	0.038	F	7
	Colombia	150 SC	5-8	0.03	F	
	Ecuador	150 SC	5-8	0.03	F	21
	Jordan	150 SC	2	(0.0075)	F	3
	Netherlands	150 SC	3-5	0.23 (0.015)	G	3
	Paraguay	150 SC	5-8	0.038	F	7
	Spain	150 SC 150 SC	2-3 2-3	0.18 0.23	F G	3 3
Vegetables	Saudi Arabia	150 SC	2	(0.011)	F	7
	United Arab Emirates	150 SC	2	(0.0038)	F	7
Watermelon	Jordan	150 SC	2	(0.0075)	F	

F: field, G: greenhouse.

RESIDUES RESULTING FROM SUPERVISED TRIALS

Supervised residue trials have been carried out world-wide on a wide range of crops such as citrus fruits, pome fruits, stone fruits, berries, various types of vegetables, oilseeds and fodder items. In nearly all trials teflubenzuron has been used as a 15% SC formulation (150 g/l suspension concentrate). In some trials teflubenzuron formulated as an emulsion concentrate has been applied as well (25 g/l; 33.3 g/l; 100 g/l). Trials were carried out in different climatic areas, e.g. Northern and Southern Europe, Northern and Southern America, South Africa and the Far East.

Many of the field studies cover a wide range of spray regimes for such reasons as the need to obtain data on rates of decline or to examine the effects of high application rates. In most trials the application rate was given as concentration of active ingredient in the spray solution and as kg of active ingredient per hectare if the spray volume was known. In the few trials in which different rates were used in multiple applications the individual rates are given in the Tables.

Residues have been rounded to 2 significant figures. Where replicates of the same sample have been analysed the mean has been calculated. In most of the studies carried out in the USA, replicate samples were analysed; in these cases all the results are shown. Underlined residues are from treatments according to GAP. Double-underlined residues have been used for the estimation of Supervised Trials Median Residue (STMR) levels.

Table 5. List of Tables of residues from supervised trials on crops.

Table No.	Commodity, country, year
Table 6	Grapefruit, USA (1987)
Table 7	Oranges, Brazil (1984, 1985, 1989), South Africa (1984, 1985, 1986), USA (1987)
Table 8	Apples, France (1982, 1983, 1984, 1985), Germany (1982, 1983, 1984, 1985, 1993), Italy (1984) Slovakia (1986), South Africa, UK (1985), USA (1987)
Table 9	Pears, France (1982, 1984, 1985, 1990), Germany (1993), Italy (1983, 1984)
Table 10	Cherries, Germany (1991)
Table 11	Plums, Germany (1991, 1992), Italy (1988)
Table 12	Nectarines, Italy (1988)
Table 13	Peaches, France (1982, 1983, 1984), Italy (1986)
Table 14	Grapes, France (1982, 1983, 1984), Germany (1982, 1983), Italy (1983, 1984)
Table 15	Raspberries, blackberries, blueberries, Germany (1984, 1985)
Table 16	Persimmons, Korea (1992)
Table 17	Kiwifruit, New Zealand (1986, 1987)
Table 18	Head cabbages, Brazil (1989), Germany (1982, 1983, 1984, 1985), Philippines (1985), Malaysia (1984), UK (1985), USA (1987)
Table 19	Broccoli, Germany (1984)
Table 20	Brussels sprouts, The Netherlands (1985)
Table 21	Cucumbers, Germany (1987), The Netherlands (1987)

Table No.	Commodity, country, year
Table 22	Peppers, Italy (1985, 1988), Korea (1992)
Table 23	Egg plants, Italy (1988)
Table 24	Tomatoes, Brazil (1986, 1989), Germany (1987), Italy (1987), UK (1986, 1993), USA (1987)
Table 25	Mushrooms, Germany (1984, 1985), The Netherlands (1987)
Table 26	Chinese cabbage, Philippines (1985), Malaysia (1985), The Netherlands (1986)
Table 27	Peas, France (1983)
Table 28	Soya beans, Brazil (1985, 1989), USA (1987)
Table 29	Potatoes, Brazil (1985), France (1984), Germany (1982, 1983, 1986, 1987), Italy (1985), Slovakia (1987), USA (1987)
Table 30	Maize, France (1982, 1983, 1984), Germany (1982, 1983, 1986, 1987), Italy (1984)
Table 31	Cotton, Brazil (1989), Guatemala (1984, 1985), Mexico (1984), USA (1987)
Table 32	Coffee beans, Brazil (1989)
Table 33	Alfalfa forage, green grass, Italy (1988)
Table 34	Soya bean forage and hay, USA (1987)

Citrus fruits (Tables 6-7). The use of teflubenzuron on citrus is registered in African countries, where one or two treatments with 0.003-0.011 kg ai/hl are recommended with PHIs of 7 (United Arab Emirates), 21 (Saudi Arabia) and 30 days (South Africa).

In one trial in the USA, grapefruit treated 3 times at a rate of 0.11 kg ai/ha were harvested 45 days after last treatment. In the samples taken, residues of teflubenzuron in whole fruit were <0.05 mg/kg.

Table 6. Residues of teflubenzuron in grapefruit from supervised trials, USA, 1987.

Application			PHI, days	Sample	Residues, mg/kg	Report No.
kg ai/hl	kg ai/ha	No.				
0.005	0.112	3	45	whole fruit	<0.05	HAS A025.001

Nineteen residue trials were carried out on oranges in Brazil, South Africa and the USA during 1984-1989. In one US trial teflubenzuron was applied three times at a rate of 0.11 kg ai/ha. Whole fruits analysed for teflubenzuron showed residues <0.05 mg/kg 45 or 76 days after the last treatment. Samples from trials in South Africa and Brazil were analysed to determine the distribution of residues between edible pulp and inedible peel. As expected, increasing the spray concentration or the number of applications resulted in increased residues. In nine studies in South Africa the rate of decline of residues was determined after 1 or 2 applications of 0.094-2.3 kg ai/ha. Residues in the peel decreased from 0.33-11 mg/kg at day 0 to 0.28-6.8 mg/kg at day 53 or 64, and were <0.05-0.12 mg/kg in the pulp both on days 0 and 64.

Two supervised residue trials were carried out on oranges in 1989 in Brazil. After two pre-harvest applications of 0.06 kg/ha or 0.12 kg/ha, residues after 30 days were <0.01 mg/kg in the pulp and 0.34-0.36 mg/kg in the peel.

In six further trials conducted in South Africa and Brazil samples were taken 159 and 160 days after single applications of 0.009-0.03 kg ai/hl sprays. Residues in the peel were <0.05-2.2 mg/kg, and in the pulp <0.05-0.1 mg/kg.

Table 7. Residues of teflubenzuron in oranges from supervised trials.

Country, year	Application kg ai/hl	kg ai/ha	No.	PHI, days	Sample	Residues, mg/kg	Report No.
Brazil, 1984	0.009	0.09	1	159	pulp peel	<0.05 2.2	BRA84100601
Brazil, 1985	0.009	0.09	2	68	pulp peel	<0.05 1.3	BRA85100601
Brazil, 1989	0.003	0.06	2	30	pulp peel	<0.01 0.34	SHGR.90.016
Brazil, 1989	0.06	0.12	2	30	pulp peel	<0.01 0.36	SHGR.90.016
South Africa, 1984	0.012	0.58	1	0 0 1 1 2 2 5 5 8 16 33 33 64 64	pulp peel pulp peel pulp peel pulp peel pulp pulp pulp peel pulp peel	0.08 3.0 0.16 2.7 0.11 2.9 0.09 2.5 <0.05 0.05 0.01 2.4 0.02 1.9	13406-532-2701
South Africa, 1984	0.024	1.2	1	0 0 1 1 2 2 5 5 8 16 33 33 64	pulp peel pulp peel pulp peel pulp peel pulp pulp pulp peel pulp	0.11 3.6 0.13 4.3 0.26 3.4 0.23 3.4 0.10 0.07 0.02 3.9 0.07	13406-532-2702

Country, year	Application kg ai/hl	kg ai/ha	No.	PHI, days	Sample	Residues, mg/kg	Report No.
				64	peel	3.1	
South Africa, 1984	0.048	2.3	1	0	pulp	0.12	13406-532-2703
				0	peel	11	
				1	pulp	0.30	
				1	peel	10	
				2	pulp	0.33	
				2	peel	8.2	
				5	pulp	0.32	
				5	peel	8.9	
				8	pulp	0.39	
				16	pulp	0.28	
				33	pulp	0.12	
				33	peel	7.2	
				64	pulp	0.12	
				64	peel	6.8	
South Africa, 1985	0.016		1	160	pulp	<0.05	CU 85-87-A
				160	peel	<0.05	
South Africa, 1985	0.02		1	160	pulp	<0.05	CU 85-87-B
				160	peel	0.06	
South Africa, 1985	0.025		1	160	pulp	<0.05	CU 85-87-C
				160	peel	0.06	
South Africa, 1985	0.03		1	160	pulp	<0.05	CU 85-87-D
				160	peel	<0.05	
South Africa, 1986	0.004	0.094	1	0	pulp	<0.05	86/01/I/K/1A
				0	peel	0.33	
				1	pulp	<0.05	
				1	peel	0.41	
				4	pulp	<0.05	
				4	peel	0.28	
				6	pulp	<0.05	
				6	peel	0.39	
				8	pulp	<0.05	
				8	peel	0.29	
				14	pulp	<0.05	
				14	peel	0.36	
				25	pulp	<0.05	
				25	peel	0.24	
				53	pulp	<0.05	
				53	peel	0.28	
South Africa, 1986	0.008	0.19	1	0	pulp	<0.05	86/01/I/K/1B
				0	peel	0.94	
				1	pulp	0.05	
				1	peel	0.92	
				4	pulp	<0.05	
				4	peel	0.59	
				6	pulp	<0.05	
				6	peel	0.85	
				8	pulp	<0.05	
				8	peel	0.82	
				14	pulp	<0.05	

Country, year	Application kg ai/hl	kg ai/ha	No.	PHI, days	Sample	Residues, mg/kg	Report No.
				14	peel	0.77	
				25	pulp	<0.05	
				25	peel	0.60	
				53	pulp	<0.05	
				53	peel	0.59	
South Africa, 1986	0.004	0.094	2	0	pulp	<0.05	86/01/I/K/1C
				0	peel	0.55	
				7	pulp	<0.05	
				7	peel	0.37	
				22	pulp	<0.05	
				22	peel	0.35	
				35	pulp	<0.05	
				35	peel	0.47	
				49	pulp	<0.05	
				49	peel	0.45	
South Africa, 1986	0.008	0.19	2	0	pulp	0.10	86/01/I/K/1D
				0	peel	1.4	
				7	pulp	<0.05	
				7	peel	1.3	
				22	pulp	<0.05	
				22	peel	0.91	
				35	pulp	<0.05	
				35	peel	0.95	
				49	pulp	0.07	
				49	peel	0.87	
South Africa, 1986	0.004		2	0	pulp	<0.05	C86-268A
				0	peel	0.73	
				7	pulp	<0.05	
				7	peel	0.36	
				14	pulp	<0.05	
				14	peel	0.65	
				34	pulp	0.10	
				34	peel	0.26	
South Africa, 1986	0.07		1	0	pulp	0.06	C86-268B
				0	peel	2.6	
				7	pulp	<0.05	
				7	peel	1.3	
				14	pulp	<0.05	
				14	peel	1.6	
				34	pulp	0.20	
				34	peel	0.43	
South Africa, 1987	0.006	0.11	3	45	whole fruit	<0.05	HAS A025.001
				45		<0.05	
				76		<0.05	
				76		<0.05	

Underlined values are from treatments according to GAP of the United Arab Emirates (PHI 7 days) or South Africa (PHI 30 days)

Pome fruits. Teflubenzuron is registered for use on apples, pears and quinces in France, apples and pears in Greece, Italy, Jordan, The Netherlands, Portugal, Spain, Switzerland and the United Arab Emirates, and apples in Argentina and Poland. One to three treatments with 0.06 (Spain) to 0.3 kg ai/ha (Switzerland) are recommended with PHIs of 7 (United Arab Emirates) to 30 (Greece, apple) and 60 days (Greece, pear).

Apples (Table 8). Supervised trials on a number of varieties of apples have been conducted in Slovakia, France, Germany, Italy, South Africa, the UK and the USA between 1982 and 1993. No GAP was available for Germany or the UK, but the trials in these countries could be related to Dutch GAP. After 1-4 treatments with 0.11-0.21 kg ai/ha (0.011 kg ai/hl) the highest residue after PHIs of 24-28 days was 0.37 mg/kg.

Many residue trials were conducted in France, but they did not accord with French or other Southern European GAP.

The results of 5 Italian trials were evaluated with reference to Southern European GAP (Italy, Greece and Switzerland). Two of the trials (2 treatments with 0.24-0.27 kg ai/ha approximated both Swiss GAP (2-3 treatments, 0.3 kg ai/ha, 21-day PHI) and Greek (2-3 treatments, 0.21 kg ai/ha, 30-day PHI). The residues were 0.46 and 0.48 mg/kg at 21 days and 0.45 and 0.51 mg/kg at 28 days. Two other complied with the Italian GAP of 1-3 treatments at 0.15-0.16 kg ai/ha and a PHI of 14 days. The residues were 0.23 and 0.27 mg/kg.

No GAP was available for the USA so the results could not be evaluated.

Table 8. Residues of teflubenzuron in apples from supervised trials.

Country, year	Application kg ai/hl	kg ai/ha	No.	PHI, days	Residues, mg/kg	Report No.
France, 1982	0.008	0.098	6	28	0.25	I8264.35.02
France, 1982	0.008	0.098	6	19	0.27	I8264.35.01
France, 1983	0.009	0.098	6	21	0.34	I8364.35.01
France, 1983	0.009	0.098	6	27	0.35	I8364.35.02
France, 1984	0.005 0.005 0.005	0.05 0.05 0.05	4 4 4	42 42 42	0.1 0.06 0.04	FR84405046018B
France, 1984	0.011 0.011 0.011	0.1 0.1 0.1	4 4 4	42 42 42	0.2 0.15 0.22	FR84405046018C
France, 1984	0.015 0.015 0.015	0.15 0.15 0.15	4 4 4	42 42 42	0.37 0.18 0.27	FR84405046018D
France, 1984	0.001	0.11	8	27	0.68	I8451.3501A
France, 1984	0.01	0.15	8	27	1.2	I8451.3501B
France, 1984	0.008	0.11	9	6	0.49	I8451.3601A

Country, year	Application kg ai/hl	kg ai/ha	No.	PHI, days	Residues, mg/kg	Report No.
France, 1984	0.01	0.15	9	6	0.63	I8451.3601B
France, 1985	0.005 0.009 0.014	0.14 0.27 0.41	2 2 2	100 100 100	<0.07 <0.06 0.16	16082.86A
France, 1985	0.005 0.009 0.014	0.11 0.23 0.34	2 2 2	100 100 100	<0.05 0.08 <0.07	16082.86B
France	0.005	0.05	1	118	<0.03	15734.87
France	0.017	0.05	1	107	<0.03	
France	0.005	0.05	1	136	<0.03	
France	0.005	0.05	1	126	<0.03	
France	0.01	0.05	1	121	<0.04	
France	0.005	0.05	2	83	<0.04	
France	0.005	0.05	2	81	0.03	
France	0.005	0.05	2	41	0.08	
France	0.01	0.05	2	80	<0.03	
France	0.005	0.05	3	30	0.04	
France	0.004	0.05	3	35	0.03	
France	0.005	0.05	3	82	<0.03	
France	0.005	0.05	3	28	0.06	
France	0.005	0.05	3	50	<0.03	
France	0.005	0.05	3	42	0.06	
France	0.005	0.05	3	47	0.03	
France	0.005	0.05	3	16	0.08	
France	0.005	0.05	3	47	0.07	
France	0.005	0.05	3	5	0.08	
France	0.005	0.05	3	16	0.11	
France	0.005	0.05	3	48	0.07	
France	0.005	0.05	6	38	0.09	
France	0.005	0.05	6	54	0.10	
France	0.005	0.05	6	30	0.09	
Germany, 1982	0.011	0.14	4	0 14 21	0.32 0.29 0.20	C820972

Country, year	Application kg ai/hl	kg ai/ha	No.	PHI, days	Residues, mg/kg	Report No.
				28	0.23	
				35	0.20	
				42	0.26	
Germany, 1982	0.011	0.14	4	0	0.27	C821072
				14	0.17	
				21	0.19	
				28	0.19	
				35	0.15	
				42	0.13	
Germany, 1982	0.011	0.14	1	0	0.31	C827572
				14	0.15	
				28	0.09	
				42	0.07	
				56	<0.05	
				70	<0.05	
				84	<0.05	
Germany, 1983	0.011	0.16	2	0	0.23	C832872
	0.005	0.08	1	14	0.21	
				28	0.19	
				35	0.19	
				42	0.18	
Germany, 1983	0.011	0.21	2	0	0.43	C83280101
	0.005	0.10	1	14	0.23	
				28	0.24	
				35	0.22	
				42	0.17	
Germany, 1983	0.011	0.21	2	0	0.46	C83280501
	0.005	0.10	1	14	0.38	
				28	0.36	
				35	0.31	
				42	0.28	
Germany, 1983	0.011	0.21	2	0	0.13	C83280601
	0.005	0.10	1	14	0.13	
				28	0.05	
				35	0.09	
				42	0.09	
Germany, 1984	0.011	0.12	4	0	0.35	C840672
				14	0.29	
				28	0.27	
				35	0.23	
				42	0.20	
Germany, 1984	0.011	0.21	4	0	0.70	C84060101
				14	0.56	
				28	0.37	
				35	0.35	
				50	0.30	
Germany, 1984	0.011	0.21	4	0	0.39	C84060601
				14	0.23	

Country, year	Application kg ai/hl	kg ai/ha	No.	PHI, days	Residues, mg/kg	Report No.
				28	0.29	
				35	0.21	
				49	0.18	
Germany, 1985	0.032	0.16	4	0	0.40	C852272
				3	0.29	
				7	0.38	
				14	0.28	
				21	0.22	
Germany, 1985	0.042	0.17	4	0	0.27	C852206
				3	0.24	
				7	0.25	
				14	0.23	
				21	0.16	
Germany, 1985	0.042	0.21	4	0	0.36	C852201
				3	0.16	
				7	0.13	
				14	0.12	
				21	0.12	
Germany, 1993	0.021	0.16	3	0	0.27	SHE-9308
				7	0.23	
				14	0.12	
				20	0.16	
Italy, 1984	0.008	0.15	1	0	0.29	I84/31/07/03
				7	0.20	
				14	0.23	
				21	0.19	
				28	0.18	
Italy, 1984	0.008	0.15	1	0	0.40	I84/31/07/04
				7	0.19	
				14	0.27	
				21	0.20	
				28	0.18	
Italy, 1984	0.014	0.24	1	0	0.86	I84/35/07/02
	0.014	0.27	1	7	0.73	
				14	0.66	
				21	0.46	
				28	0.51	
Italy, 1984	0.014	0.24	2	0	0.39	I84/35/07/03
				7	0.53	
				14	0.41	
				21	0.48	
				28	0.45	
Italy, 1984	0.014	0.24	1	0	1.3	I84/35/07/05
	0.014	0.27	3	7	0.87	
				14	0.56	
				21	0.65	
				28	0.60	

Country, year	Application kg ai/hl	kg ai/ha	No.	PHI, days	Residues, mg/kg	Report No.
Slovakia, 1986	0.015	0.15	4	48	0.53	CU87-389
Slovakia, 1986	0.015	0.15	4	64	0.28	CU87-388
South Africa	0.011	0.26	6	16	0.66	ZAF86I00501
UK, 1985	0.011	0.11	2	20	0.09	GBR85I00201
UK, 1985	0.011	0.11	2	24	0.11	GBR85I00202
UK, 1985	0.011	0.11	2	24	0.23	GBR85I00203
USA, 1987	0.006	0.11	4	5 5 11 11 20 20	0.25 0.12 0.26 0.12 0.17 <0.05	HAS A025.001
USA, 1987	0.024	0.11	4	7 7 15 15 30 30	0.06 <0.05 0.10 0.08 0.11 0.06	HAS A025.001
USA, 1987	0.024	0.11	4	7 7 7 7 7 7 7 7 7 7 7 15 15 15 15 15 15 30 30 30 30 30	0.10 0.07 0.12 <0.05 0.12 0.07 0.12 0.12 0.07 0.09 0.09 0.16 0.12 0.10 0.12 <0.05 0.08 0.05 0.16 0.10 0.08 0.09	HAS A025.001
USA, 1987	0.12	0.56	4	7 7 7 7 7 7 7 7	0.24 0.55 0.33 0.14 0.10 0.11 0.15 0.16	HAS A025.001

Country, year	Application kg ai/hl	kg ai/ha	No.	PHI, days	Residues, mg/kg	Report No.
				15	0.13	
				15	0.07	
				15	0.10	
				15	0.07	
				15	0.57	
				30	0.21	
				30	0.26	
				30	0.51	
				30	<0.05	
				30	0.30	
				30	0.10	
				30	0.27	
USA, 1987	0.012	0.11	4	7	0.09	HAS A025.001
				7	0.07	
				15	0.14	
				15	0.13	
				30	<0.05	
				30	<0.05	
USA, 1987	0.005	0.11	4	6	0.15	HAS A025.001
				6	0.13	
				12	0.12	
				12	0.10	
				12	0.12	
				12	0.10	
				24	0.18	
				24	0.08	
				24	0.11	
				24	0.11	
USA, 1987	0.012	0.11	4	10	0.05	HAS A025.001
				10	0.06	
				20	0.07	
				20	0.08	
				40	<0.05	
				40	0.06	

Underlined residues in German and UK trials are from treatments which approximate Dutch GAP.
Double-underlined Italian residues are from treatments which approximate Italian, Greek or Swiss GAP.

Pears (Table 9). Trials on eight varieties of pears were carried out in France, Germany and Italy. As in the trials on apples the spray regimes were widely spread. When samples were taken at maturity after 3 or fewer applications of 0.023-0.3 kg ai/ha the residues of teflubenzuron were ≤0.66 mg/kg.

Most of the trials could not be evaluated. Only three Italian trials were approximately in accord with Swiss GAP. The residues at a 21-day PHI ranged from 0.43 to 0.71 mg/kg after 2 or 3 treatments at 0.27 kg ai/ha.

Table 9. Residues of teflubenzuron in pears from supervised trials.

Country, year	Application kg ai/hl	kg ai/ha	No.	PHI, days	Residues, mg/kg	Report No.
France, 1982		0.30	1	71	0.20	I82653501
France, 1984	0.003	0.028-0.033	4	27	<0.05	FR84145146018A
France, 1984	0.005	0.055-0.065	4	27	<0.05	FR84145146018B
France, 1984	0.010	0.11-0.13	4	27	0.07	FR84145146018C
France, 1984	0.015	0.17-0.20	4	27	0.16	FR84145146018D
France, 1984	0.003 0.005 0.010 0.015	0.023 0.045 0.09 0.14	1 1 1 1	67 67 67 67	<0.05 <0.05 <0.05 <0.05	FR84145146018A-D
France, 1984	0.011	0.12	1	80	<0.05	I8454.34.01A
France, 1984	0.015	0.17	1	80	<0.05	I8454.34.01B
France, 1984	0.007	0.11	1	71	<0.05	I8454.36.01A
France, 1984	0.008	0.15	1	71	0.06	I8454.36.01B
France, 1985	0.011	0.26	5	28	0.30	16082.86
France, 1985	0.014	0.34	5	28	0.25	16082.86
France, 1985	0.011	0.26	6	24	0.38	16082.86
France, 1985	0.014	0.34	6	24	0.38	16082.86
France, 1990	0.008 0.010	0.075 0.10	2 2	49 49	0.04 0.07	S/FR/E/90/117
France, 1990	0.008 0.010	0.075 0.10	2 2	56 56	0.09 0.09	S/FR/E/90/118
France, 1990	0.008 0.011	0.083 0.11	2 2	72 72	0.03 0.04	S/FR/E/90/211
France, 1990	0.008 0.011	0.075 0.10	2 2	80 80	0.02 0.07	S/FR/E/90/441
Germany, 1993	0.021	0.16	3	0 7 14 21	0.47 0.26 0.26 0.26	SHE-9308
Italy, 1983	0.014	0.34	4	54	0.58	C83220701
Italy, 1983	0.014	0.27	2	54	0.28	C83220702
Italy, 1984	0.014	0.27	4	0 7 14 21	2.1 1.4 1.5 1.3	I84/33/07/01

Country, year	Application kg ai/hl	kg ai/ha	No.	PHI, days	Residues, mg/kg	Report No.
				28	1.2	
Italy, 1984	0.014	0.27	2	0 7 14 21 28	1.2 0.55 0.53 <u>0.43</u> 0.18	I84/33/07/11
Italy, 1984	0.014	0.27	3	0 7 14 21 28	1.0 0.65 0.67 <u>0.60</u> 0.66	I84/33/07/13
Italy, 1984	0.014	0.27	2	0 7 14 21 28	0.86 0.77 0.67 <u>0.71</u> 0.52	I84/33/07/02

Double-underlined residues are from treatments which approximated Swiss GAP.

Stone fruits. Teflubenzuron is registered in Switzerland for stone fruits and Italy for peaches and nectarines with 1-3 treatments of 0.12 kg ai/ha and a 21-day PHI. In Poland, one treatment of 0.11 kg ai/ha and a PHI of 28 days are recommended for orchards. In Saudi Arabia two applications of 0.011 kg ai/hl are registered for peaches with a PHI of 21 days.

Cherries (Table 10). Three trials on cherries were conducted in 1991 in Germany. Two applications were made at a rate of 0.16 kg ai/ha with an interval of 14 days. Fruit samples were taken immediately after the final application and 7, 14, 21 and 28 days later. The residues declined from 0.65-1.2 mg/kg on day 0 to 0.18- 0.21 mg/kg on day 28, calculated on the whole fruit.

Table 10. Residues of teflubenzuron in cherries from supervised trials, Germany, 1991.

Application kg ai/hl	kg ai/ha	No.	PHI, days	Residues, mg/kg[1]	Report No.
	0.16	2	0 7 14 21 28	0.87 0.38 0.49 <u>0.24</u> 0.18	SHTR.93.010 -9117-01
	0.16	2	0 7 14 21 28	1.2 1.2 0.47 <u>0.24</u> 0.20	SHTR.93.010 -9117-02
	0.16	2	0 7 14	0.65 0.76 0.56	SHTR.93.010 -9117-03

Application			PHI,	Residues,	Report No.
kg ai/hl	kg ai/ha	No.	days	mg/kg[1]	
			21	0.25	
			28	0.21	

[1] Calculated as whole fruit including stone
Underlined residues are from treatments which approximate Swiss GAP.

Plums (Table 11). Data on plums are confined to trials in Germany and Italy. After two applications of either 0.12 kg ai/ha (Italy) or 0.16 kg ai/ha (Germany) the residues of teflubenzuron in samples taken at harvest were <0.01-0.08 mg/kg.

Table 11. Residues of teflubenzuron in plums from supervised trials.

Country, year	Application kg ai/hl	kg ai/ha	No.	PHI, days	Sample	Residues, mg/kg	Report No.
Germany, 1991	0.015	0.16	2	0	pulp	0.12	SHTR.93.009
				7	pulp	0.09	-0118-01
				14	pulp	0.05	
				21	fruit[1]	0.04	
				28	fruit[1]	0.01	
Germany, 1991	0.015	0.16	2	0	pulp	0.11	SHTR.93.009
				7	fruit[1]	0.04	-9118-2
				14	fruit[1]	0.05	
				21	fruit[1]	0.04	
Germany, 1991	0.015	0.16	2	0	fruit[1]	0.01	SHTR.93.009
				7		<0.01	-9118-03
				14		0.01	
				21		≤0.01	
				28		<0.01	
Germany, 1991	0.015	0.16	2	0	fruit[1]	0.03	SHTR.93.009
				7		0.01	-9118-04
				14		<0.01	
				21		≤0.01	
				28		<0.01	
Germany, 1992	0.015	0.16	2	0	fruit[1]	0.02	SHTR.93.012
				6		0.02	
				13		0.02	
				20		0.01	
				27		0.02	
Italy, 1988	0.006	0.12	2	30	fruit	0.08	I03608
Italy, 1988	0.006	0.12	2	30	fruit	0.04	I02607
Italy, 1988	0.006	0.12	2	21	fruit	0.03	I01606
Italy, 1988	0.006	0.12	2	21	fruit	0.03	SHGR.89.061
Italy, 1988	0.006	0.12	2	30	fruit	0.04	SHGR.89.061
Italy, 1988	0.006	0.12	2	30	fruit	0.08	SHGR.89.061

[1] Calculated as whole fruit including stone
[2] Double-underlined residues are from treatments which approximate Swiss GAP

Nectarines (Table 12). Two trials were carried out in Italy in 1988. After two applications of 0.12 kg ai/ha (0.006% ai in the spray solution) residues in the pulp were 0.08 mg/kg and 0.04 mg/kg at 35 days and 56 days after the last treatment respectively.

Table 12. Residues of teflubenzuron in nectarines from supervised trials, Italy, 1988.

Application			PHI,	Sample	Residues,	Report No.
kg ai/hl	kg ai/ha	No.	days		mg/kg	
0.006	0.12	2	35	pulp	0.08	I04/602
0.006	0.12	2	56	pulp	0.04	I05/603

Peaches (Table 13). In ten trials in France and Italy peaches were treated 1-6 times with the 150 SC formulation at rates of 0.1-0.19 kg ai/ha. At harvest, residues ranged from <0.05 to 0.61 mg/kg.

Table 13. Residues of teflubenzuron in peaches from supervised trials.

Country,	Application			PHI,	Residues,	Report No.
year	kg ai/hl	kg ai/ha	No.	days	mg/kg	
France, 1982	0.016 0.008	0.10 0.10	1 1	21	<u>0.13</u>	I8262.35.01
France, 1982	0.009 0.010	0.10 0.10	1 1	24	<u>0.24</u>	I8262.35.02
France, 1983	0.007	0.10	3	20	<u>0.10</u>	I8362.36.01
France, 1983	0.006	0.10	3	20	<u>0.34</u>	I8362.36.02
France, 1984	0.011	0.11	6	21	0.19	I8456.36.01A
France, 1984	0.015	0.15	6	20	<0.05	I8456.36.01B
France, 1984	0.011	0.11	6	21	0.61	I8456.36.02A
France, 1984	0.015	0.15	6	21	0.52	I8456.36.02B
Italy, 1986	0.008	0.19	1	20	0.43	I86/01/07/01
Italy, 1986	0.008	0.19	2	39	0.34	I86/01/07/02

Underlined residues are from treatments which approximate Italian and Swiss GAP

Berries and other small fruits (Tables 14-15). The only registered uses are on grapes. The treatments are 2 x 0.09-0.096 kg ai/ha in Italy and Spain with a PHI of 28 days, and in Switzerland with a PHI of 21 days. In Saudi Arabia two treatments are registered with a spray concentration of 0.011 kg ai/hl and a 21-day PHI.

Nine varieties of grape were treated in France, Germany and Italy between 1982 and 1984 1-

3 times at rates of 0.1-0.3 kg ai/ha. After one or two applications 0.15-0.23 kg ai/ha the residues of teflubenzuron in samples taken 21-28 days after the last treatment were between <0.05 and 0.72 mg/kg. The application rates in most of the trials were much higher than the rates close to 0.1 kg ai/ha permitted by GAP.

Table 14. Residues of teflubenzuron in grapes from supervised trials.

Country, year	Application kg ai/hl	kg ai/ha	No.	PHI, days	Residues, mg/kg	Report No.
France, 1982	0.27	0.30	1	66	0.86	I8260.35.01
France, 1982	0.30	0.30	1	58	3.3	I8260.35.02
France, 1983	0.27	0.30	1	58	0.58	I8361.35.01
France, 1983	0.21	0.30	1	58	0.41	I8361.35.02
France, 1984	0.10	0.11	1	70	<0.05	I8455.35.01a
France, 1984	0.14	0.15	1	70	0.17	I8455.35.01b
France, 1984	0.006	0.025	1	77	0.02	FR8480508a
France, 1984	0.013	0.05	1	77	0.03	FR8480508b
France, 1984	0.025	0.10	1	77	0.11	FR8480508c
France, 1984	0.038	0.15	1	77	0.14	FR8480508d
Germany, 1982	0.015	0.18	2	0 21 28 35 42	0.25 0.09 0.14 0.17 0.12	C821472
Germany, 1982	0.015 0.015	0.15 0.23	1 1	0 21 28 35 42	0.27 0.20 <0.05 0.17 0.21	C82140301
Germany, 1983	0.011 0.011	0.16 0.21	1 1	0 21 28 35 42	0.17 0.17 0.15 0.26 0.15	C832772
Germany, 1983	0.011 0.011	0.16 0.21	1 1	0 21 28 35 42 49 56 63	0.37 0.23 0.24 0.19 0.22 0.22 0.16 0.13	C83270301
Germany, 1983	0.011	0.16	2	0 21 28	0.13 0.08 0.07	C83270601

Country, year	Application kg ai/hl	kg ai/ha	No.	PHI, days	Residues, mg/kg	Report No.
				35 42 56	0.05 0.04 0.05	
Italy, 1984	0.014	0.27	3	0 7 14 21 28	4.0 2.0 2.4 2.3 2.1	I84/36/07/01
Italy, 1984	0.014	0.27	1	0 7 14 21 28	3.1 1.8 1.1 1.0 1.0	I84/36/07/04
Italy, 1984	0.009	0.18	2	0 7 14 21 28	0.55 0.68 0.54 0.38 <0.05	I84/36/07/12
Italy, 1984	0.009	0.18	2	0 7 14 21 28	2.7 1.4 0.82 0.72 0.67	I84/36/07/13
Italy, 1984	0.014	0.27	1	0 7 14 21 28	1.7 1.4 1.1 1.0 0.94	I84/36/07/05

In Germany, teflubenzuron is registered for use in forests against larvae of *Tenthredinidae spp.* and free-eating caterpillars (1 x 0.023 kg ai/ha). As a result, wild berries and fruits are treated unintentionally. For consumer safety, eight supervised residue trials were carried out on wild raspberries, blackberries and blueberries. The worst case was simulated by application of approximately twice the registered rate. Berries were sampled at various dates after single applications of 0.045 kg ai/ha. Residues were <0.05-0.09 mg/kg in raspberries and blackberries and 0.03-0.15 mg/kg in blueberries.

Table 15. Residues of teflubenzuron in wild berries from supervised trials in Germany.

Year	Application kg ai/hl	kg ai/ha	No.	PHI, days	Residues, mg/kg	Report No.
Raspberries						
1984	0.004	0.045	1	0 3 7 14	0.06 <0.05 <0.05 <0.05	C84100401
1984	0.004	0.045	1	0 3 7 14	0.09 <0.05 <0.05 <0.05	C84100402
Blackberries						
1984	0.004	0.045	1	0 3 7 14	<0.05 <0.05 <0.05 <0.05	C84110401
1984	0.004	0.045	1	0 3 7 14	<0.05 <0.05 <0.05 <0.05	C84110402
1985	0.045	0.045	1	0 1 2 7 14	0.06 0.07 0.08 0.05 0.04	C851803
Blueberries						
1985	0.045	0.045	1	0 1 2 7 14	0.10 0.08 0.11 0.07 0.09	C85190501
1985	0.045	0.045	1	0 1 2 7 14	0.09 0.10 0.11 0.05 0.03	C85190502
1985	0.045	0.045	1	0 1 2 7 14	0.08 0.12 0.15 0.06 0.08	C85190503

Persimmons (Table 16). In a group of 5 trials in Korea in 1992 persimmons were treated 2-6 times with a 5% SC formulation at a rate of 0.25 kg ai/ha. Residues in samples taken 3-45 days after the

last treatment ranged between 0.02 and 0.09 mg/kg. No GAP was available to evaluate the results.

Table 16. Residues of teflubenzuron in persimmons from supervised trials in Korea, 1992. Whole fruit analysed.

Application kg ai/ha	No.	PHI, days	Residues, mg/kg	Report No.
0.25	2	45	0.02	KORE.92.002
0.25	3	3 7 15 30	0.06 0.05 0.03 0.03	KORE.92.002
0.25	4	3 7 15	0.07 0.04 0.04	KORE.92.002
0.25	5	3 7	0.09 0.06	KORE.92.002
0.25	6	3	0.09	KORE.92.002

Kiwifruit (Table 17). Four residue trials were carried out in New Zealand in 1986/87. The rates of application were 0.094, 0.19 and 0.25 kg ai/ha. Residues determined in whole fruit 16 and 99 days after the final application were 0.23-3.6 mg/kg and 0.28 mg/kg respectively. No GAP was available to evaluate the results.

Table 17. Residues of teflubenzuron in kiwifruit from supervised trials in New Zealand, 1986/7. Whole fruit analysed.

Application kg ai/hl	kg ai/ha	No.	PHI, days	Residues, mg/kg	Report No.
0.008	0.19	7	16	1.3	CU 88554A
0.010	0.25	7	16	3.6	CU 88554B
0.010	0.094	7	16	0.23	CU 88554C
0.010	0.25	3	99	0.28	CU 88554D

Head cabbages (Table 18). Registered uses exist for red, white and Savoy cabbage in Germany (1 x 0.06 kg ai/ha, 14-day PHI), for red and white cabbage in The Netherlands (2-4 x 0.06 kg ai/ha, 14-day PHI), and for head cabbage in Indonesia (0.025 kg ai/ha, no further information), Italy (1 x 0.03 kg ai/ha, 7-day PHI), Jordan (2 x 0.0075 kg ai/hl, 14-day PHI), Poland (1 x 0.03 kg ai/ha, 14-day PHI) and Switzerland (1 x 0.045 kg ai/ha, 14-day PHI).

Ten trials on Savoy cabbage were conducted in Germany in 1982-1985. After applying 3 x 0.06 kg ai/ha, all residues of teflubenzuron were <0.05 mg/kg at the recommended PHI of 14 days. Residues in 2 UK trials on Savoy cabbage treated once with 0.06 kg ai/ha were 0.05 and 0.17 mg/kg at day 14.

Four US trials on white and red cabbage were available, each with analyses of duplicate samples with and without wrapper leaves. Residues after applying 6 x 0.045 kg ai/ha were <0.05 to 0.36 mg/kg in samples with wrapper leaves and <0.05 to 0.11 mg/kg without wrapper leaves.

In 2 trials in Brazil (1 x 0.015 kg ai/ha, 1 x 0.03 kg ai/ha) residues were <0.01 mg/kg 3 and 7 days after treatment.

One trial was carried out in Malaysia and one in the Philippines. The residues were <0.05 mg/kg 18 and 7 days, respectively, after applying 6 or 9 x 0.045 kg ai/ha.

Table 18. Residues of teflubenzuron in head cabbage from supervised trials.

Country, year	Application			PHI, days	Sample	Residues, mg/kg	Report No.
	kg ai/hl	kg ai/ha	No.				
Head cabbage							
Brazil, 1989	0.004	0.015	1	3 7	head	<0.01 <0.01	SHGR.90.015
Brazil, 1989	0.008	0.030	1	3 7	head	<0.01 <0.01	SHGR.90.015
Malaysia, 1984	0.007	0.045	6	18	head	<0.05	MYR84I01101
USA, 1987	0.014	0.045	6	0 0 3 3 7 7 14 14	head w/o head with head w/o head with head w/o head with head w/o head with	0.18, 0.37 0.09, 0.42 0.18, 0.08 0.67, 0.37 <0.05, 0.06 0.36, 0.15 0.11, 0.10 0.36, 0.26	HAS A025.001
USA, 1987	0.006	0.045	6	0 0 3 3 7 7 14 14	head w/o head with head w/o head with head w/o head with head w/o head with	<0.05, <0.05 0.26, 0.45 <0.05, <0.05 0.33, 0.31 <0.05, <0.05 0.17, 0.21 <0.05, <0.05 0.18, 0.18	HAS A025.001
USA, 1987	0.017	0.045	6	0 0 3 3 7 7 14 14	head w/o head with head w/o head with head w/o head with head w/o head with	<0.05, <0.05 0.15, 0.14 <0.05, <0.05 <0.05, 0.20 <0.05, <0.05 0.14, 0.11 <0.05, <0.05 0.09, 0.07	HAS A025. 01
USA, 1987	0.024	0.045	6	0 0 3 3 7 7	head w/o head with head w/o head with head w/o head with	<0.05, <0.05 0.10, 0.14 <0.05, <0.05 0.10, 0.10 <0.05, <0.05 0.15, <0.05	HAS A025. 01

Country, year	Application			PHI, days	Sample	Residues, mg/kg	Report No.
	kg ai/hl	kg ai/ha	No.				
				14 14	head w/o head with	<0.05, <0.05 <0.05, <0.05	
Savoy cabbage							
Germany, 1982	0.012	0.06	3	0 14 28 35 42	head	0.75 <u>≤0.05</u> <0.05 <0.05 <0.05	C821172
Germany, 1982	0.010	0.06	3	0 14 28 35 42	head	<0.05 <u>≤0.05</u> <0.05 <0.05 <0.05	C82110101
Germany, 1983	0.015	0.06	3	0 14 28 35 42	head	0.41 <u>≤0.05</u> <0.05 <0.05 <0.05	C832972
Germany, 1983	0.010	0.06	3	0 17 31 37 44	head	0.99 <u>≤0.05</u> <0.05 <0.05 <0.05	C83290101
Germany, 1983	0.015	0.06	3	0 14 21 28 34 40	head	0.25 <u>≤0.05</u> <0.05 <0.05 <0.05 <0.05	C83290601
Germany, 1984	0.010	0.06	3	0 7 14 21 28	head	0.98 <0.05 <u>≤0.05</u> <0.05 <0.05	C840872
Germany, 1984	0.008	0.06	3	0 7 15 20 27	head	<0.05 <0.05 <u>≤0.05</u> <0.05 <0.05	C84080101
Germany, 1985	0.008	0.06	3	0 3 7 10 14	head	0.10 0.05 <0.05 <0.05 <u>≤0.05</u>	C851501
Germany, 1985	0.010	0.06	3	0 3 7 10 14	head	<0.05 <0.05 <0.05 <0.05 <u>≤0.05</u>	C851505
Germany,	0.010-	0.06	3	0 3	head	0.31 0.22	C851572

Country, year	Application			PHI, days	Sample	Residues, mg/kg	Report No.
	kg ai/hl	kg ai/ha	No.				
1985	0.012			7 14 21		<0.05 <u><0.05</u> <0.05	
UK, 1985	0.02	0.06	1	13	head	<u>0.05</u>	GBR85I00101
UK, 1985	0.02	0.06	1	13	head	<u>0.17</u>	GBR85I00102
White cabbage							
Philippines, 1985	0.005	0.045	9	0 3 5 7	head	0.12 <0.05 <0.05 <0.05	PHI85I00201

w/o: without wrapper leaves
with: with wrapper leaves
Double-underlined residues were from treatments according to German GAP, but 3 treatments instead 1 were carried out

<u>Broccoli</u> (Table 19). Teflubenzuron is registered for use on broccoli in The Netherlands with 2-4 treatments at 0.06 kg ai/ha and a PHI of 14 days. In two German trials corresponding to Dutch GAP the residues were 0.13 and 0.19 mg/kg at day 14.

Table 19. Residues of teflubenzuron in broccoli from supervised trials in Germany, 1984. Whole broccoli analysed.

Application			PHI,	Residues,	Report No.
kg ai/hl	kg ai/ha	No.	days	mg/kg	
0.010-0.012	0.06	3	0 7 14 21 28	0.42 0.24 <u>0.13</u> 0.06 <0.05	C 842772
0.010-0.012	0.06	3	0 7 14 21 28	0.73 0.38 <u>0.19</u> 0.08 <0.05	C 842872

Underlined residues are from treatments according to Dutch GAP

<u>Brussels sprouts</u> (Table 20). Teflubenzuron is currently registered in The Netherlands, where 6-8 treatments at 0.09 kg ai/ha with a PHI of 14 days are recommended.

Eight residue trials were conducted in The Netherlands with 4, 5 and 6 applications at 0.06 or 0.09 kg ai/ha. The residues of teflubenzuron after 14 days were 0.1-0.28 and 0.12-0.48 mg/kg after treatment with 0.06 and 0.09 kg ai/ha respectively.

Table 20. Residues of teflubenzuron in Brussels sprouts from supervised trials in The Netherlands, 1985. Whole plants analysed.

Application			PHI,	Residues, mg/kg	Report No.
kg ai/hl	kg ai/ha	No.	days		
0.006	0.06	6	14 21	0.18 0.2	85-162A
0.006	0.06	5	14	0.15	85-163A
0.006	0.06	5	14 21	0.28 0.2	85-164A
0.006	0.06	4	14	0.1	85-165
0.009	0.09	6	14 21	0.39 0.24	85-162B
0.009	0.09	5	14	0.24	85-163B
0.009	0.09	5	14 21	0.48 0.34	85-164B
0.009	0.09	4	14	0.12	85-165

Cucumbers (Table 21). There are registered field and glasshouse uses on cucurbits in Spain (2-3 x 0.18 kg ai/ha, 3-day PHI), glasshouse uses on cucumbers and gherkins in The Netherlands (3-5 x 0.23 kg ai/ha, 3-day PHI), and field uses on cucumbers and gherkins in Jordan (0.0075 kg ai/hl, 3-day PHI). In Saudi Arabia 2 field applications with 0.011 kg ai/hl are used on cucumbers.

Three indoor trials were carried out in Germany. The residues after 3 days were 0.03 and 0.07 mg/kg from approximately 3 x 0.09 kg ai/ha, and 0.14 mg/kg from 3 x 0.18 kg. In two field trials in Italy, where 3 x 0.075 kg ai/ha were applied, the residues after 3 days were 0.02 and 0.19 mg/kg.

Table 21. Residues of teflubenzuron in cucumbers from supervised trials, 1987. Whole cucumbers analysed.

Country	Application kg ai/hl	kg ai/ha	No.	PHI, days	Residues, mg/kg	Report No.
Indoors						
Germany	0.008	0.09	3	0 1 3 7 10 14	0.03 0.03 0.03 0.02 0.02 0.01	C 872722
Germany	0.007 0.008	0.067 0.009	1 2	0 1 3 5 7 10 14	0.08 0.09 0.07 0.05 0.04 0.02 0.01	C 872730
Germany		0.18	3	0 1 3 5 7 10 14	0.20 0.22 0.14 0.15 0.06 0.04 0.02	C 872720
Outdoors						
Italy	0.008	0.075	3	0 2 7 10 14 21	0.08 0.02 0.03 0.02 0.03 <0.01	I87160710
Italy	0.008	0.075	3	0 2 7 10 14 21	0.13 0.19 0.12 0.06 0.02 <0.01	I87160711

Peppers (Table 22). Teflubenzuron is registered for field use on sweet peppers in Italy (1-2 x 0.08 kg ai/ha, 10-day PHI), Jordan (2 x 0.0075 kg ai/hl), Saudi Arabia (2 x 0.011 kg ai/hl) and Spain (2-3 x 0.18 kg ai/hl, 3-day PHI), and on chilli peppers in Indonesia at 0.1 kg ai/ha. Glasshouse use on sweet peppers is registered in The Netherlands with 3-5 applications and in Spain with 2 or 3 applications of 0.23 kg ai/ha and a PHI of 3 days.

The Meeting reviewed 6 trials from Italy, 4 of them according to Italian GAP with one treatment of 0.075 kg ai/ha. The residues were 0.08-0.11 mg/kg 10 days after application.

Five trials (2-5 x 0.1 kg ai/ha) in Korea showed residues from <0.05 to 0.11 mg/kg. No GAP was available to evaluate the trials.

Table 22. Residues of teflubenzuron in peppers from supervised field trials.

Country, year	Application kg ai/hl	kg ai/ha	No.	PHI, days	Residues, mg/kg	Report No.
Italy, 1985	0.0038	0.038	1	28	<0.05	I6083.86
Italy, 1985	0.008	0.075	1	28	<0.05	I6083.86
Italy,1988	0.008	0.075	1	0 0 3 3 7 7 10 14	0.16 0.09 0.09 0.07 0.15 0.08 <u>0.11</u> 0.07	ITA88001
Italy, 1988	0.008	0.075	1	0 0 3 3 7 7 10 14 14	0.11 0.12 0.12 0.14 0.10 0.11 <u>0.09</u> 0.11 0.15	ITA88002
Italy, 1988	0.008	0.075	1	0 3 7 10 14	0.13 0.08 0.12 <u>0.11</u> 0.07	SHGR.89.058
Italy, 1988	0.008	0.075	1	0 3 7 10 14	0.12 0.13 0.11 <u>0.10</u> 0.13	SHGR.89.058
Korea, 1992		0.10	2	45	<0.05	KORE.92.04
Korea, 1992		0.10	3	3 7 15 30	<0.05 <0.05 <0.05 <0.05	KORE.92.04
Korea, 1992		0.10	4	3 7 15	<0.05 <0.05 <0.05	KORE.92.04
Korea, 1992		0.10	5	3 7	0.06 <0.05	KORE.92.04
Korea, 1992		0.10	6	3	0.11	KORE.92.04

Double-underlined residues are from trials according to Italian GAP.

<u>Egg plants</u> (Table 23). Teflubenzuron is registered for field use on egg plants in Italy with 1-2 x

0.022-0.024 kg ai/ha, 10-day PHI. The GAP for field use in Jordan, Saudi Arabia and Spain, and for glasshouse use in The Netherlands and Spain, is the same as for sweet peppers.

The Meeting reviewed 6 trials from Italy, 4 of them according to Italian GAP with 1 treatment at 0.023 kg ai/ha. The residues were <0.01 mg/kg 10 days after application.

Table 23. Residues of teflubenzuron in egg plants from supervised field trials in Italy.

Year	Application			PHI,	Residues,	Report No.
	kg ai/hl	kg ai/ha	No.	days	mg/kg	
1985	0.001	0.011	1	28	<0.05	I6083.86
1985	0.002	0.023	1	28	<0.05	I6083.86
1988	0.002	0.023	1	0 0 3 3 7 7 10 14	0.02 0.02 <0.01 0.01 <0.01 <0.01 <0.01 <0.01	ITA88003
1988	0.002	0.023	1	0 0 3 3 7 7 10 14	0.02 0.02 <0.01 0.01 <0.01 <0.01 <0.01 <0.01	ITA88004
1988	0.002	0.023	1	0 3 7 10 14	0.02 0.01 <0.01 <0.01 <0.01	SHGR.89.059
1988	0.002	0.023	1	0 3 7 10 14	<0.01 <0.01 <0.01 <0.01 <0.01	SHGR.89.059

Tomatoes (Table 24). Teflubenzuron is currently registered for glasshouse use in The Netherlands (3-5 x 0.23 kg ai/ha, 3-day PHI), and for glasshouse and field use in Spain (2-3 x 0.18-0.23 kg ai/ha, 3-day PHI). Field treatments are registered in Brazil and Paraguay (5-8 x 0.038 kg ai/ha with a PHI of 7 days), Argentina (3-8 x 0.075 kg ai/ha, PHI 7 days), Columbia and Ecuador (5-8 x 0.03 kg ai/ha and in Ecuador a PHI of 21 days). In Jordan, 2 applications of 0.0075 kg ai/hl and a 3-day PHI are allowed.

Field use. Six trials were conducted in Brazil. After applying 3-5 x 0.03-0.09 kg ai/ha, residues ranged from 0.05 to 0.15 mg/kg after 6 or 7 days. Two Italian trials with 4 x 0.075 kg ai/ha resulted

in residues of 0.1 and 0.28 mg/kg 2 days after application.

Four trials were carried out in the USA. The report did not state whether the trials were outdoors or in greenhouses, but as they were in Fresno, California, it is very likely that they were outdoors. Residues from <0.05 to 0.1 mg/kg were found 3 days after treatment with 5 x 0.028-0.056 kg ai/ha.

Glasshouse use. Three trials in Germany were with 3-4 treatments at 0.09-0.17 kg ai/ha. The residues were 0.1, 0.17 and 0.47 mg/kg 3 days after the last application.

Table 24. Residues of teflubenzuron in tomatoes from supervised trials.

Country, year	Application kg ai/hl	kg ai/ha	No.	PHI, days	Residues, mg/kg	Report No.
Indoors						
Germany, 1987	0.008	0.09	3	0 1 3 5 7 10 14	0.12 0.13 0.10 0.13 0.13 0.08 0.08	C 872630
Germany, 1987	0.008	0.17	4	0 1 3 5 7 10 14	0.67 0.59 0.47 0.37 0.51 0.31 0.28	C 872620
Germany, 1987	0.007 0.008	0.11 0.14	1 2	0 1 3 5 7 10 14	0.15 0.23 0.17 0.08 0.09 0.07 0.08	C 872621
UK, 1986	0.022	0.17	4	0 1 2 3	0.55 0.65 0.32 0.20	GBR86I00101
UK, 1993	0.015	0.23	5	3	0.36	CFS 1994-060
UK, 1993	0.075-0.077	1.1-1.2	5	3	1.7	CFS 1994-060

Outdoors						
Brazil, 1986	0.004	0.045	5	0 4 6 8 12 18	0.05 0.08 <u>0.05</u> 0.06 0.09 0.05	BRA86I00402
Brazil, 1986	0.007	0.09	5	0 4 6 8 12 18	0.19 0.13 0.15 0.09 0.22 0.09	BRA86I00502
Brazil, 1986	0.004	0.045	5	0 4 6 8 12 18	0.10 0.05 <u>0.06</u> <0.05 <0.05 <0.05	BRA86I00902
Brazil, 1986	0.007	0.09	5	0 4 6 8 12 18	0.14 0.09 0.08 0.05 0.04 0.03	BRA86I01002
Brazil, 1989	0.006	0.03	3	0 7 14	0.09 0.10 0.05	SHGR.90.014
Brazil, 1989	0.006	0.03	3	0 7 14	0.07 0.12 0.01	SHGR.90.014
Italy, 1987	0.008	0.075	4	0 2 7 10 14 21	0.25 0.10 0.18 0.23 0.18 0.11	I87150710
Italy, 1987	0.008	0.075	4	0 2 7 10 14 21	0.35 0.28 0.29 0.17 0.13 0.12	I87150711
USA, 1987	0.006	0.028	5	0 0 1 1 3 3 7 7	<0.05 0.07 0.09 0.09 0.06 0.09 0.08 0.08	HAS A025.001

				14	<0.05	
				14	<0.05	
USA, 1987	0.012	0.056	5	0	0.16	HAS A025.001
				0	<0.05	
				1	0.10	
				1	0.13	
				3	<0.05	
				3	0.10	
				7	0.09	
				7	0.13	
				14	0.07	
				14	0.06	
USA, 1987	0.008	0.056	5	0	0.08	HAS A025.001
				0	0.08	
				1	<0.05	
				1	<0.05	
				3	0.06	
				3	0.07	
				7	0.07	
				7	<0.05	
				14	0.09	
				14	0.09	
USA, 1987		0.056	5	0	<0.05	HAS A025.001
				0	<0.05	
				3	<0.05	
				3	<0.05	
				7	<0.05	
				7	<0.05	
				14	0.05	
				14	<0.05	

The underlined residue in the UK trial CFS 1994-060 is from a treatment which complies with Dutch GAP

Mushrooms (Table 25). Uses exist in Belgium (3 kg ai/ha, 14-day PHI) and Italy (1 x 4.8-6 kg ai/ha, 45-day PHI). In a trial with 4 replicates on cultivated mushrooms in The Netherlands the residues were all <0.05 mg/kg 25 days after applying 2 x 4.9 mg/kg.

In Germany, wild mushrooms may be treated unintentionally by the use of teflubenzuron in forests against larvae of *tenthredinidae spp.* and free-eating caterpillars at 0.023 kg ai/ha. The worst case was simulated by the application of approximately twice this rate (0.045 kg ai/ha) in 3 trials in Germany. The residues on days 0, 1 and 2 were all <0.05 mg/kg in two trials and 0.07, 0.07 and 0.05 mg/kg respectively in the third.

Table 25. Residues of teflubenzuron in mushrooms from supervised trials. Whole mushrooms analysed.

Country, year	Application			PHI,	Residues,	Report No.
	kg ai/hl	kg ai/ha	No.	days	mg/kg	
Wild mushrooms						

Country, year	Application kg ai/hl	kg ai/ha	No.	PHI, days	Residues, mg/kg	Report No.
Germany, 1984	0.004	0.045	1	0 1 2 7 14	0.07 0.07 0.05 <0.05 <0.05	C 84090401
Germany, 1984	0.004	0.045	1	0 1 2 7 14	<0.05 <0.05 <0.05 <0.05 <0.05	C 84090402
Germany, 1985	0.045	0.045	1	0 1 2 7 14	<0.05 <0.05 <0.05 <0.05 <0.05	C 851603
Cultivated mushrooms						
Netherlands, 1987	0.16	4.9	2	25 25 25 25	<0.05 <0.05 <0.05 <0.05	87-139

Chinese cabbage (Table 26). Teflubenzuron is currently registered only in The Netherlands. It is recommended for field use at a rate of 0.06 kg ai/ha 2-4 times a season with a PHI of 14 days.

Two trials were carried out in The Netherlands. After applying 1 x 0.06 or 0.09 kg ai/ha the residues were 0.22 and 0.31 mg/kg at day 14.

Further trials were conducted in Malaysia (4 x 0.045 kg ai/ha) and the Philippines (9 x 0.045 kg ai/ha). The residues were 0.16, 0.93 and 2.8 mg/kg 7 days after treatment. No GAP was available for Asian countries with which to evaluate the results.

Table 26. Residues of teflubenzuron in Chinese cabbage from supervised trials.

Country, year	Application kg ai/hl	kg ai/ha	No.	PHI, days	Residues, mg/kg	Report No.
Netherlands, 1986	0.006	0.06	1	1 14 21	2 0.22 0.06	86.158A
Netherlands, 1986	0.009	0.09	1	1 14 21	3.2 0.31 0.1	86.158B
Malaysia, 1985	0.004	0.045	4	7	0.93	MYS85I01002
Malaysia, 1985	0.007	0.045	4	7	2.3	MYS85I01001
Philippines, 1985	0.005	0.045	9	0 3 5 7	1.6 0.65 0.34 0.16	PHI85I00301

Peas, immature seeds (Table 27). No information on GAP was available for peas. One trial was conducted in France. The residues were 0.19 mg/kg in green peas with pods and <0.05 mg/kg in the peas 21 days after 2 applications of 0.045 kg ai/hl (0.23 kg ai/ha).

Table 27. Residues of teflubenzuron in peas from supervised trials in France, 1983.

Application kg ai/hl	kg ai/ha	No.	PHI, days	Sample	Residues, mg/kg	Report No.
0.045	0.22	2	21 21	peas with pod seeds	0.19 <0.05	I8353.34.01

Soya beans (Table 28). Teflubenzuron is registered for use on soya beans in Brazil (0.0075-0.023 kg ai/ha) and Paraguay (2-3 treatments of 0.0075 kg ai/ha) with a PHI of 30 days in both countries.

Six trials were carried out in Brazil, four of them according to GAP. The residues were all <0.05 mg/kg in the beans, and 0.17 and 0.29 mg/kg in two samples of hulls.

One study involving replicated trials with various application rates was carried out in the USA. Except in one trial, residues in the dried beans were below the LOD of 0.05 mg/kg after 1 or 2 treatments at 0.022-0.34 kg ai/ha 28-43 days after application. In the exceptional trial residues in the dried beans were <0.05, 0.07, 0.16, 0.28 and 0.34 mg/kg from 2 x 0.034 kg ai/ha.

Table 28. Residues of teflubenzuron in soya beans from supervised trials.

Country, year	Application kg ai/hl	kg ai/ha	No.	PHI, days	Sample	Residues, mg/kg	Report No.
Brazil, 1989	0.006	0.015	2	21	seed	<0.01	SHGR.90.017A
				30		<0.01	
	0.012	0.030	2	21	seed	<0.01	
				30		<0.01	
Brazil, 1989	0.006	0.015	2	21	seed	<0.01	SHGR.90.017B
				30		<0.01	
	0.012	0.030	2	21	seed	<0.01	
				30		<0.01	
Brazil, 1985	0.012	0.030	1	53	hull	0.17	BRA85I00801
				53	seed	<0.05	
Brazil, 1985	0.036	0.090	1	53	hull	0.29	BRA85I00901
				53	seed	<0.05	
USA, 1987	0.012	0.022	1	47	seed	<0.05	HAS A025.001
	0.012	0.022	1	47		<0.05	
	0.12	0.22	1	47		<0.05	
	0.12	0.22	1	47		<0.05	
	0.015	0.022	1	30	seed	<0.05	
	0.015	0.022	1	30		<0.05	
	0.15	0.22	1	30		<0.05	
	0.15	0.22	1	30		<0.05	
	0.024	0.022	1	43	seed	<0.05	
	0.024	0.022	1	43		<0.05	
	0.24	0.22	1	43		<0.05	
	0.24	0.22	1	43		<0.05	
	0.015	0.022	1	42	seed	<0.05	
	0.015	0.022	1	42		<0.05	
	0.15	0.22	1	42		<0.05	
	0.15	0.22	1	42		<0.05	
	0.12	0.34	1	28	seed	<0.05	
	0.12	0.34	1	28		<0.05	
	0.015	0.034	2	30	seed	0.34	
	0.015	0.034	2	30		0.28	
	0.015	0.034	2	30		0.07	
	0.015	0.034	2	30		0.16	
	0.015	0.034	2	35		<0.05	
	0.018	0.034	2	30	seed	<0.05	
	0.018	0.034	2	30		<0.05	

Potatoes (Table 29). Teflubenzuron is registered for use on potatoes in Germany (1 x 0.045 kg ai/ha),

Italy (1-2 x 0.024 kg ai/ha), Poland (1-2 x 0.038 kg ai/ha), Saudi Arabia (2 x 0.011 kg ai/hl), Spain (1-2 x 0.022 kg ai/ha) and Switzerland (0.038 kg ai/ha). The PHIs range from 14 to 28 days.

Trials on potatoes were conducted in the following countries: 2 in Brazil (4 x 0.06 or 0.12 kg ai/ha), 2 in France (1 x 0.046-0.26 kg ai/ha), 3 in Germany (2 x 0.052 kg ai/ha), 2 in Italy (1 x 0.045 kg ai/ha), 1 in Slovakia (1 x 0.022 kg ai/ha) and 1 study in the USA with replicated applications of 5 x 0.034-0.17 kg ai/ha. No residues above 0.05 mg/kg (LOD) were found in any sample at any PHI (7-79 days).

Table 29. Residues of teflubenzuron in potatoes from supervised trials. Tubers analysed.

Country, year	Application kg ai/hl	kg ai/ha	No.	PHI, days	Residues, mg/kg	Report No.
Brazil, 1985	0.015	0.06	4	24	<0.05	BRA85I01201
Brazil, 1985	0.030	0.12	4	24	<0.05	BRA85I01501
France, 1984	0.002	0.043	1	49	<0.05	FR8413511
	0.005	0.085	1	49	<0.05	
	0.010	0.17	1	49	<0.05	
	0.015	0.23	1	49	<0.05	
France, 1984		0.03	1	7	<0.05	FR8414521
		0.06	1	7	<0.05	
		0.12	1	7	<0.05	
		0.18	1	7	<0.05	
Germany, 1982	0.01	0.052	2	0	<0.05	C821272
				64	<0.05	
Germany, 1982	0.01	0.052	2	0	<0.05	C82120101
				14	<0.05	
Germany, 1982	0.01	0.052	2	14	<0.05	C82120501
				41	<0.05	
Italy, 1985	0.01	0.045	1	21	<0.05	I84370701
Italy, 1985	0.01	0.045	1	35	<0.05	I84370703
Slovakia, 1987		0.022	1	79	<0.05	CU-88-492
USA, 1987		0.034	5	16	<0.05	HAS A025.001
		0.034	5	16	<0.05	
		0.17	5	16	<0.05	
	0.007	0.034	5	14	<0.05	
	0.007	0.034	5	14	<0.05	
	0.036	0.17	5	14	<0.05	
	0.004	0.034	5	14	<0.05	
	0.017	0.034	5	14	<0.05	
	0.017	0.034	5	14	<0.05	
	0.017	0.034	5	24	<0.05	
	0.017	0.034	5	24	<0.05	
	0.08	0.17	5	24	<0.05	

Country, year	Application kg ai/hl	kg ai/ha	No.	PHI, days	Residues, mg/kg	Report No.
	0.08	0.17	5	24	<0.05	

Cereals. Teflubenzuron is registered for general use on cereals only in Switzerland, with 0.06 kg ai/ha and a PHI of 42 days, but is approved for use on maize in Columbia, Ecuador and Italy, and for sorghum in Columbia and Ecuador: supervised trials were reported only on maize.

Maize (Table 30). GAP in Columbia and Ecuador consists in 2 treatments at 0.045 kg ai/ha, with a PHI of 21 days in Ecuador, and in Italy 2 x 0.15-0.16 kg ai/ha with a PHI of 28 days.

Supervised trials were in France (6), Germany (10), Italy (2) and Slovakia (3). In the French trials (1 x 0.11-0.3 kg ai/ha) the residues were 0.16 to 6 mg/kg in the whole plant and <0.01 or <0.05 mg/kg in the grain 62-108 days after application. In Germany, 1 x 0.15 kg ai/ha gave residues of 0.41-1.9 mg/kg in the whole plant at 28 days and <0.01 or <0.05 mg/kg in the grain at 39-114 days after application. The residues found at 63-75 days in the grain in two Slovakian trials were <0.05 mg/kg. Two trials in Italy with 2 x 0.15 kg ai/ha complied with GAP. At 28 days the residues were 3.6 and 3.9 mg/kg in the whole plant and <0.05 mg/kg in the grain.

Table 30. Residues of teflubenzuron in maize from supervised trials.

Country, year	Application kg ai/hl	kg ai/ha	No.	PHI, days	Sample	Residues, mg/kg	Report No.
France, 1982	0.051	0.26	1	62	plant	2.0	I82.55.33.01
France, 1982	0.051	0.26	1	74 74	plant grain	3.2 <0.05	I82.55.33.02
France, 1983	0.06	0.3	1	70 84	plant grain	6.0 <0.05	I83.57.34.01
France, 1984	0.023	0.11	1	66 108	plant grain	0.48 <0.05	I84.57.34.02a
France, 1984	0.05	0.15	1	66 108	plant grain	0.51 <0.05	I84.57.34.02b
France, 1984	0.01	0.05	1	101 101	plant grain	0.16 <0.01	FR8460501
France, 1984	0.02	0.10	1	101 101	plant grain	0.53 <0.01	FR8460501
France, 1984	0.03	0.15	1	101 101	plant grain	0.54 <0.01	FR8460501
Germany, 1982	0.025	0.15	1	0 14 28 42 56	plant	3.9 2.1 1.4 1.1 0.90	C821372

Country, year	Application kg ai/hl	kg ai/ha	No.	PHI, days	Sample	Residues, mg/kg	Report No.
				70		<0.05	
Germany, 1982	0.025	0.15	1	0 14 28 42 56 71	plant	2.1 1.6 0.70 1.0 0.60 <0.05	C82130501
Germany, 1983	0.015	0.15	1	0 14 28 39 39 69	plant grain	2.6 1.8 1.9 1.9 <0.05 <0.05	C833072
Germany, 1983	0.030	0.15	1	0 14 28 44 49	plant grain	1.4 2.1 1.4 1.4 <0.05	C83300301
Germany, 1983	0.038	0.15	1	0 15 31 45	plant	3.8 3.4 1.8 2.3	C83300401
Germany, 1983	0.019	0.15	1	0 14 28 44 63	plant grain	2.4 1.1 0.92 0.96 <0.05	C83300501
Germany, 1983	0.019	0.15	1	0 14 28 39 51	plant grain	1.6 1.8 1.9 0.58 <0.05	C83300601
Germany, 1986	0.038	0.15	1	0 28 56 111	plant grain	3.2 1.4 0.57 <0.05	C862272
Germany, 1986	0.038	0.15	1	0 25 56 98	plant grain	4.8 1.3 0.62 <0.05	C862205
Germany, 1987	0.038	0.15	1	0 28 56 114	plant grain	3.7 0.41 0.63 <0.01	C871772
Italy, 1984	0.025	0.15	2	0 7 14 21	plant	6.0 5.0 5.5 3.5	I84/38/07/11

Country, year	Application kg ai/hl	kg ai/ha	No.	PHI, days	Sample	Residues, mg/kg	Report No.
				28		3.6	
				28	grain	<0.05	
Italy, 1984	0.025	0.15	2	0	plant	5.0	I84/38/07/12
				7		3.7	
				14		3.0	
				21		2.8	
				28		3.9	
				28	grain	<0.05	

Cotton (Table 31). Teflubenzuron is registered for use on cotton in Argentina (2 x 0.011 kg ai/ha, 21-day PHI), Brazil (0.0075-0.1 kg ai/ha, PHI 30 days), Paraguay (2-3 x 0.0075 kg ai/ha, PHI 30 days), Colombia (2 x 0.019 kg ai/ha), Ecuador (2 x 0.019-0.045 kg ai/ha) and Guatemala (2-3 x 0.075 kg ai/ha).

Residue trials were conducted in Brazil (4), Guatemala (2), Mexico (1) and the USA (one study with various rates and concentrations). The residues in the seed were <0.01 mg/kg after 31 days from 2 x 0.03 or 0.06 kg ai/ha in Brazil, and <0.05 mg/kg at 6-8 days from 14-15 x 0.039 kg ai/ha in Guatemala and 12 x 0.06-0.08 kg ai/ha at 18 days in Mexico. In the US trials the residues were <0.05, 0.07, 0.08, 0.11, 0.24 and 13 mg/kg, and 0.78, 3.1 and 4.6 mg/kg after the application of 12 x 0.045 and 12 x 0.45 kg ai/ha, respectively, at 14 or 18 days after treatment.

Table 31. Residues of teflubenzuron in cotton from supervised trials. Seed analysed.

Country, year	Application kg ai/hl	kg ai/ha	No.	PHI, days	Residues, mg/kg	Report No.
Brazil, 1989	0.012	0.03	2	31	<0.01	SHGR.90.019
Brazil, 1989	0.024	0.06	2	31	<0.01	SHGR.90.019
Brazil, 1989	0.012	0.03	2	30	<0.01	SHGR.90.019
Brazil, 1989	0.024	0.06	2	30	<0.01	SHGR.90.019
Guatemala, 1984	0.14	0.039	14	6	<0.05	GTM84I00301
Guatemala, 1985	0.14	0.039	15	8	<0.05	GTM85I00101
Mexico, 1984	0.30-0.40	0.06-0.08	12	18	<0.05	MEX84I00101
USA, 1987	0.012	0.045		14	0.11	HAS A025.001
	0.012	0.045		14	0.24	
	0.12	0.45		14	3.1	
	0.048	0.045	12	14	<0.05	
	0.048	0.045	12	14	0.08	
	0.48	0.45	12	14	0.78	
	0.019	0.045	12	18	0.07	
	0.019	0.045	12	18	13	
	0.19	0.45	12	18	4.6	

Coffee beans (Table 32). The only registered uses of teflubenzuron on coffee are in Brazil (0.038 kg ai/ha) and Kenya (1-2 x 0.11 kg ai/ha) at PHIs of 30 days in both countries. In two residue trials in Brazil, 2 applications of 0.075 or 0.15 kg ai/ha gave residues of 0.6 and 1.7 mg/kg respectively, after 35 days.

Table 32. Residues of teflubenzuron in coffee beans from supervised trials, Brazil, 1989.

Application kg ai/hl	kg ai/ha	No.	PHI, days	Residues, mg/kg	Report No.
0.01	0.075	2	35	0.60	SHGR.90.018
0.02	0.15	2	35	1.7	SHGR.90.018

Alfalfa forage and green grass (Table 33). Two supervised trials on alfalfa and one on green grass were carried out in Italy (1 x 0.075 kg ai/ha). The residues in alfalfa forage declined from 1.4 and 2 mg/kg at day 3 to 0.18 and 1.2 mg/kg at day 28 after application, and in grass from 12 mg/kg at day 3 to 0.71 mg/kg at day 28. No information on GAP was available to evaluate the trials.

Table 33. Residues of teflubenzuron in forage (alfalfa and grass) from supervised trials in Italy, 1988. Green forage analysed.

Application kg ai/hl	kg ai/ha	No.	PHI, days	Residues, mg/kg	Report No.
Alfalfa					
0.008	0.075	1	0	4.8	SHGR.89.007
			3	2.0	
			7	1.7	
			14	2.2	
			21	0.06	
			28	0.18	
0.008	0.075	1	0	0.49	SHGR.89.007
			3	1.4	
			7	0.72	
			14	0.48	
			21	0.46	
			28	1.2	
Grass					
0.008	0.075	1	3	12	SHGR.89.008
			7	5.2	
			14	7.4	
			21	1.5	
			28	0.71	

Soya bean forage and hay (Table 34). Teflubenzuron is registered for use on soya beans in Brazil (0.0075-0.023 kg ai/ha) and in Paraguay (2-3 treatments of 0.075 kg ai/ha) with PHIs of 30 days.

Residues were determined in soya bean forage in 8 trials and in hay in 4 in the USA. After applying 1 or 2 x 0.022 kg ai/ha the residues in the forage and hay were 0.17-0.56 and 0.19-1.3 mg/kg respectively at 14 days.

Table 34. Residues of teflubenzuron in soya bean forage and hay from supervised trials in the USA, 1987.

Application kg ai/hl	kg ai/ha	No.	PHI, days	Sample	Residues, mg/kg	Report No.
0.012	0.022	1	0	forage	0.97	HAS A025.001
			0		1.1	
			3		0.71	
			3		0.73	
			7		1.0	
			7		0.27	
			14		0.49	
			14		0.30	
0.012	0.022	2	0	forage	0.93	HAS A025.001

Application kg ai/hl	kg ai/ha	No.	PHI, days	Sample	Residues, mg/kg	Report No.
			0		1.2	
			3		0.45	
			3		0.31	
			7		0.22	
			7		0.26	
			14		0.36	
			14		0.17	
			0	hay	2.2	
			0		1.7	
			3		1.2	
			3		0.96	
			7		<0.05	
			7		<0.05	
			14		0.08	
			14		0.19	
					0.19	
0.015	0.022	1	0	forage	0.59	HAS A025.001
			0		0.60	
			3		0.53	
			3		0.28	
			7		0.06	
			7		0.66	
			14		0.22	
			14		0.30	
0.015	0.022	2	0	forage	0.61	HAS A025.001
			0		0.66	
			3		1.1	
			3		0.66	
			7		0.50	
			7		1.0	
			14		0.54	
			14		0.51	
			0	hay	0.86	
			0		1.1	
			3		<0.05	
			3		1.8	
			7		1.9	
			7		0.80	
			14		0.91	
			14		0.26	
0.024	0.022	1	0	forage	1.3	HAS A025.001
			0		0.54	
			3		0.44	
			3		0.67	
			7		0.44	
			7		0.46	
			14		0.53	
			14		0.48	
			0	hay	1.0	
			0		2.3	
			3		2.1	
			3		0.17	
			7		1.6	

Application kg ai/hl	kg ai/ha	No.	PHI, days	Sample	Residues, mg/kg	Report No.
			7		2.4	
			14		0.60	
			14		1.3	
0.024	0.022	2	0	forage	0.58	HAS A025.001
			0		0.58	
			3		0.60	
			3		0.34	
			7		0.46	
			7		0.34	
			14		0.34	
			14		0.45	
0.015	0.022	1	0	forage	0.97	HAS A025.001
			0		0.92	
			3		0.98	
			3		0.93	
			7		0.79	
			7		0.86	
			14		0.56	
			14		0.46	
0.015	0.022	2	0	forage	0.67	HAS A025.001
			0		0.76	
			3		0.48	
			3		0.31	
			7		0.28	
			7		0.62	
			14		0.28	
			14		0.41	
			0	hay	0.57	
			0		1.3	
			3		0.77	
			3		0.88	
			7		0.65	
			7		1.6	
			14		0.62	
			14		0.77	

Animal feeding studies

The results of metabolism studies with goats and poultry indicated that teflubenzuron *per se* was the main terminal residue.

Dairy cattle (Table 35). Cameron and Puglis (1989a) fed dairy cows (4/treatment) with teflubenzuron for a period of 28 days with 10 ppm, 30 ppm or 100 ppm in the diet. Three cows in each test group were slaughtered 17-24 h after the final administration. Two additional cows fed at the highest level were maintained on the basal diet for a further 7 or 14 days after the end of dosing to provide data on depletion. Milk samples were taken from three days before administration to termination. Samples of milk and tissues (subcutaneous fat, peritoneal fat, liver, kidney, skeletal muscle) were analysed for residues of teflubenzuron. Liver samples were also analysed for the metabolite E-115, 1-(3,5-dichloro-2,4-difluorophenyl)-3-(2,6-difluoro-3-hydroxybenzoyl)urea.

The results of the analyses are given in Table 35. Low concentrations of teflubenzuron were detected in the liver and/or kidney of some animals. There was no indication of any correlation with the feeding level (or with the withdrawal time in the high-dose group); positive values were found in the liver of one control animal and the kidney of another. All residues were below 0.05 mg/kg. Residues of the metabolite E-115 in liver samples were <0.05 mg/kg (LOD).

Peritoneal fat contained only low residues of teflubenzuron (all <0.03 mg/kg). Two positive samples were from the control group. There was little evidence of any dose-related trend. Residue concentrations at or close to the LOD (0.01 mg/kg) were recorded in subcutaneous fat in the high-dose group. No residues were found in any muscle or milk samples (<0.01 mg/kg).

Table 35. Residues in dairy cattle dosed with teflubenzuron (Cameron and Puglis, 1989a).

Group	Cow	Teflubenzuron, mg/kg					
		Kidney	Liver	Muscle	Peritoneal fat	Subcutaneous fat	Milk
A (control)	1	0.015	<0.01	<0.01	0.026	<0.01	<0.01
	2	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	3	<0.01	0.017	<0.01	0.024	<0.01	<0.01
B (10 ppm)	4	0.018	0.025	<0.01	0.015	<0.01	<0.01
	5	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	6	<0.01	<0.01	<0.01	0.028	<0.01	<0.01
C (30 ppm)	7	<0.01	<0.01	<0.01	0.015	<0.01	<0.01
	8	<0.01	<0.01	<0.01	0.017	<0.01	<0.01
	9	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
D (100 ppm)	10	<0.01	<0.01	<0.01	0.011	0.01	<0.01
	11	<0.01	<0.01	<0.01	0.015	<0.01	<0.01
	12	0.017	<0.01	<0.01	0.015	<0.01	<0.01
7 days withdrawal	13	0.041	<0.01	<0.01	0.016	<0.01	<0.01
14 days withdrawal	14	<0.01	<0.01	<0.01	0.020	0.016	<0.01

Poultry (Tables 36 and 37). Groups of ten domestic hens were fed teflubenzuron at levels of 0.5 or 1.5 ppm and a group of 30 at 5 ppm in the diet for 28 days by Cameron and Puglis (1989b). An 18-day acclimatization period was followed by a 28-day treatment period and 7- and 14-day withdrawal periods (two groups of birds treated with 5 ppm). Eggs were collected for analysis. On day 28 ten hens from each group were slaughtered and the remaining hens in the highest dose group placed on a residue-free diet and slaughtered 7 or 14 days later. Samples of eggs and tissues were analysed for teflubenzuron and liver samples also for the metabolite 1-(3,5-dichloro-2,4-difluorophenyl)-3-(2,6-difluoro-3-hydroxybenzoyl)urea (E-115).

Table 36 shows the results of the analyses of eggs. Residues of teflubenzuron were detected in all treated groups and a dose-related trend was observed. Residues above the LOD (0.01 mg/kg) were first measured at day 14 in the 0.5 and 1.5 ppm groups and at day 3 in the high-dose group. Residues in the highest dose group declined during the withdrawal period and

were below the LOD after 42 days .

Table 36. Residues of teflubenzuron in eggs (Cameron and Puglis, 1989b).

Day	Teflubenzuron, mg/kg			
	Dose group A (control)	Dose group B (0.5 ppm)	Dose group C (1.5 ppm)	Dose group D (5 ppm)
-1	<0.01	<0.01	<0.01	<0.01
3	-	-	-	0.03
5	-	-	-	0.11
7	<0.01	<0.01	<0.01	0.14
10	-	-	-	0.16
14	<0.01	0.04	0.06	0.28
17	-	-	-	0.29
21	<0.01	0.03	0.08	0.20
24	-	-	-	0.29
26	-	-	-	0.30
28	<0.01	0.03	0.08	0.22
30	-	-	-	0.17
35	-	-	-	0.08
42	-	-	-	<0.01

The results of the tissue analyses are given in Table 37. Teflubenzuron was found in all types of tissue analysed, at levels which increased with the dose. The highest concentrations occurred in abdominal fat (0.7 mg/kg in the highest dose group).

The results indicated that residues of teflubenzuron in the liver persisted after 7 or 14 days withdrawal, although it should be noted that high residues were found in the livers of control birds as well. In other tissues residues of teflubenzuron declined when treatment was stopped. Residues of the metabolite E-115 in liver samples were below the LOD (<0.05 mg/kg).

Table 37. Residues in hens dosed with teflubenzuron (Cameron and Puglis, 1989b).

Group	Teflubenzuron, mg/kg, mean				
	Kidney	Liver	Muscle	Abdominal fat	Subcutaneous fat (skin + underlying fat)
A (control)	-	0.37	<0.01	<0.01	<0.01
B (0.5 ppm)	0.015	0.041	<0.01	0.077	0.028
C (1.5 ppm)	0.016	0.043	0.014	0.23	0.081
D (5 ppm)	0.036	0.081	0.038	0.70	0.32
7 days withdrawal	<0.01	0.086	<0.01	0.016	<0.01
14 days withdrawal	0.014	0.092	<0.01	<0.01	<0.01

FATE OF RESIDUES IN STORAGE AND PROCESSING

In processing

The results of processing studies are summarized in Table 38.

Apples. Processing studies were carried out in the USA and Germany. In one of the two US trials residues were found in whole fruit and wet pomace, and were concentrated in dry pomace; residues in juice were <0.05 mg/kg. At a fivefold application rate residues of 0.06-0.07 mg/kg were found in juice. In Germany residues were found in the whole fruit and washed whole fruit, and were concentrated in pomace and further concentrated in dried apples. Residues were lowered in apple sauce and below the LOD (<0.01 mg/kg) in juice.

Plums and cherries. Plums were processed to jam and dried prunes in a study in Germany. Residues were <0.01 mg/kg in destoned fruit and jam, and low (0.02 mg/kg) in prunes.

Cherries treated twice with teflubenzuron at a rate of 0.16 kg ai/ha were processed into preserve, jam and juice. Residues in these commodities were respectively 0.4, 0.3 and 0.7 times those in the unprocessed fruit and ranged from 0.12 to 0.21 mg/kg.

Grapes. Grapes were used to make juice and wine in Germany. The residues in the grapes were 0.12-0.26 mg/kg, but no residues were measurable in any of the corresponding juice or wine samples where the LOD was 0.01 or 0.05 mg/kg.

Potatoes. In two studies in the USA potatoes were treated 5 times at 0.034 or 0.17 kg ai/ha and processed to chips and French fries. Residues were <0.05 mg/kg in all samples from the lower rate. After the fivefold application rate, residues were still <0.05 mg/kg in tubers and chips, and <0.05 and 0.1 mg/kg in fries.

Soya beans. A single processing study was carried out on soya beans in the USA. Beans were treated once with 0.34 kg ai/ha and sampled 28 days after treatment. The residues were <0.05 mg/kg in the unprocessed seeds, beans and hulls, and 0.08 mg/kg in the meat. After processing to the oils, the residue in crude oil was <0.05 mg/kg, in refined oil 0.06 mg/kg and in refined bleached oil <0.05 and 0.14 mg/kg.

Cotton seed. Three processing studies were conducted in Guatemala and Mexico. Cotton plants were treated 12-15 times with teflubenzuron at rates of 0.039 to 0.08 kg ai/ha. The residues of teflubenzuron in the seeds, refined oil and presscake were below the LOD (<0.01 or <0.05 mg/kg). Fibre contained residues between 0.25 and 2.1 mg/kg and residues in the raw oil were at or below the LOD of 0.1 mg/kg.

Tomatoes. A study was carried out in the USA. The residues in the raw tomatoes were not given so transfer factors could not be derived, but residues in the juice were below the LOD (0.05 mg/kg) and the concentrate and the purée contained residues between 0.07 and 0.11 mg/kg. The highest residues were in the pomace at 0.94-1.2 mg/kg.

Table 38. Residues of teflubenzuron in processed commodities.

Commodity Country	Rate, kg ai/ha	No. of applications	PHI, days	Sample	Residues, mg/kg	Report No.
Apples	0.11	4	30	fruit	0.1	HAS A025.001

Commodity Country	Rate, kg ai/ha	No. of applications	PHI, days	Sample	Residues, mg/kg	Report No.
USA				fruit wet pomace wet pomace juice juice dry pomace dry pomace	0.1 0.13 0.16 <0.05 <0.05 2.5 1.9	
Apples USA	0.56	4	30	fruit fruit wet pomace wet pomace juice juice dry pomace dry pomace	<0.05 <0.05 0.4 0.51 0.07 0.06 6.2 5.8	HAS A025.001
Apple Germany	0.16	3	14	fruit washed fruit juice pomace sauce dried apples	0.12 0.19 <0.01 0.46 0.03 1.4	SHE-9308
Plums Germany	0.16	2	21	fruit destoned jam prunes	<0.01 <0.01 0.02	SHTR.93.009
Cherries Germany	0.16	2	21	fruit destoned preserve juice jam	0.31 0.12 0.21 0.1	SHTR.93.010
Grapes Germany	0.18	2	42	fruit juice wine	0.12 <0.05 <0.05	C 82 14 72
Grapes Germany	0.16/2.0	1/1	35	fruit juice wine	0.26 <0.01 <0.01	C 83 27 72
Grapes Germany	0.16/2.0	1/1	56	fruit juice wine	0.16 <0.01 <0.01	C 83 27 03 01
Potatoes USA	0.034	5	24	tuber tuber chips chips french fries french fries	<0.05 <0.05 <0.05 <0.05 <0.05 <0.05	HAS A025.001
Potatoes USA	0.17	5	24	tuber tuber chips	<0.05 <0.05 <0.05	HAS A025.001

Commodity Country	Rate, kg ai/ha	No. of applications	PHI, days	Sample	Residues, mg/kg	Report No.
				chips french fries french fries	<0.05 0.1 <0.05	
Soya beans USA	0.34	1	28	seeds beans hulls meat crude oil refined oil refined bleached oil refined bleached oil	<0.05 <0.05 <0.05 0.08 <0.05 0.06 <0.05 0.14	HAS A025.001
Cotton Guatemala	0.039	14	6	seeds raw oil refined oil presscake fibre	<0.05 <0.1 <0.1 <0.05 0.93	GTM84100301
Cotton Guatemala	0.039	15	8	seeds raw oil refined oil presscake fibre	<0.05 <0.1 <0.1 <0.05 2.1	GTM85100101
Cotton Mexico	0.06-0.08	12	18	seeds raw oil refined oil presscake fibre	<0.05 0.1 <0.1 <0.05 0.25	MEX84100101
Tomatoes USA	0.056	5	0	concentrate concentrate pomace pomace juice juice puree puree	0.1 0.11 1.2 0.94 <0.05 <0.05 0.07 0.08	HAS A025.001

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

No information was available.

NATIONAL MAXIMUM RESIDUE LIMITS

The following national MRLs were reported to the Meeting.

Residue definition: teflubenzuron.

Country	Commodity	MRL, mg/kg
Belgium	Mushrooms	0.05
Brazil	Coffee beans	0.5
	Cotton	0.01
	Soya bean	0.01
	Tomato	0.1
France	Apple	0.5
	Pear	0.5
	Quince	0.5
Germany	Berries (wild)	0.2
	Mushrooms (wild)	0.2
	Orange	0.5
	Other plant commodities	0.05
	Pome fruits	1
Italy	Apple	1
	Cabbage	0.5
	Egg plant	0.5
	Grapes (wine)	1 (0.01)
	Maize (grain)	0.1
	Mushrooms	0.2
	Nectarine	1
	Peach	1
	Pear	1
	Peppers	0.5
	Potato	0.1
Kenya	Coffee beans	0.5
Netherlands	Apple	0.5
	Broccoli	0.05
	Brussels sprouts	0.5
	Cabbage, Red	0.05
	Cabbage, White	0.05

Country	Commodity	MRL, mg/kg
	Chinese cabbage	0.5
	Cauliflower	0.05
	Cucumber	0.2
	Cucurbits	0.2
	Egg plant	0.5
	Gherkin	0.2
	Melon	0.2
	Pear	0.5
	Peppers	0.5
	Squash, Summer	0.2
	Tomato	0.5
Spain	Apple	0.5
	Cucumber	0.2
	Cucurbits	0.2
	Egg plant	0.5
	Grapes	0.5
	Pear	0.5
	Peppers	0.5
	Potato	0.05
	Tomato	0.5
Switzerland	Apple	0.3
	Cabbage	0.05
	Cabbage, Savoy	0.05
	Cereal grains	0.05
	Grapes	0.3
	Pear	0.3
	Potato	0.05
	Stone fruits	0.3
South Africa	Citrus fruits	0.5
	Lychee	0.5

APPRAISAL

Teflubenzuron is an acylurea insecticide whose major use is for the control of a wide range of insect pests (*lepidopterous* and *coleopterous* larvae being most sensitive) and some mites in fruits, vegetables, cereals, nuts and seeds. At the request of the manufacturer, the compound was removed from the review schedule of the FAO Panel of the 1994 JMPR and its residue aspects were reviewed for the first time by the present Meeting.

Teflubenzuron is formulated as suspension concentrates containing 50 or 150 g ai/l. The active ingredient is a crystalline solid, virtually insoluble in water, soluble in medium-polarity solvents, not hydrolysed at pH 5 but hydrolysed at room temperature and pH 7 and 9 with half-lives of 8 months and 8 days respectively.

The fate of residues has been studied in animals, plants and soil.

Studies on rats, lactating goats and laying hens showed poor absorption from the gastrointestinal tract, rapid elimination mainly in the faeces or excreta, excretion largely as the unchanged parent compound, and in the case of goats no accumulation in any organ or tissue, or milk.

In goats and hens, 96-99% of administered doses were eliminated in the faeces or excreta and most of the radioactivity was associated with the parent compound. That portion of a dose which is absorbed appears to be metabolized in the liver and conjugated before elimination, mainly in the bile. In hens only, the absorbed fraction appears to be passed into body tissues, especially fatty tissues and egg yolk. The major part of the residue in fat and egg yolk was identified as teflubenzuron. The elimination of radioactivity in the milk of goats accounted for 0.03% of the total administered dose.

Studies of plant metabolism with foliar applications of teflubenzuron to apple trees, potato plants and cotton plants have shown that the insecticide does not penetrate into leaves, fruit or potato tubers. More than 98% of the extractable radioactivity was in the unchanged compound and was situated on the surface of the treated plant part. It was concluded that there is no systemic transport and no metabolism.

In a study on spinach, the residue was also almost all (99%) on the surface. The parent compound amounted to 95% of the total radioactive residue at day 0 and 77% at day 15. The fact that the radioactivity was almost completely removed by surface extraction indicates that any significant degradation is photolytic rather than metabolic.

Investigation of the degradation of teflubenzuron in soil showed that microbiological activity is of primary importance. In very humic soil degradation was more rapid than in sandy loam, with a half-life of two weeks in humic sand and six weeks in sandy loam soil. In sandy loam, teflubenzuron was degraded about six times as rapidly under anaerobic as under aerobic conditions.

Under both aerobic and anaerobic conditions, 3,5-dichloro-2,4-difluorophenylurea and 3,5-dichloro-2,4-difluoroaniline were the major products.

The adsorption and desorption of teflubenzuron was studied in four different types of soil. It was found that sand adsorbed 96.9%, sandy loam 98.8%, silt loam 99.1%, and clay loam 99.4% of the amount dissolved in the aqueous control sample after a 6-h contact period, and 6.1%, 3.7%, 1.3%

and 1.3% of the adsorbed radioactivity respectively could be desorbed again during two desorption periods of 24 h each.

Teflubenzuron itself shows practically no tendency to migrate once it is applied to soil. This is attributable to the very low solubility of the compound in water, very slight leaching and high adsorption to all types of soil tested.

Residues were not significant in rotational crops. After applying [^{14}C]teflubenzuron (0.5 kg ai/ha) and ageing the soil for 30, 120 or 360 days, the total radioactive residues expressed as teflubenzuron equivalents were 0.007, 0.006 and 0.002 mg/kg in head lettuce, 0.005, 0.003 and 0.002 mg/kg in wheat grain, 0.24, 0.088 and 0.035 mg/kg in wheat straw, and 0.026, 0.013 and 0.005 mg/kg in carrots. The results show that rotational crops, with the exception of cereal straw, will not contain residues above 0.05 mg/kg.

The biodegradation of [^{14}C]teflubenzuron was determined in two water/sediment systems. The half-life was 6-7 weeks. Two major degradation products were found; one was identified as 3,5-dichloro-2,4-difluorophenylurea.

Analytical methods are available for the determination of teflubenzuron residues in plant and animal materials, soil, water and air. Teflubenzuron is extracted from plants with acetone, from soil with an acetone/water mixture and from animal products such as muscle, liver, kidney, fat, skin, milk and eggs with acetonitrile or methanol. It is extracted from water on a C_{18}-"Bondelut" solid-phase column. Clean-up is carried out by solvent partition followed by gel-permeation chromatography and/or silica gel column chromatography. The residue is determined by reversed-phase HPLC with ultraviolet detection at 254 nm or by capillary gas chromatography with mass-selective detection. The limits of determination were 0.01 mg/kg in plants, soil and animal products and 0.0001 mg/l in water. In some supervised trials (e.g. on potatoes), an analytical method with an LOD of 0.05 mg/kg was used. For the analysis of air, the air is sucked through a Tenax or XAD column and the adsorbed teflubenzuron is eluted and determined by reversed-phase HPLC with UV detection or by GLC with a mass-selective detector as a confirmatory method. The LOD was 10 µg/m^3. The stability of stored analytical samples of teflubenzuron in apples, pears, potatoes and cabbage was investigated over a 3-year period. Losses of the insecticide were from about 6% from cabbage to 25% from apples.

Because the residues in plants and animal products are generally mostly the parent compound, the Meeting concluded that for both regulatory and risk assessment purposes the residue should be defined as teflubenzuron. The log P_{OW} of 4.56 and the results of a feeding study on laying hens, with residues in eggs and fat, indicate the fat-soluble nature of teflubenzuron. In contrast to the results with hens, a feeding study on dairy cattle showed little or no transfer of the pesticide from animal feed in milk, fat and tissues.

Definition of the residue for compliance with MRLs and for estimation of dietary intake: teflubenzuron

The residue is fat-soluble.

Supervised residue trials gave the following results.

Citrus fruits. The use of teflubenzuron is registered in the United Arab Emirates and South Africa, where 1-2 treatments with a spray concentration of 0.0038 or 0.003 kg ai/hl and PHIs of 7 or 30 days are recommended respectively. In Saudi Arabian GAP the spray concentration is 0.011 kg ai/hl with

a 21-day PHI. In total, 20 supervised trials were carried out in Brazil, South Africa and the USA.

The whole fruit was analysed only in three US trials (one on grapefruit, two on oranges) but there is no registered use in the USA. No residue was found in the whole fruit (<0.05 mg/kg), 45 and 76 days after three spray treatments (0.005-0.006 kg ai/hl, 0.11 kg ai/ha).

One trial in Brazil and three in South Africa approximated South African GAP. The residues were <0.01, <0.05 (2) and 0.1 mg/kg in the pulp and 0.24, 0.26, 0.34 and 0.47 mg/kg in the peel at PHIs of 25-35 days. In three trials, samples were also analysed after 6 or 7 days, the PHI in the United Arab Emirates. The residues were all <0.05 mg/kg in the pulp and 0.36, 0.37 and 0.39 mg/kg in the peel. Calculation of the residues in the whole fruit was not possible because no further data were reported.

The Meeting concluded that no acceptable residue data on a whole fruit basis had been submitted and could not estimate a maximum residue level for teflubenzuron in citrus fruits.

Pome fruits. Teflubenzuron is registered for use on apples and pears in Europe (France, Greece, Italy, The Netherlands, Poland, Portugal, Spain, Switzerland), Africa (Jordan, The United Arab Emirates) and Argentina (apples only).

The residue data after 1-4 treatments of apples with 0.11-0.21 kg ai/ha (0.011 kg ai/hl) at PHIs of 24-28 days in Germany and the UK could be related to Dutch GAP (1-3 x 0.11-0.16 kg ai/ha, 0.011 kg ai/hl, 28-day PHI). The residues in ten German and two UK trials were 0.05, 0.09, 0.11, 0.19 (2), 0.23 (2), 0.24, 0.27, 0.29, 0.36 and 0.37 mg/kg.

The Italian trials on apples were evaluated with reference to the Southern European GAP (Italy, Greece and Switzerland). Two trials approximated Italian GAP with 1-3 treatments of 0.15-0.16 kg ai/ha and a PHI of 14 days. The residues were 0.23 and 0.27 mg/kg. Two other trials with 2 treatments at 0.24-0.27 kg ai/ha were close to Greek GAP (2-3 treatments, 0.21 kg ai/ha, 30-day PHI) and resulted in residues of 0.45 and 0.51 mg/kg after 28 days.

Swiss GAP (2-3 treatments, 0.3 kg ai/ha, 21-day PHI) was approximated by one Italian trial on apples and three on pears (2-3 treatments with 0.24-0.27 kg ai/ha, 21-day PHI). The residues were 0.65 mg/kg in apples, and 0.43, 0.6 and 0.71 mg/kg in pears.

Many residue trials on apples and pears were conducted in France, but they did not accord with French or other European GAP. No GAP was available for the USA so the results could not be evaluated.

The Meeting estimated a maximum residue level of 1 mg/kg for teflubenzuron in pome fruits.

The teflubenzuron residues in apples in rank order in the German and UK trials were 0.05, 0.09, 0.11, 0.19 (2), 0.23 (2), 0.24, 0.27, 0.29, 0.36 and 0.37 mg/kg. The residues from trials on apples and pears in Southern Europe belonged to another population: 0.23, 0.27, 0.43, 0.45, 0.51, 0.6, 0.65 and 0.71 mg/kg. The Meeting therefore estimated an STMR level of 0.48 mg/kg for teflubenzuron in pome fruits.

Stone fruits. Teflubenzuron is registered in Switzerland for stone fruits, in Italy for peaches and nectarines, and in Saudi Arabia for peaches with 1-3 treatments of 0.11-0.12 kg ai/ha and a 21-day PHI. In Poland, one treatment of 0.11 kg ai/ha and a PHI of 28 days are recommended for orchards.

Three German trials on cherries with 2 x 0.16 kg ai/ha, approximately Swiss GAP, resulted in residues of 0.24 (2) and 0.25 mg/kg.

The Meeting concluded that there were not enough data to estimate a maximum residue level for teflubenzuron in cherries.

Trials on plums which approximated Swiss GAP were available from Germany (5) and Italy (6). After two applications of 0.16 kg ai/ha in Germany residues of teflubenzuron in samples taken at day 21 were <0.01 (2), 0.01 and 0.04 (2) mg/kg. After two applications of 0.12 kg ai/ha in Italy the residues at 21-30 days were 0.03 (2), 0.04 (2) and 0.08 (2) mg/kg.

The Meeting estimated a maximum residue level of 0.1 mg/kg for teflubenzuron in plums.

The residues from all the relevant trials in rank order were <0.01 (2), 0.01, 0.03 (2), 0.04 (4) and 0.08 (2) mg/kg, with a median value of 0.04 mg/kg. The Meeting estimated an STMR level of 0.04 mg/kg for teflubenzuron in plums.

Two trials on nectarines were carried out in Italy. After two applications of 0.12 kg ai/ha the residues in the fruits were 0.08 mg/kg and 0.04 mg/kg at 35 and 56 days after the last treatment respectively. No information on residues at the approved PHI of 21 days was available.

In eight trials in France and two in Italy peaches were treated 1-6 times with 0.1-0.19 kg ai/ha. Four trials with 2-3 x 0.1 kg ai/ha approximated Italian and Swiss GAP and resulted in residues of 0.1, 0.13, 0.24 and 0.34 mg/kg at 20-24 days.

The Meeting could not estimate a maximum residue level for teflubenzuron in nectarines and peaches because there were insufficient data from trials according to GAP.

Berries and other small fruits. The only registered uses of teflubenzuron on berries are on grapes with 2 x 0.09-0.096 kg ai/ha in Italy, Spain (both PHIs 28 days) and Switzerland (PHI 21 days), and 2 x 0.011 kg ai/hl and a 21-day PHI in Saudi Arabia.

Trials on grapes were conducted in France, Germany and Italy between 1982 and 1984. In the 10 French trials, grapes were treated once at rates of 0.1-0.3 kg ai/ha. Residues of teflubenzuron 58-77 days after treatment ranged from <0.05 to 0.86 mg/kg. In the 5 German trials, two treatments of 0.15-0.23 kg ai/ha gave residues 21 days after the last treatment between 0.08 and 0.23 mg/kg. Residues in 5 Italian trials with 1-3 treatments of 0.18-0.27 kg ai/ha were <0.05-2.1 mg/kg at 28 days. Generally the application rates in the trials were much higher than the rates close to 0.1 kg ai/ha permitted by GAP, or the PHI was excessive.

The Meeting concluded that since no data were provided from trials according to GAP it could not estimate a maximum residue level for teflubenzuron in grapes.

Uses on other berries are not registered but eight German trials on wild raspberries, blackberries and blueberries were reported. In Germany, teflubenzuron is registered for use in the forest against larvae of *Tenthredinidae spp.* and free-eating caterpillars (1 x 0.023 kg ai/ha). As result of this, wild berries and fruits are treated unintentionally. The worst case was simulated by application of approximately twice the approved rate (0.045 kg ai/ha). In raspberries and

blackberries, the residues were <0.05 (2), 0.06 (2) and 0.09 mg/kg, ≤0.05 (4) and 0.08 mg/kg, and ≤0.05 (4) and 0.05 mg/kg at 0, 2 or 3, and 7 days respectively. The residues in blueberries in three trials, which appeared to be from a different population, were 0.08, 0.09 and 0.1 mg/kg, 0.11 (2) and 0.12 mg/kg, and 0.05, 0.06 and 0.07 mg/kg at 0, 2 and 7 days.

The Meeting accepted that there was an indirect use on wild berries, but because the data were limited and the commodities are not in international trade it did not estimate maximum residue levels for teflubenzuron on wild raspberries, blackberries or blueberries.

Persimmons. Data were available from a group of 5 trials in Korea. Persimmons were treated 2-6 times with 0.25 kg ai/ha. Residues in samples taken 3-45 days after the last treatment were 0.02-0.09 mg/kg. No GAP was available to evaluate the trials.

Kiwifruit. Four residue trials were carried out in New Zealand. The application rates were 0.094, 0.19 and 0.25 kg ai/ha. Residues determined in whole fruit 16 and 99 days after the final application were 0.23-3.6 mg/kg and 0.28 mg/kg respectively. No GAP was available to evaluate the trials.

Head cabbages. There are registered uses on red, white and Savoy cabbage in Germany (1 x 0.06 kg ai/ha, 14-day PHI), on red and white cabbage in The Netherlands (2-4 x 0.06 kg ai/ha, 14-day PHI), and on head cabbages in Indonesia (0.025 kg ai/ha, no further information), Italy (1 x 0.03 kg ai/ha, 7-day PHI), Jordan (2 x 0.0075 kg ai/hl, 14-day PHI), Poland (1 x 0.03 kg ai/ha, 14-day PHI) and Switzerland (1 x 0.045 kg ai/ha, 14-day PHI).

Four US trials on white and red cabbage, each with analyses of duplicate samples with and without wrapper leaves, were reported. Residues after applying 6 x 0.045 kg ai/ha were <0.05-0.36 mg/kg in samples with wrapper leaves and <0.05-0.11 mg/kg without wrapper leaves 14 days after treatment. One trial was carried out in Malaysia and one in the Philippines. After applying 6 or 9 x 0.045 kg ai/ha, residues were <0.05 mg/kg after 18 and 7 days respectively. In two trials in Brazil (1 x 0.015 kg ai/ha, 1 x 0.03 kg ai/ha) residues were <0.01 mg/kg 3 or 7 days after treatment. No information on GAP was available for the USA, Brazil, Malaysia or the Philippines with which to evaluate the data from the trials in these countries.

Ten trials on Savoy cabbage were conducted in Germany, 1982-1985. After applying 3 x 0.06 kg ai/ha, all residues were <0.05 mg/kg at the recommended PHI of 14 days. Residues in two UK trials on Savoy cabbage treated once according to German GAP with 0.06 kg ai/ha were 0.05 and 0.17 mg/kg at 14 days.

The Meeting estimated a maximum residue level of 0.2 mg/kg for teflubenzuron in head cabbages.

The teflubenzuron residues in the ten German and two UK trials in rank order were <0.05 (10), 0.05 and 0.17 mg/kg. The median residue was below the LOD (0.05 mg/kg). The Meeting estimated an STMR level of 0.05 mg/kg.

Broccoli. Teflubenzuron is registered for use on broccoli in The Netherlands with 2-4 treatments of 0.06 kg ai/ha and a PHI of 14 days.

Two German trials according to Dutch GAP were reported. The residues were 0.13 and 0.19 mg/kg at day 14.

The Meeting concluded that insufficient data were available to estimate a maximum residue level for teflubenzuron in broccoli.

Brussels sprouts. Teflubenzuron is registered in The Netherlands, where 6-8 treatments of 0.09 kg ai/ha with a PHI of 14 days are recommended.

Eight residue trials were conducted in The Netherlands with 4, 5 or 6 applications, four at 0.06 and four at 0.09 kg ai/ha. After treatment with 0.09 kg ai/ha, the residues were 0.12 to 0.48 mg/kg at 14 days. The residues after 14 days in the 4 trials with 0.06 kg ai/ha were of the same order, 0.1-0.28 mg/kg, and support the conclusion that a maximum residue level of 0.5 mg/kg is appropriate. The residues in rank order were 0.1, 0.12, 0.15, 0.18, 0.24, 0.28, 0.39 and 0.48 mg/kg, giving a median of 0.21 mg/kg.

The Meeting estimated an STMR level of 0.21 mg/kg and a maximum residue level of 0.5 mg/kg for Brussels sprouts.

Cucumbers. Teflubenzuron is registered for field and glasshouse uses on cucurbits in Spain (2-3 x 0.18 kg ai/ha, 3-day PHI), for glasshouse use on cucumbers and gherkins in The Netherlands (3-5 x 0.23 kg ai/ha, 3-day PHI), for field treatments of cucumbers and gherkins (2 x 0.0075 kg ai/hl, 3-day PHI) in Jordan, and for field use on cucumbers with 2 x 0.011 kg ai/hl in Saudi Arabia.

Three indoor trials were carried out in Germany. The residues after 3 days were 0.03 and 0.07 mg/kg from approximately 3 x 0.09 kg ai/ha, and 0.14 mg/kg from 3 x 0.18 kg. In two field trials in Italy, where 3 x 0.075 kg ai/ha were applied, the residues after 3 days were 0.02 and 0.19 mg/kg.

The Meeting concluded that there were insufficient data from trials according to GAP to estimate a maximum residue level for teflubenzuron in cucumbers.

Peppers. Teflubenzuron is registered for field use on sweet peppers in Italy (1-2 x 0.08 kg ai/ha, 10-day PHI), Jordan (2 x 0.0075 kg ai/hl), Saudi Arabia (2 x 0.011 kg ai/hl) and Spain (2-3 x 0.18 kg ai/hl, 3-day PHI), and on chilli peppers in Indonesia at 0.1 kg ai/ha. Glasshouse use on sweet peppers is registered in The Netherlands with 3-5 applications and in Spain with 2 or 3 applications of 0.23 kg ai/ha and a PHI of 3 days.

The Meeting reviewed 6 trials from Italy, 4 of them (all in 1988) according to Italian GAP with 1 treatment of 0.075 kg ai/ha. The residues were 0.09, 0.1 and 0.11 mg/kg (2) 10 days after application.

Trials were reported from Korea but no information on GAP was available for their evaluation.

The Meeting concluded that only 4 trials according to GAP, carried out in one year, were insufficient to estimate a maximum residue level for peppers, which are a major crop.

Egg plants. Teflubenzuron is registered for field use on egg plants in Italy with 1-2 x 0.022-0.024 kg ai/ha, 10-day PHI. The GAP for field use in Jordan, Saudi Arabia and Spain, and for glasshouse use in The Netherlands and Spain, is the same as for sweet peppers.

The Meeting reviewed 6 outdoor trials from Italy. In four of them which complied with Italian GAP with 1 treatment at 0.023 kg ai/ha the residues were all <0.01 mg/kg 10 days after application. No residue data were available for glasshouse use.

The Meeting concluded that the data were insufficient to estimate a maximum residue level.

Tomatoes. Teflubenzuron is currently registered for glasshouse use in The Netherlands (3-5 x 0.23 kg ai/ha, 3-day PHI), and for glasshouse and field use in Spain (2-3 x 0.18-0.23 kg ai/ha, 3-day PHI). In Brazil and Paraguay, 5-8 field treatments at 0.038 kg ai/ha with a PHI of 7 days are recommended. Further registered field uses exist in Argentina, Colombia, Ecuador, and Jordan.

Four trials were carried out in the USA. Residues from <0.05 to 0.1 mg/kg were found 3 days after treatment with 5 x 0.028-0.056 kg ai/ha. No information on GAP was available for the USA with which to evaluate the data.

Two of 6 field trials in Brazil, with 5 x 0.045 kg ai/ha, approximated GAP and resulted in residues of 0.05 and 0.06 mg/kg 6 days after treatment, two trials at twice this rate gave residues of 0.08 and 0.15 mg/kg, and in the third pair of trials (3 x 0.03 kg ai/ha) 0.1 and 0.12 mg/kg were found at 7 days. Two Italian field trials at 4 x 0.075 kg ai/ha, with residues of 0.1 and 0.28 mg/kg at day 2, could not be evaluated against Spanish GAP because the rate was too low.

Three indoor trials with 3 or 4 treatments at 0.09-0.17 kg ai/ha were reported from Germany, but there are no registered uses there. One of them could be evaluated against Dutch GAP and showed a residue of 0.47 mg/kg 3 days after application. Two of three UK trials at 4 x 0.17 kg ai/ha, 5 x 1.14-1.17 kg ai/ha, and 5 x 0.23 kg ai/ha approximated Dutch and Spanish glasshouse uses and showed residues of 0.2 and 0.36 mg/kg 3 days after treatment.

The Meeting concluded that there were insufficient data from trials according to GAP for field and glasshouse uses to estimate a maximum residue level for teflubenzuron in tomatoes.

Mushrooms. Uses on cultivated mushrooms exist in Belgium (3 kg ai/ha, 14-day PHI) and Italy (1 x 4.8-6 kg ai/ha, 45-day PHI).

One trial with four replicated plots of cultivated mushrooms was conducted in The Netherlands. The residues were all <0.05 mg/kg 25 days after applying 2 x 4.9 kg ai/ha.

The Meeting concluded that the data from trials according to GAP were insufficient to estimate a maximum residue level for teflubenzuron in cultivated mushrooms.

In 3 trials on wild mushrooms in Germany with 1 x 0.045 kg ai/ha the residues on days 0, 1 and 2 were all <0.05 mg/kg in two trials and 0.07, 0.07 and 0.05 mg/kg respectively in the third.

The Meeting accepted that there was an indirect use on wild mushrooms from the German use of teflubenzuron in forests (1 x 0.023 kg ai/ha), but because the data were limited and wild mushrooms are not in international trade it did not estimate a maximum residue level for wild mushrooms.

Chinese cabbage. Teflubenzuron is currently registered only in The Netherlands. It is recommended for field use at a rate of 0.06 kg ai/ha 2-4 times a season with a PHI of 14 days.

Two trials were carried out in the Netherlands. After applying 1 x 0.06 or 0.09 kg ai/ha the residues were 0.22 and 0.31 mg/kg at day 14.

Trials were also conducted in Malaysia and the Philippines but no information on GAP for Asian countries was available from which to evaluate the results.

The Meeting concluded that there were insufficient data from trials according to GAP to estimate a maximum residue level.

Peas. One trial with 2 x 0.045 kg ai/hl was conducted in France. The residues were 0.19 mg/kg in peas with pods and <0.05 mg/kg in the peas after 21 days. No information on GAP was available to evaluate the trial.

Soya beans. Teflubenzuron is registered for the use on soya beans in Brazil (0.0075-0.023 kg ai/ha) and Paraguay (2-3 x 0.0075 kg ai/ha) with PHIs of 30 days.

Six trials were carried out in Brazil (1-2 x 0.015-0.09 kg ai/ha). Four of them (2 x 0.015-0.03 kg ai/ha) were within the wide range of Brazilian GAP but were at only 2 sites. The residues were <0.01 mg/kg 30 days after treatment.

One study with replicated trials at various application rates was carried out in the USA. Residues in the seeds after 2 treatments with 0.034 kg ai/ha (0.015 kg ai/hl) and a PHI of 30 days were <0.05-0.34 mg/kg. No information on relevant GAP was available.

The Meeting concluded that the data from trials according to GAP were insufficient to estimate a maximum residue level for teflubenzuron in soya beans, a major crop.

Potatoes. Teflubenzuron is registered for use on potatoes in Germany (1 x 0.045 kg ai/ha), Italy (1-2 x 0.024 kg ai/ha), Poland (1-2 x 0.038 kg ai/ha), Saudi Arabia (2 x 0.011 kg ai/hl), Spain (1-2 x 0.022 kg ai/ha) and Switzerland (0.038 kg ai/ha). The PHIs range from 14 to 28 days.

Data were available from 11 trials in Brazil, France, Germany, Italy, Slovakia and the USA, but most of them were not according to GAP or no information on relevant GAP was available. Only 2 German trials (2 x 0.052 kg ai/ha, 14-day PHI) approximated GAP. Teflubenzuron was not detected in any of the samples (<0.05 mg/kg), even from exaggerated application rates at short PHIs.

It was concluded from a study of teflubenzuron metabolism and kinetics in potato plants that teflubenzuron does not penetrate into the leaves, stems or tubers if it is sprayed on the foliage. No systemic transport or metabolism occurs in the plants.

In view of the results of the metabolism study and the absence of residues in the trials, the Meeting concluded that sufficient information was available to estimate a maximum residue level for potatoes of 0.05* mg/kg as being a practical limit of determination, and estimated an STMR level of nil for teflubenzuron in potatoes.

Maize. Uses of teflubenzuron exist in Colombia and Ecuador with 2 treatments of 0.045 kg ai/ha and in Ecuador a PHI of 21 days. The insecticide is registered in Italy for use on maize at 2 x 0.15-0.16 kg ai/ha with a PHI of 28 days, and in Switzerland for cereals at 0.06 kg ai/ha with a 42-day PHI.

Supervised trials were reported from France (6), Germany (10), Italy (2) and Slovakia (3),

but information on GAP was available only from Italy. None of the northern European trials approximated Swiss GAP nor the French trials Italian GAP (they were at exaggerated rates and/or longer PHIs). Two trials in Italy with 2 x 0.15 kg ai/ha corresponded with GAP. At day 28 the residues were 3.6 and 3.9 mg/kg in the whole plant and <0.05 mg/kg in the grain.

The data from trials according to GAP were insufficient to estimate a maximum residue level.

Cotton seed. Teflubenzuron is registered for uses on cotton in Argentina (2 x 0.011 kg ai/ha, 21-day PHI), Brazil (0.0075-0.1 kg ai/ha, 30-day PHI), Paraguay (2-3 x 0.0075 kg ai/ha, 30-day PHI), Colombia (2 x 0.019 kg ai/ha), Ecuador (2 x 0.019-0.045 kg ai/ha) and Guatemala (2-3 x 0.075 kg ai/ha).

Seven trials in Latin America were within the wide range of Brazilian GAP. No residues above the LODs of 0.01 or 0.05 mg/kg were found in 4 Brazilian trials (2 x 0.03 or 2 x 0.06 kg ai/ha, PHI 31 days), 2 trials in Guatemala (14-15 x 0.039 kg ai/ha, PHI 6-8 days), or 1 trial in Mexico (12 x 0.06-0.08 kg ai/ha, PHI 18 days). The residues in rank order were <0.01 (4), <0.05 mg/kg (3).

In 9 US trials the residues were <0.05, 0.07, 0.08, 0.11, 0.24 and 13 mg/kg from 12 x 0.045 kg ai/ha, and 0.78, 3.1 and 4.6 mg/kg from 12 x 0.45 kg ai/ha 14 or 18 days after treatment. No information on GAP was available to evaluate the trials.

The results of the 6 US trials with 12 treatments at 0.045 kg ai/ha are inconsistent with the "nil" residues in Latin America with similar application rates. For this reason and because Brazilian GAP is reported to have such a wide range of application rates, the Meeting could not estimate a maximum residue level for cotton seed.

Coffee beans. The registered uses of teflubenzuron on coffee plants are in Brazil at 0.038 kg ai/ha and in Kenya with 1 or 2 treatments at 0.11 kg ai/ha, with PHIs of 30 days in both countries.

Two residue trials were conducted in Brazil. After 2 applications of 0.075 or 0.15 kg ai/ha and a PHI of 35 days, the residues were 0.6 and 1.7 mg/kg respectively.

There were insufficient data to estimate a maximum residue level.

Alfalfa forage and green grass. Two supervised trials on alfalfa and 1 on green grass were carried out in Italy (1 x 0.075 kg ai/ha). The residues in alfalfa forage declined from 1.4 and 2 mg/kg at day 3 to 0.18 and 1.2 mg/kg at day 28 after application. The residue is grass was 0.71 mg/kg at 28 days. No information on GAP was available to evaluate the trials.

Soya bean forage and hay. Teflubenzuron is registered for use on soya beans in Brazil (0.0075-0.023 kg ai/ha) and Paraguay (2-3 x 0.0075 kg ai/ha) with PHIs of 30 days.

Eight trials were carried out in the USA with 1 or 2 x 0.022 kg ai/ha. The residues in the forage and hay were 0.17-0.56 mg/kg and 0.19-1.3 mg/kg, respectively, at day 14. No information on GAP was available to evaluate the results.

Because no residue data were submitted from South America, the Meeting could not estimate a maximum residue level for teflubenzuron in soya bean forage or hay.

Animal products. When dairy cows were fed with feed containing 10, 30 or 100 ppm teflubenzuron

for 28 days, the residues of teflubenzuron in subcutaneous fat, peritoneal fat, liver, kidney and skeletal muscle were <0.05 mg/kg. Low concentrations of teflubenzuron were detected in the liver or kidney of some animals (0.015-0.041 mg/kg). There was no indication of any correlation with the dose level, or with the withdrawal period in the high-dose group. Two apparent residues were found in the control group. Residues of the metabolite E-115, 1-(3,5-dichloro-2,4-difluorophenyl)-3-(2,6-difluoro-3-hydroxybenzoyl)urea, in liver samples were <0.05 mg/kg (the LOD).

Only low residues of teflubenzuron occurred in peritoneal fat (0.011-0.028 mg/kg). Residues at or close to the LOD (0.01 mg/kg) were recorded in subcutaneous fat in the high-dose group, but 0.016 mg/kg was found 14 days after withdrawal.

No residues were found in any muscle or milk samples (<0.01 mg/kg).

An obstacle to estimating maximum residues for meat and milk is the lack of sufficient residue data on typical feed items (green forage, fruit pomace, cereal grains, pulses, oil seed), for which no MRLs could be recommended. The Meeting concluded that in the absence of such recommendations no maximum residues for teflubenzuron in products of ruminant origin could be estimated.

Laying hens were fed teflubenzuron at levels of 0.5 ppm, 1.5 ppm and 5 ppm in the diet for 28 days. Residues were detected in the eggs of all treated groups and a dose-related trend was observed. Values above the LOD of 0.01 mg/kg were first measured at day 14 in the groups treated with 0.5 ppm (0.04 mg/kg) and 1.5 ppm (0.06 mg/kg). In the high-dose group the first measured mean residue was 0.03 mg/kg at day 3, a mean maximum of 0.30 mg/kg was reached at day 26 and the residues declined during the withdrawal period. They were below the LOD after 42 days in the highest dose group.

Residues of teflubenzuron were found in all types of tissue analysed. The highest concentrations occurred in abdominal fat (0.7 mg/kg in the highest dose group). In the 0.5 ppm group residues were <0.01 mg/kg in muscle and 0.028 mg/kg in subcutaneous fat. Residues in the liver of hens kept on a teflubenzuron-free diet after dosing persisted for 7 or 14 days after withdrawal. Residues of the metabolite E-115 in liver samples were below the LOD (<0.05 mg/kg). High positive results were found in the livers of control birds.

Again the lack of residue data on typical poultry feed items (cereals, pulses) prevented the Meeting from estimating maximum residue levels for teflubenzuron in poultry commodities.

Processing studies on apples, plums, cherries, grapes, potatoes, tomatoes, soya beans and cotton were made available to the Meeting. With most of these crops (apples, plums, tomatoes, potatoes, soya beans, cotton seed) even exaggerated application rates did not produce sufficiently high residues in the raw commodity to estimate transfer factors. The Meeting was also unable to confirm the reported results for the processed products in the absence of details of the processing procedures.

In general, residues were reduced in canning fruit and processing to juice and wine but concentrated during soya bean oil production, by drying fruits, and in producing pomace. This is to be expected because of the fat-soluble nature of the active ingredient or the reduced water content of the processed products.

The only information on residues in the edible portions of food commodities came from

separate analyses of the pulp and peel of citrus fruit. Although the data were insufficient to estimate a maximum residue level for citrus, they indicated that residues in citrus pulp are likely to be less than 10% of the residue in the peel.

No information was provided on residues in commodities in commerce or at consumption.

RECOMMENDATIONS

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 estimation of dietary intake): teflubenzuron.

The residue is fat-soluble.

Commodity		Recommended MRL, mg/kg	Estimated STMR, mg/kg	PHI on which estimates are based, days
CCN	Name			
VB 0402	Brussels sprouts	0.5	0.21	14
VB 0041	Cabbages, Head	0.2	0.05	14
FS 0014	Plums (including Prunes)	0.1	0.04	21
FP 0009	Pome fruits	1	0.48	14-30
VR 0589	Potatoes	0.05*	0	7-35

FURTHER WORK OR INFORMATION

Desirable

1. Physical and chemical properties of the pure active ingredient.

2. Further processing studies on apples and plums to allow the calculation of transfer factors.

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THIRAM (DITHIOCARBAMATES, 105)

EXPLANATION

Thiram was originally evaluated in 1965 (toxicology) and 1967 (toxicology and residues) and is included in the dithiocarbamate group of compounds.

Thiram is a protective fungicide used as a foliar treatment on fruits, vegetables and ornamentals to control *Botrytis* species, rust, scab and storage diseases, and as a seed treatment to control seedling blights and a number of fungi that cause "damping off" in seedlings. Thiram formulations are registered for use in many countries.

The compound was evaluated at the present Meeting within the CCPR periodic review programme.

IDENTITY

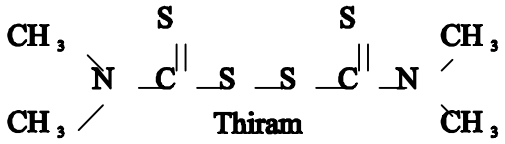
ISO common name: thiram

Chemical name

IUPAC: tetramethylthiuram disulfide
bis(dimethylthiocarbamoyl) disulfide

CA: tetramethylthioperoxydicarbonic diamide

CAS registry no: 137-26-8
CIPAC no: 24
EEC no: 205-286-2
Structural formula:



Molecular formula: $C_6H_{12}N_2S_4$

Molecular mass: 240.4

Physical and chemical properties

Pure active ingredient

Vapour pressure:	2.3 mPa at 25°C (by gas saturation method, Lemal 1985).
Melting point:	155-156°C.
Octanol/water partition coefficient:	54 ± 14. Log P_{OW} 1.73 (Lemal, 1983).
Solubility:	16.5 mg/l in water at 20° ± 1°C. 69.7 g/l in acetone at 25°C 205 g/l in chloroform at 25°C <10 g/l in ethanol at 25°C
Specific gravity:	1.36 g/cm^3 at 20°C.
Hydrolysis:	half-life <1 day at pH 9 half-life 6 days at pH 7 half-life 77 days at pH 5
Photolysis:	photodegradation half-life in water at pH 5 and 25°C is 8.8 hours

Lemal (1985) measured the vapour pressure of thiram by a gas saturation method. Nitrogen gas was passed through thiram coated on a support material with a very high surface area and maintained at 25°C, then through a cotton wool dust filter followed by traps containing methanol. The contents of the absorption traps were analysed by HPLC. The vapour pressure was calculated from the volume of nitrogen passed through the apparatus and the amount of thiram collected in the trap. In two runs the measured vapour pressure at 25°C was 2.4 and 2.2 mPa.

Lemal (1983) measured the octanol-water partition coefficient of thiram according to OECD Guideline 107 (OECD 1981). In a series of tests the values for log P_{OW} ranged from 1.568 to 1.851, with a median value of 1.782 and a mean of 1.734.

Technical material

Purity: 98.5%
Melting range: 135-146°C.

Formulations

80WG water dispersible granules containing 800 g/kg thiram.
80WP wettable powder containing 800 g/kg thiram.
75WG water dispersible granules containing 750 g/kg thiram.
65WP wettable powder containing 650 g/kg thiram.
50WG water dispersible granules containing 500 g/kg thiram.

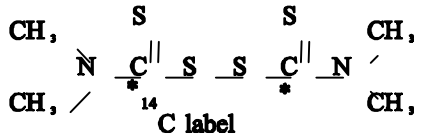
Combinations with other fungicides:
SILBOS DF water dispersible granules containing 640 g/kg thiram.
SILBOS T wettable powder containing 640 g/kg thiram.

DIRAC Express water dispersible granules containing 530 g/kg thiram.
RONILAN T wettable powder containing 530 g/kg thiram.

METABOLISM AND ENVIRONMENTAL FATE

Animal metabolism

Information was made available to the Meeting on metabolism studies in rats, lactating goats and laying hens.



Gay *et al.* (1992) summarized the conclusions from a series of metabolism studies on rats with [*thiocarbonyl*-^{14}C]thiram. When rats were dosed orally with [^{14}C]thiram much of the ^{14}C (40-60%) was eliminated as volatiles in exhaled air, 25-35% was excreted in the urine and 2-5% in the faeces. After an interval of 96 hours 2-3% of the ^{14}C remained in the tissues. Polar metabolites and conjugates were identified in the urine.

Groups of rats (5 male + 5 female) were given single doses of [^{14}C]thiram at 125 mg/kg bw for the high-dose group and 1.9 mg/kg bw for the low-dose group (Gay, 1987). Excreta were collected for 7 days, when the animals were slaughtered for tissue collection. The total recovery of the administered ^{14}C was low, with 21 and 29% in the urine, 3.9 and 2.8% in the tissues and 4.1 and 2.4% in the tissues (recoveries in low-dose and high-dose groups respectively). The low total recovery suggested loss in exhaled air.

Exit gases were collected for 96 hours from metabolism cages containing groups of 3 rats given single oral doses of [^{14}C]thiram at 2.1-2.5 mg/kg bw (Norris, 1989). The majority of the volatile ^{14}C was produced in the first 24 hours, and its rate of production peaked between 2 and 4 hours after dosing. The total ^{14}C recovered in the expired air was 57-63% and in the urine 25-43% of the dose. The composition of the exhaled ^{14}C was not examined in this experiment, but the trapping system would have collected CO_2, CS_2 and COS.

Rats (6 male + 6 female) were dosed orally for 14 days with unlabelled thiram at 2 mg/kg bw then with [^{14}C]thiram at 2 mg/kg bw on day 15 (Nomeir and Markham, 1990). Expired air and excreta were collected for a further 96 hours and then the animals were slaughtered for tissue collection. Approximately 33-35% of the administered ^{14}C was eliminated in the urine, 2.6-5.3% in the faeces and 47-48% in the expired air. The nature of the volatile ^{14}C was not investigated in detail, but 75-85% of it was collected in a KOH trap suggesting CO_2 or COS, with the remainder collected in a reagent for CS_2.

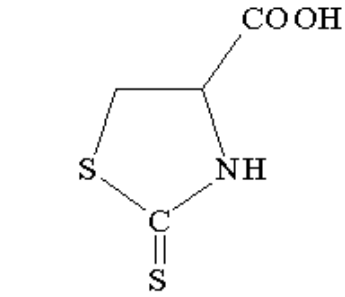
Norris (1991) identified by HPLC the metabolites in the urine of the rats from the single dose study of Gay (1987) and the multiple dose study of Nomeir and Markham (1990). There were no sex differences in the metabolism but the proportions of some of the metabolites depended on the dosage level and the time after dosing. Table 1 lists the proportions of the metabolites as percentages

of the ^{14}C in the urine samples.

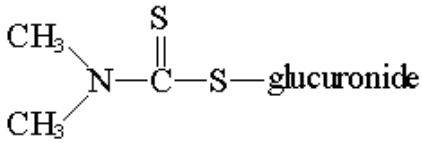
Table 1. Levels of metabolites (as % of total ^{14}C in the sample) in urine of rats given [^{14}C]thiram as single doses 125 mg/kg bw or multiple doses of 2 mg/kg bw/day for 15 days (Norris, 1991).

Metabolite	^{14}C, % of total in sample			
	Single dose		Multiple dose	
	Male, 0-24 hr sample	Female, 0-24 hr sample	Male, 4-8 hr sample	Female, 4-8 hr sample
U_1	13	7.8	1.4	1.7
Unidentified			0.3	0.4
U_2	5.6	8.7	12	11
Unidentified			5.1	4.0
U_3	37	42	40	36
U_4	3.4	2.4	1.6	0.6
U_5	34	36	36	42

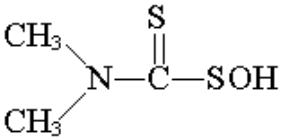
U_1 2-thioxo-4-thiazolidinecarboxylic acid



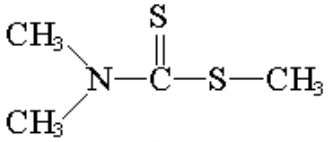
U_2 dimethyldithiocarbamoyl glucuronide



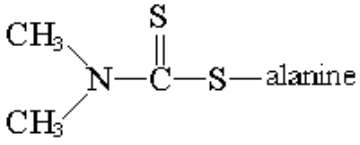
U_3 dimethyldithiocarbamoylsulfenic acid



U_4 methyl dimethyldithiocarbamate



U_5 dimethyldithiocarbamoylalanine



McManus (1991) fed rats with 50 ppm unlabelled thiram in the diet for 9 weeks and then gave them single oral doses of [^{14}C]thiram. The five urinary metabolites shown above were identified by HPLC and mass spectrometry.

Dalvi and Deoras (1986) showed that CS_2 was present in expired air from rats dosed with thiram by intraperitoneal injection.

Residues in the tissues, milk and excreta were measured in lactating goats (2 goats for the low dose (×1) and 1 for the high dose (×10), each animal weighing approx 40-50 kg) dosed orally twice daily for 4 consecutive days by capsule with [*thiocarbonyl*-^{14}C]thiram equivalent to 2.5 and 3.3 ppm (×1) and 23 ppm (×10) thiram in the feed (Norris, 1993b). The feed consumption was 2 kg /animal/day. The animals were milked twice daily; milk production was at least 800 ml per day. Respiration gases were collected from the low-dose animals for 10 hours after the final dose. Milk and excreta were collected throughout, and the goats were slaughtered 13 hours after the final dose for tissue collection. The ^{14}C was distributed as shown below.

Sample	^{14}C, % of (low) dose	
	Goat 1	Goat 2
Milk	1.3	1.0
Urine	9.2	9.6
Faeces	4.6	5.6
Tissues	7.2	7.5
Expired air	48	39
Total	70	63

A major part of the ^{14}C was eliminated in the respired gases, with smaller amounts in the urine and faeces. Approximately 60-70% of the dose was accounted for, but the value for the expired air is an estimate based on the 10-hour collection period. The value for the tissues includes the stomach and intestine contents, which accounted for almost half of the 7.2 and 7.5% reported.

The level of ^{14}C in the milk reached a plateau within 1.5 to 3 days of the first dose, and that in the urine by day 2 or 3, but the level in the faeces may still have been increasing at the completion of the study.

The ethanol+diethylamine traps (for CS_2 and COS) on the expired air collected 9.3 and 2.8% of the dose. Most of the ^{14}C in the expired air was present as CO_2.

The metabolism of thiram was quite extensive and much of the ^{14}C in the milk and tissues was present as very polar extractable material or bound residues. ^{14}C was present in glycogen, amino acids, proteins, lactose and saponifiable lipids. The only xenobiotic metabolites detected were CS_2 and COS. It is likely that thiram is rapidly converted to dimethyldithiocarbamate and then to dimethylamine and CS_2. CS_2 is converted to COS and carbonate. [^{14}C]carbonate then enters fat, protein and carbohydrates.

Table 2. Total ^{14}C (as thiram) in milk from goats dosed orally by capsule for 4 days with [^{14}C]thiram equivalent to 2.5 and 3.3 ppm (×1) and 23 ppm (×10) in the feed (Norris, 1993b).

Day	Total ^{14}C as thiram, mg/kg		
	x 1 dose		x 10 dose
0.5	0.024	0.027	0.27
1	0.036	0.040	0.33
1.5	0.051	0.054	0.47
2	0.044	0.047	0.36
2.5	0.056	0.064	0.40
3	0.048	0.060	0.45
3.5	0.045	0.070	0.48
4	0.050	0.074	0.48
% of dose in milk	*1.3*	*1.0*	*1.8*

Table 3. Distribution of radiolabel in tissues from goats dosed orally for 4 days by capsule with [^{14}C]thiram equivalent to 2.5 and 3.3 ppm (×1) and 23 ppm (×10) in the feed (Norris, 1993b).

Sample	Goats dosed at 2.5 and 3.3 ppm		Goat dosed at 23 ppm	
	^{14}C as % of total administered ^{14}C	^{14}C as thiram, mg/kg	^{14}C as % of total administered ^{14}C	^{14}C as thiram, mg/kg
Muscle	0.81 1.1	0.008 0.013	1.1	0.12
Kidneys	0.049 0.064	0.055 0.093	0.064	0.68
Liver	2.4 2.4	0.51 0.58	3.4	7.0
Blood	0.42 0.49	0.021 0.030	0.52	0.27
Fat	0.035 0.064	0.005 0.013	0.075	0.12

Residues in the tissues, eggs and excreta were measured in laying White Leghorn hens (groups of 4 for the low dose and 6 for the high dose, each bird weighing approx 1.5 kg) dosed orally by capsule once daily for 4 days with [*thiocarbonyl*-^{14}C]thiram equivalent to 0.6 and 6.0 ppm thiram in the feed (Norris, 1993a). The feed consumption was 80-110 g/bird/day. Eggs and excreta were collected throughout, and the birds were slaughtered 24 hours after the final dose for tissue collection.

Table 4 shows the distribution of the ^{14}C. The total recovery of the administered dose was only 61-68%. The likely reason is loss as volatiles. Liver was the tissue with the highest level of ^{14}C. A high percentage of the ^{14}C was extractable with chloroform or aqueous methanol, or was made soluble on enzymic or acid digestion.

The residues extracted by aqueous methanol from the liver were determined by reversed-phase HPLC and the resultant three peaks, comprising 4.7% of the total ^{14}C in the liver, were identified by mass spectrometry as dimethyldithiocarbamate ornithine, 2-thioxo-4-thiazolidinecarboxylic acid and dimethyldithiocarbamate glucuronide. Approximately 48% of the ^{14}C in the liver was identified as incorporated into natural products such as acids, amino acids, peptides and proteins. The ^{14}C residues in the other tissues were at quite low levels were characterized by ion-exchange chromatography also as natural products.

Thiram itself was identified in an aqueous methanolic extract of the excreta. Other

metabolites in the extract appeared to be conjugates of dimethyldithiocarbamate.

Table 4. Distribution of radiolabel in tissues, eggs and excreta from hens dosed orally for 4 days with [*thiocarbonyl*-^{14}C]thiram equivalent to 0.6 and 6.0 ppm thiram in the feed (Norris, 1993a).

Sample	Hens dosed at 0.6 ppm		Hens dosed at 6.0 ppm	
	^{14}C as % of total administered ^{14}C	^{14}C as thiram, mg/kg	^{14}C as % of total administered ^{14}C	^{14}C as thiram, mg/kg
Egg white, days 1-4	0.09	0.00-0.003	0.069	0.003-0.020
Egg yolk, days 1-4	0.075	0.00-0.006	0.058	0.001-0.044
Excreta, days 1-4	66	0.14-0.18	59	1.2-1.5
Muscle, breast	0.18	0.002	0.19	0.025
Muscle, thigh	0.17	0.003	0.21	0.036
Liver	1.2	0.11	1.0	0.89
Kidney	0.086	0.041	0.12	0.51
Fat	0.027	0.002	0.013	0.009
Heart	0.017	0.008	0.025	0.11
Blood	0.20	0.007	0.23	0.081
GI tract	0.49	0.007	0.44	0.053
Gizzard	0.035	0.004	0.040	0.041
Skin	0.049	0.003	0.052	0.029

The metabolic pathways are shown in Figure 1.

Plant metabolism

Information was made available to the Meeting on metabolism studies on apples and grapes when thiram was applied to the fruit and leaves, and on soya beans, cotton, wheat and sugar beet when it was used as a seed treatment.

Wyss-Benz (1994) applied [*thiocarbonyl*-^{14}C]thiram to the apples and leaves of two apple trees at a dose equivalent to 29.5 kg ai/ha (5 times the label rate) and harvested the fruit and leaves 0, 14, 28, 56 and 101 days after treatment. Surface radioactivity was removed for measurement by washing the fruit or leaves first with acetonitrile and then with acetonitrile/water (9+1). Apples, leaves, peel and pulp were subjected to an exhaustive extraction procedure with acetonitrile and water. Surface-washed apples from days 28 to 101 were homogenized and separated into juice and press cake for extraction and analysis.

The levels and distribution of ^{14}C on the surface and within the fruit and leaves are recorded in Table 5. Initially, as expected, most of the residue was on the fruit surface, but by day 14 only half of the remaining residue was on the surface. The ^{14}C incorporated into the fruit (not removed by acetonitrile washing) was quite persistent.

Figure 1. Metabolism of thiram by animals

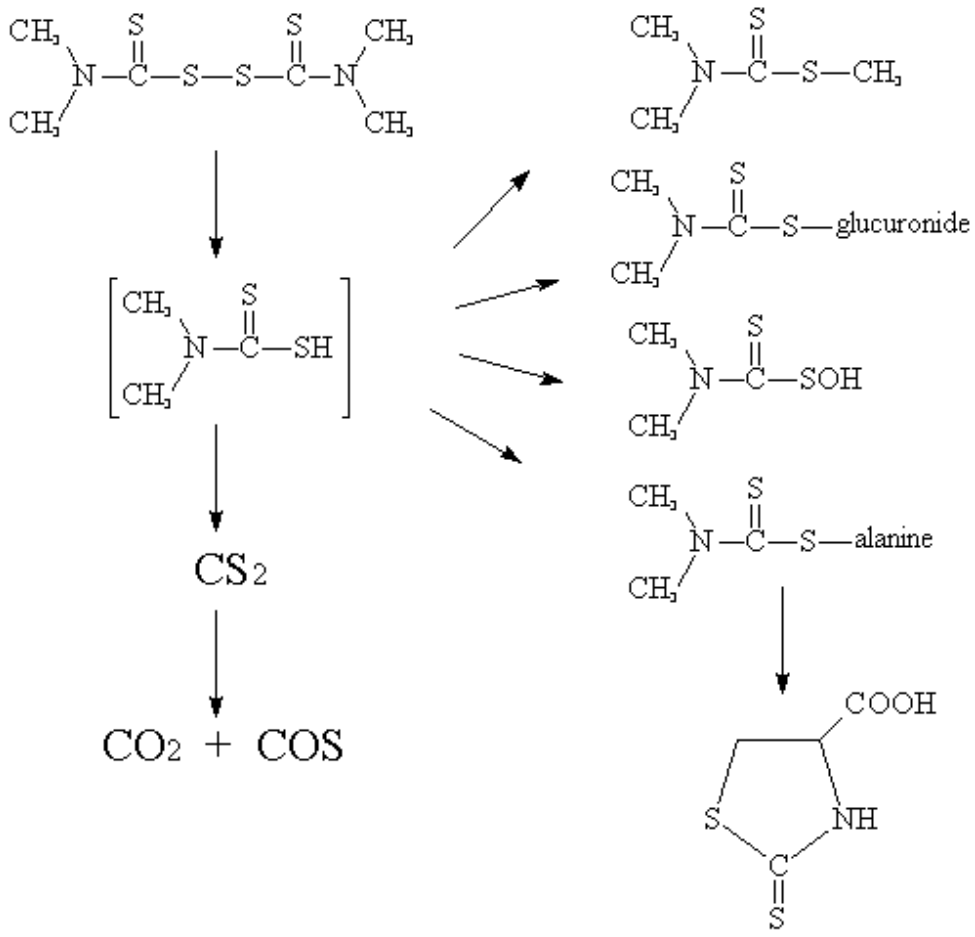


Table 5. Residues on the surface and within apples and leaves harvested after treating fruit and leaves of two apple trees with [^{14}C]thiram at a dose equivalent to 29.5 kg ai/ha (Wyss-Benz, 1994). Apple juice and press cake were prepared from acetonitrile-washed apples.

Sample	^{14}C in the apples or leaves expressed as mg thiram/kg and as % of total ^{14}C									
	0 days		14 days		28 days		56 days		101 days	
	^{14}C as thiram, mg/kg	%	^{14}C as thiram, mg/kg	%	^{14}C as thiram, mg/kg	%	^{14}C as thiram, mg/kg	%	^{14}C as thiram, mg/kg	%
FRUIT										
MeCN washings of apples	173	94	10.9	51	0.92	13	0.58	8.0	0.038	0.9
Apple juice					2.1	29	3.2	44	1.98	47
Apple press cake					4.3	58	3.5	48	2.2	53
MeCN-washed apples	11.6	6.3	10.4	49						
Apples - TOTAL	185		21		7.3		7.3		4.2	
LEAVES										

Sample	^{14}C in the apples or leaves expressed as mg thiram/kg and as % of total ^{14}C									
	0 days		14 days		28 days		56 days		101 days	
	^{14}C as thiram, mg/kg	%	^{14}C as thiram, mg/kg	%	^{14}C as thiram, mg/kg	%	^{14}C as thiram, mg/kg	%	^{14}C as thiram, mg/kg	%
MeCN washings of leaves	3094	82	380	57	125	30	23	8.5	4.9	3.7
MeCN-washed leaves	700	18	292	43	296	70	253	92	128	96
Leaves - TOTAL	3794		672		421		276		133	

The distribution of residues between peel and pulp in 5 apples harvested on day 101 was investigated further and is shown right. The major part of the ^{14}C residue was in the pulp.

Sample	^{14}C as thiram	of total residue
Washings	0.081 mg/kg of whole apples	2.7
Peel	5.2 mg/kg	38
Pulp	1.8 mg/kg	60

The residues in the extracts and acetonitrile washings were characterised by TLC and HPLC. A minor unidentified metabolite, more polar than thiram, was found in the surface washings (Table 6). Thiram was not found within the fruit except on day 0 by TLC at a level estimated to be less than 0.5 mg/kg. The main radioactive residue was very polar. The incorporated residue contained only a small percentage of the dimethyldithiocarbamoyl moiety (Table 7) as demonstrated by the release of small amounts of CS_2 under acid digestion. The ^{14}C was probably incorporated into natural plant products. The fruit and leaves showed different patterns of metabolites.

Table 6. Occurrence of thiram and unidentified metabolite RO in acetonitrile washings from fruit and leaves after treatment of apple trees with [^{14}C]thiram at a dose equivalent to 29.5 kg ai/ha (Wyss-Benz, 1994).

Fraction	^{14}C as thiram, mg/kg				
	0 days	14 days	28 days	56 days	101 days
FRUIT					
Thiram	164	9.5	0.78	<0.3	<0.3
Metabolite RO	7.1	*n*	*n*	*n*	*n*
Non-resolved ^{14}C	1.8	1.3	0.14	0.58	0.038
LEAVES					
Thiram	3008	367	103	*n*	*n*
Metabolite RO	86	13	21	*n*	*n*
Non-resolved ^{14}C	-	-	-	23	4.9

n: no detectable residues of ^{14}C (detection limit not stated)

Table 7. CS_2-liberating residues in acetonitrile-washed apples harvested at intervals from apple trees treated with [^{14}C]thiram at a dose equivalent to 29.5 kg ai/ha (Wyss-Benz, 1994).

Fraction	^{14}C as mg thiram equivalents per kg apple					
	0 days	14 days	28 days	56 days	101 days	101 days peel + pulp
Total residue, A	11.6	10.4	6.4	6.7	4.2	2.9
CS_2-liberating residue, B	0.104	0.214	0.23	0.22	0.12	0.058
B as % of total of A	0.9	2.1	3.5	3.3	2.8	2.0

Morgenroth and Wyss-Benz (1995) applied [*thiocarbonyl*-^{14}C]thiram four times to the grapes and leaves of two grape vines (variety Blauburgunder) at a dose equivalent to 3.2 kg ai/ha (maximum label rate). The growth stages for the 4 applications were 50% petal fall, closure of grapes, change of grape colour and 1 month before maturity. Fruit and leaves were harvested on days 0, 14 and 27 after the final treatment.

Surface radioactivity was removed for measurement by washing the fruit or leaves first with acetonitrile and then with acetonitrile/water (8+2). Surface-washed grapes were homogenized and separated into juice and press cake for extraction and analysis. Washed leaves and fruit components were subjected to an exhaustive extraction procedure with acetonitrile and water.

The total ^{14}C residues in the grapes and the acetonitrile surface washings are recorded in Table 8. The residues were quite persistent, with approximately one third of the total residues still on the fruit surface 27 days after the final application.

Two unidentified metabolites RO and R1, both more polar than thiram, were detected on the surface of the grapes by HPLC (Table 9), but the levels were substantially lower than those of thiram. Thiram itself constituted most of the surface residue on both fruit and leaves.

Approximately 5% of the residue incorporated within the fruit liberated CS_2 on acid digestion (Table 10), suggesting that it contained the dimethyldithiocarbamoyl moiety.

Most of the ^{14}C residue in the juice was shown by ultrafiltration to have a molecular weight below 500, and most could not be more positively identified. A glucose conjugate, 1-(*N*,*N*-dimethylthiocarbamoylthio)-1-deoxy-â-D-glucose hydrate, was identified at low levels in the juice from grapes harvested 27 days after the final application.

Much of the ^{14}C in the grapes was very polar or unextractable and had probably become incorporated into natural products.

Table 8. Residues on the surface and within grapes and leaves harvested after vines were treated 4 times with [^{14}C]thiram at a dose equivalent to 3.2 kg ai/ha (Morgenroth and Wyss-Benz, 1995). Grape juice and press cake were prepared from acetonitrile-washed grapes.

Sample	^{14}C in apples and leaves expressed as mg thiram/kg and as % of total ^{14}C or leaves.

	0 days		14 days		27 days	
	^{14}C as thiram mg/kg	%	^{14}C as thiram mg/kg	%	^{14}C as thiram mg/kg	%
FRUIT						
MeCN washings of grapes	8.6	61	3.5	38	3.4	35
Grape juice	3.1	22	3.6	39	4.2	44
Grape press cake	2.4	17	2.0	23	2.1	21
Grapes - TOTAL	14.1		9.1		9.7	
LEAVES						
MeCN washings of leaves	616	90	92	57	12	13
MeCN-washed leaves	69	10	69	43	81	87
Leaves - TOTAL	685		161		93	

Table 9. Thiram and unidentified metabolites RO and R1 in acetonitrile washings from fruit and leaves after treatment of grape vines with [^{14}C]thiram (Morgenroth and Wyss-Benz, 1995).

	^{14}C as thiram, mg/kg		
	0 days	14 days	27 days
FRUIT			
Thiram	8.5	3.2	3.2
Metabolite RO	0.20	0.24	<0.3
Metabolite R1	<0.3	0.082	<0.3
Non-resolved ^{14}C	-	-	0.21
LEAVES			
Thiram	608	85	9.8
Metabolite RO	8.1	6.9	<0.3
Non-resolved ^{14}C	-	-	2.1

Table 10. CS_2-liberating residues in acetonitrile-washed grapes harvested at intervals after vines were treated 4 times with [^{14}C]thiram at a dose equivalent to 3.2 kg ai/ha (Morgenroth and Wyss-Benz, 1995).

Fraction	^{14}C residues as mg thiram equivalents per kg grapes		
	0 days	14 days	27 days
Total residue	5.4	5.6	6.3
CS_2-liberating residue	0.24	0.33	0.35
CS_2 residue as % of total	4.5	5.8	5.6

Womer and Balba (1978, 1979) showed that wheat seedlings (5 weeks old) grown in a sandy loam soil from [*dimethylamine*-^{14}C]thiram-treated seed (334 mg thiram per kg seed) contained 0.25 mg/kg of ^{14}C expressed as thiram, of which 0.019 mg/kg was thiram. Some plants were grown to maturity; ^{14}C levels, as thiram, in the seed, chaff and straw were 0.05, 0.27 and 0.35 mg/kg respectively. The thiram level in the straw was <0.025 mg/kg.

After 4 weeks 62% of the recovered ^{14}C was in the soil (35% in plants) within 3 cm of the treated seeds, with a further 2.9% in the next zone 1.3 cm beyond the first. No ^{14}C was detectable beyond that zone. The pots (15 cm) had been watered with 50 ml water each day. The soil residues moved very little.

Harned and Tortora (1986) grew soya bean, cotton and wheat plants in a glasshouse and in the field from seed treated with [*thiocarbonyl*-^{14}C]thiram and measured the ^{14}C distribution in 30-day seedlings (Table 11) and mature plants (Table 12). The seed treatment rates (1×) were wheat 1.3 mg ai/g seed, cotton 1.4 mg ai/g seed, and soya beans 1.03 mg ai/kg seed. Some plants were also produced from seed treated at a fivefold rate. ^{14}C levels in the cotyledons and roots of the seedlings were higher than in the leaves and stems. In the mature plants the highest ^{14}C levels were in the roots and the lowest in the seeds. Autoradiography of the seedlings showed that the highest levels of ^{14}C were in the oldest parts of the plants.

Table 11. Distribution of ^{14}C in 30-day soya bean, cotton and wheat seedlings grown from [^{14}C]thiram-treated seed (Harned and Tortora, 1986).

Crop	Plant part	Total ^{14}C expressed as thiram, mg/kg	
		Indoor	Outdoor
Soya bean			
	leaf	0.50	0.29
	stem	1.5	0.085
	cotyledon	108	1.7
	root	8.5	2.7
Cotton			
	leaf	0.049	0.046
	stem	0.291	0.054
	cotyledon	2.6	2.5
	root	0.69	0.73
Wheat			
	leaf	1.1	0.47
	root	15	14

Table 12. Distribution of ^{14}C in mature soya bean, cotton and wheat plants grown from [^{14}C]thiram treated seed (Harned and Tortora, 1986).

Crop	Plant part	Total ^{14}C expressed as thiram, mg/kg dry weight of plant tissue			
		×1 rate		×5 rate	
		indoor	outdoor	indoor	outdoor
Soya bean					
	seed	0.019		0.15	
	pod	0.034		0.28	
	leaf	0.12		1.4	
	stem	0.29		3.2	
	root	0.87		8.0	
Cotton					
	seed	0.006		0.024	
	fibre	0.008		0.018	
	husk	0.11		0.14	
	leaf	0.035		0.094	
	stem	0.034		0.14	
	root	0.26		1.0	
Wheat					
	seed	0.078	0.005	0.51	0.036
	chaff	0.298	0.017	1.9	0.13
	leaf	0.822	0.025	4.1	0.14
	root		1.8		8.4

Nowakowski *et al.* (1986, 1987) used HPLC to separate and identify the thiram metabolites produced during the growing of soya bean, cotton and wheat seedlings in a glasshouse from seed treated at a fivefold rate as described by Harned and Tortora (1986). They showed that metabolites produced from [^{14}C]thiram in soya bean tissue cultures were similar to those obtained from plant extracts. Tissue culture was used to generate sufficient quantities for identification. They identified dimethyldithiocarbamoyl glycoside and dimethylthiocarbamoyl glycoside as the major metabolites in all the seedlings.

The two glycosides accounted for 70% of the ^{14}C in cotton seedlings and 50% in soya bean and wheat seedlings. A small amount of a cysteine conjugate was also identified. When aqueous wheat extract was treated with 50% sulphuric acid at 65°C only 3.4% of the ^{14}C was liberated as CS_2, suggesting that if any remaining metabolites contained the dithiocarbamoyl moiety they were largely unextractable.

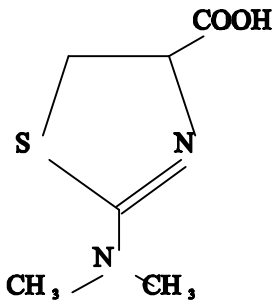
Liu and Robinson (1994a) applied [^{14}C]thiram at 0.062 g ai/ 100 g seed (the label rate) and 3.1 g ai/ 100 g seed (50×label rate) to soya bean seeds which were germinated and grown in a glasshouse. Forage, pod, seed and straw samples were taken for measurement of ^{14}C levels and identification of metabolites. The ^{14}C was relatively evenly distributed in the various plant parts (Table 13). There was some evidence that $^{14}CO_2$ had been evolved since ^{14}C was detected in control plants growing nearby.

Table 13. Distribution of ^{14}C in soya bean plants grown from [^{14}C]thiram-treated seed (Liu and

Robinson, 1994a).

Sample	^{14}C as thiram, mg/kg	
	Treatment: 1×label	Treatment: 50×label
Forage, 29 days after sowing	0.61	9.3
Forage, 69 days after sowing	0.13	4.2
Straw	0.33	9.4
Pods	0.22	6.9
Seeds	0.14	4.4

Extensive efforts were made to identify the ^{14}C compounds in the soya bean forage, straw and pods. Much of the ^{14}C had been incorporated into endogenous natural products such as sugars, fatty acids and citric acid, but the dimethyldithiocarbamoyl moiety of thiram had conjugated with amino acids and sugars. Thiram itself was not detected. The main metabolite identified was 2-dimethylamino-4-thiazolinecarboxylic acid which, in the case of the 50-fold treatment, constituted 22% of the ^{14}C in the 69-day forage sample, 18% of that in the straw, 41% in the pod and 11% in the seed.



2-(N,N-dimethylamino)-4-thiazoline carboxylic acid

Direct treatment of homogenized tissue with acidic stannous chloride released some $^{14}CS_2$, which showed that compounds or conjugates containing the dithiocarbamoyl moiety remained (Table 14).

Table 14. Nature of the volatile ^{14}C released by reacting acidic stannous chloride with homogenised tissue from soya bean plants grown in a glasshouse from seed treated with [^{14}C]thiram at 3.1 g ai/ 100 g seed (Liu and Robinson, 1994a).

Sample	Volatile ^{14}C as % of total ^{14}C in sample			
	CS_2	COS	CO_2	CO
Forage, 69 days	8.9	*n*	15.1	1.3
Straw	9.3	*n*	23.8	1.3
Pod	3.2	1.7	9.8	*n*
Seed	3.2	*n*	3.5	6.5

n: no detectable residue.

In a metabolism study [^{14}C]thiram at 0.1, 0.5 and 1.3 g ai/ 100 g seed (1, 5 and 13 times the label rate) was applied to wheat seed which was germinated and grown in a glasshouse (Johnson, 1994a; Liu and Robinson, 1994b). Forage, seed, chaff and straw samples were taken for measurement of ^{14}C levels and identification of metabolites. The ^{14}C was fairly evenly distributed among the various plant parts (Table 15). As in the soya bean study, ^{14}C was detected in control plants growing nearby suggesting that $^{14}CO_2$ had been produced.

Table 15. Distribution of ^{14}C in wheat plants grown from [^{14}C]thiram-treated seed (Liu and Robinson, 1994b).

Sample	^{14}C as thiram, mg/kg	
	Treatment: 1×label	Treatment: 13×label
Forage, 32 days after sowing	1.9	
Forage, 60 days after sowing	0.47	
Straw, 95 days	1.6	6.6 3.2[1]
Chaff, 95 days	0.32	4.9
Seed, 95 days	0.16	1.1

[1]Straw from a 5 x label rate treatment

The metabolism of thiram was similar in wheat and in soya beans. Again, much of the ^{14}C was incorporated into endogenous natural products such as sugars, fatty acids and citric acid, but there was some conjunction of the *N*,*N*-dimethyldithiocarbamoyl moiety with amino acids and sugars. Thiram itself was not detected. The main metabolite identified was 2-dimethylamino-4-thiazolinecarboxylic acid which, in the case of the 13-fold treatment, constituted 33% of the ^{14}C in the chaff, 29% in the straw, and 4% in the seed.

Direct treatment of homogenized tissue with acidic stannous chloride released some $^{14}CS_2$, which showed that compounds or conjugates containing the dithiocarbamoyl moiety remained (Table 16).

Table 16. Nature of the volatile ^{14}C released by reacting acidic stannous chloride with homogenized tissue from wheat plants grown in a glasshouse from seed treated with [^{14}C]thiram at 0.1 (×1), 0.5 (×5) or 1.3 (×13) g ai/100 g seed (Liu and Robinson, 1994b).

Sample	Total ^{14}C as thiram, mg/kg	Volatile ^{14}C as % of total ^{14}C in sample			
		CS_2	COS	CO_2	CO
Forage (X1), 60 days	0.47	*n*	*n*	*n*	*n*
Straw (×13)	6.6	2.3	*n*	21	*n*
Straw (×5)	3.2	6.8	*n*	8.4	4.7
Chaff (×13)	4.9	2.5	*n*	12	*n*
Wheat grain (×13)	1.1	8.9	*n*	8.7	*n*

n: no detectable residue.

In a metabolism study [^{14}C]thiram at 0.24 and 12 g ai/ 100 g seed (one and 50 times the label rate) was applied to sugar beet seed which was germinated and grown in a glasshouse (Johnson, 1994b; Liu and Robinson, 1994c). Samples of tops and roots were taken for measurement of ^{14}C levels and identification of metabolites. The ^{14}C levels were generally very low (Table 17). As in the soya bean and wheat studies, ^{14}C was detected in control plants growing nearby suggesting that $^{14}CO_2$ had been produced.

Table 17. Distribution of ^{14}C in sugar beet plants grown from [^{14}C]thiram-treated seed (Liu and Robinson, 1994c).

Sample	^{14}C as thiram, mg/kg	
	Treatment: 1×label	Treatment: 50×label
Immature tops, 100 days	0.006	0.15
Mature tops	0.012	0.096
Mature roots	0.008	0.32

Levels of metabolites were generally low. Incorporation of ^{14}C into sucrose, amino acids (such as glutamic acid) and acids of the citric acid cycle was established.

Direct treatment of homogenized tissue with acidic stannous chloride released some $^{14}CS_2$ from roots produced from seed treated at the 50-fold rate but levels of ^{14}C were generally too low for quantitative analysis (Table 18).

Table 18. Nature of the volatile ^{14}C released by reacting acidic stannous chloride with homogenized tissue from sugar beet plants grown in a glasshouse from seed treated with [^{14}C]thiram at 12 g ai/ 100 g seed (50-fold rate) Liu and Robinson (1994c).

Sample	Total ^{14}C as thiram, mg/kg	Volatile ^{14}C as % of total ^{14}C in sample			
		CS_2	COS	CO_2	CO
Roots	0.32	3.2	*n*	5.4	3.8
Immature tops	0.15	level too low for quantitative analysis			
Mature tops	0.096	level too low for quantitative analysis			

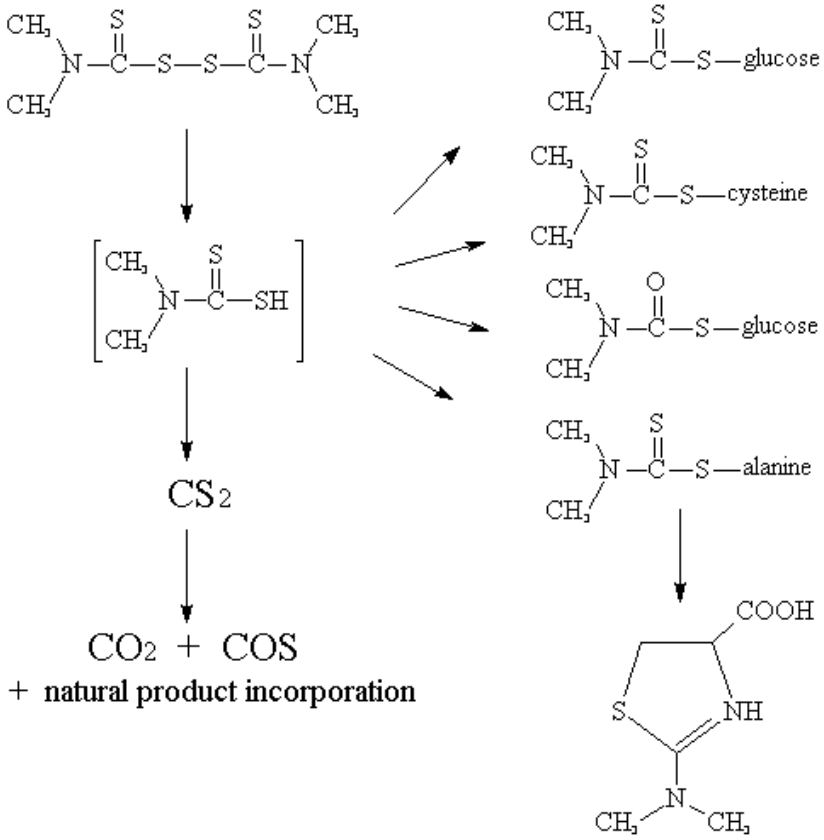
n: no detectable residue.

The metabolic pathways of thiram in plants are shown in Figure 2.

Environmental fate in soil

In a laboratory experiment [*thiocarbonyl*-^{14}C]thiram was incubated in a New York sandy loam soil in the dark at 20°C and 75% of field moisture capacity under aerobic conditions for 205 days (Morgenroth and Müller-Kallert, 1995). The initial thiram level in the soil was 20.3 mg ai/kg, chosen to represent a field application rate of 18 kg ai/ha. The system was continuously supplied with humidified air; evolved CO_2 was trapped. The characteristics of the soil were pH 6.7, organic carbon 2.4%, cation exchange capacity 14.4 meq/100g dry soil, clay 14.8%, silt 29.6%, sand 55.6%.

Figure 2. Metabolism of thiram by plants.



The levels of thiram and identified and unidentified degradation products in the soil at various sampling times are shown in Table 19. The disappearance of the parent compound was not first-order, but the half-life in the initial period was about 2 days, with 85% disappearance in 7 days and 97% in 14 days. The identity of the major product M6.5 was shown by GC-MS and HPLC-MS to be dimethyl carbamothioperoxoate. It reached its maximum level (1.8 mg/kg) at 4 days and exceeded the level of the parent compound after 42 days, but 99.8% of the parent had disappeared at this time.

Mineralisation of the residue was rapid with 9% of the applied ^{14}C evolved as $^{14}CO_2$ in 2 days and 50% within 21 days (Table 20). Bound or non-extractable residues reached a maximum of 48% of the applied dose at day 14 and thereafter declined slowly to 31% at day 205. The production of $^{14}CO_2$ was much slower when most of the residue was in the bound form. Fractionation of the soil organic matter showed that much of the unextractable ^{14}C was bound to the humin and humic acid fractions.

Table 19. Thiram and degradation products in a sandy loam soil during aerobic incubation in the dark for 205 days after an initial dosing with [^{14}C]thiram at 20.3 mg ai/kg (Morgenroth and Müller-Kallert, 1995).

Days	Thiram	Metabolite, mg/kg

		M6.1	M6.2	M6.3	M6.4	M6.5	M6.6	M6.7	M6.8	TMTU	TMU	TMTM
0	21	*n*	*n*	*n*	*n*	*n*	*n*	*n*	*n*	*n*	*n*	*n*
1	13.4	*n*	0.036	0.032	0.13	0.45	0.068	0.021	0.039	*n*	*n*	*n*
2	9.5	*n*	0.14	0.035	0.63	1.0	0.051	0.031	*n*	*n*	*n*	*n*
4	6.1	*n*	*n*	*n*	*n*	1.8	0.072	*n*	*n*	*n*	*n*	*n*
7	3.1	*n*	0.028	0.023	0.12	0.85	0.31	0.011	*n*	*n*	*n*	*n*
14	0.60	*n*	0.074	0.015	*n*	0.32	0.082	*n*	0.012	0.069	*n*	*n*
21	0.34	*n*	0.045	*n*	0.017	0.17	0.11	*n*	0.022	*n*	*n*	*n*
42	0.048	*n*	0.063	0.025	0.010	0.083	0.10	0.017	*n*	0.005	*n*	0.006
84	0.017	0.067	0.048	*n*	*n*	0.038	0.048	*n*	*n*	0.005	*n*	*n*
128	0.007	0.039	0.054	*n*	*n*	0.031	0.032	0.005	*n*	0.002	*n*	*n*
205	0.006	0.022	0.043	*n*	*n*	0.022	0.023	0.001	*n*	0.001	0.001	0.001

n: not detected
TMTM: tetramethylthiuram monosulphide
TMU: 1,1,3,3-tetramethylurea
TMTU: 1,1,3,3-tetramethylthiourea.

Table 20. Mineralisation of thiram in a sandy loam soil during aerobic incubation in the dark for 205 days after an initial dosing with [^{14}C]thiram at 20.3 mg ai/kg, as indicated by evolution of $^{14}CO_2$. (Morgenroth and Müller-Kallert, 1995).

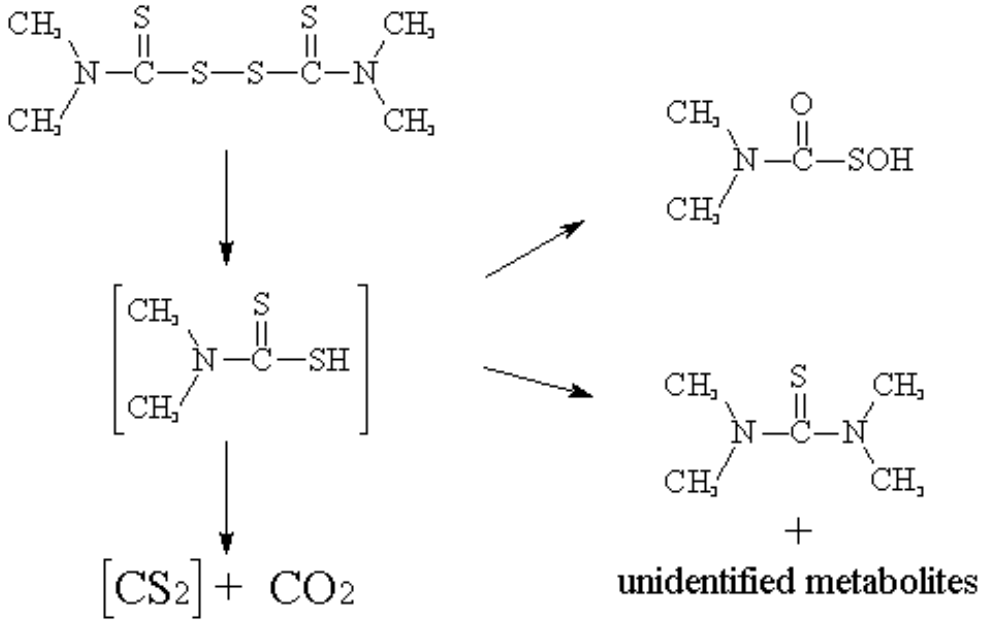
$^{14}CO_2$ as % of initial [^{14}C]thiram after days of incubation									
1	2	4	7	14	21	42	84	128	205
3.7	9.0	21.8	35.6	46.1	54.7	59.6	65.9	67.7	74.9

Burri (1995) applied [^{14}C]thiram to a thin layer of the same sandy loam soil at a rate equivalent to 18 kg ai/ha and exposed it to an artificial light source with a spectrum simulating sunlight for 21 days with a 12 hour light-dark cycle at 20°C. In both control and irradiated samples the percentage of extractable ^{14}C decreased with time, while the non-extractable ^{14}C reached 30% after 3 days and then remained reasonably constant.

The half-life for the disappearance of thiram from the control in the dark was 15.9 days and in the soil under artificial light 3.7 days. The total volatile ^{14}C produced by the soil under artificial light after 21 days amounted to 57% of the applied ^{14}C (37% as CO_2 and 20% probably as CS_2) while 11% was evolved by the control. The volatile ^{14}C compound captured in the trap matched CS_2 in an HPLC system, but was not further identified. No other products of photolysis were positively identified but they were generally minor or transient.

Morgenroth (1995) measured the adsorption and desorption properties of [^{14}C]thiram on four different soils, a sandy loam, loamy sand, silt loam and loam. Adsorption and desorption tests were carried out in 0.01M $CaCl_2$ at 20°C. Adsorption reached equilibrium after about 4 hours with high proportions of thiram adsorbed: it was not directly related to the organic carbon contents of the soils. Only small proportions of thiram could be desorbed from the soils. Thiram was judged to be slightly mobile to immobile in the soils tested.

Figure 3. Degradation of thiram in soil



Environmental fate in water/sediment systems

In a laboratory experiment [*thiocarbonyl*-^{14}C]thiram was incubated in aquatic systems of river water and sediment (Rhine) and pond water and sediment (Judenweiher) in the dark at 20°C under aerobic conditions for 101 days (Wyss-Benz, 1992). The system was continuously supplied with humidified air; evolved volatile compounds were trapped. The initial thiram levels in the water were 1.2 - 1.4 mg ai/l in the two experimental runs. The pH of the waters was 7.5-8.0. The organic carbon content of the river sediment, a loamy sand, was 0.62% and the pond sediment, a loam, 3.2%.

The decrease of thiram and the appearance of two identified products are shown in Table 21. The initial half-life of thiram was about 2 days with more than 90% disappearance within 7 days. Thiram itself was not detectable in solvent extracts of the sediments. Methyl dimethyldithiocarbamate (DMDTC-Me), CS_2 and CO_2 were identified as products. A number of other compounds were detected by HPLC, but were generally at low levels and could not be identified with any of the reference materials.

About half of the total ^{14}C disappeared from the water in 14 days. The ^{14}C in the sediments increased to a maximum of about 30% of that applied by days 14-30 and thereafter declined to 16-24% by day 101.

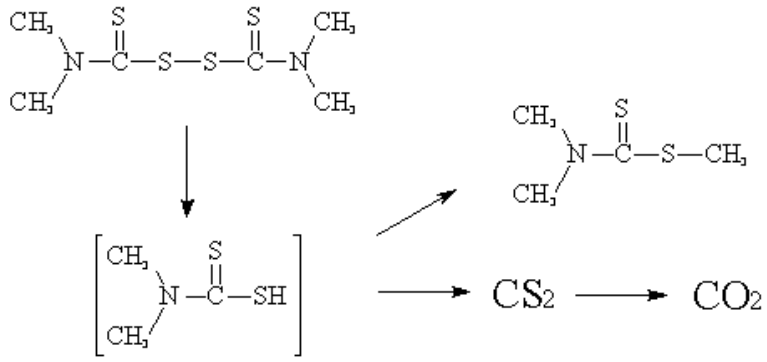
Table 21. Thiram and identified degradation products in the water phase of aquatic systems incubated with [^{14}C]thiram at 20°C in the dark under aerobic conditions (Wyss-Benz, 1992).

Days	^{14}C as thiram, mg/l					
	River			Pond		
	Thiram	DMDTC-Me	CS_2	Thiram	DMDTC-Me	CS_2
0	1.3	*n*	*n*	1.3	*n*	*n*
0.25	1.2	*n*	*n*	1.2	*n*	*n*
1	0.73	0.021	0.063	0.90	0.043	0.046
2	0.73	0.022	0.022	0.75	0.014	0.015
4	0.32	0.076	0.073	0.30	0.066	0.086
7	0.096	0.058	0.049	0.030	0.045	0.10
14	*n*	0.059	*n*	*n*	0.044	*n*
30	*n*	0.034	*n*	*n*	0.025	*n*
57	*n*	*n*	*n*	*n*	*n*	*n*
101	*n*	*n*	*n*	*n*	*n*	*n*

n: no detectable residue
DMDTC-Me: methyl dimethyldithiocarbamate

The degradation pathways of thiram in water/sedimant systems are shown in Figure 4.

Figure 4. Degradation of thiram in aerobic water/sediment systems.



METHODS OF RESIDUE ANALYSIS

Analytical methods

Thier (1979) described a regulatory analytical method for dithiocarbamate residues based on the generation of CS_2 by heating with stannous chloride and hydrochloric acid. The evolved CS_2 is collected in an ethanolic solution of cupric acetate and diethanolamine to form a colour which is measured with a spectrophotometer. Sample blank problems occur with some crops. With a 500 g sample the lower limit for reliable determination is generally around 0.1 mg/kg.

Dithiocarbamate residues can be measured by GLC head-space determination of the CS_2 evolved by treating the sample with stannous chloride and hydrochloric acid (Netherlands Ministry of Welfare, Health and Cultural Affairs, 1988). The method has been tested on many fruits and vegetables. In thiram trials on apples the limit of determination was 0.05-0.1 mg/kg (as CS_2) and recoveries were 85-121%.

Roland *et al.* (1992h) described an HPLC method for thiram residues in crops that measures thiram as the intact molecule and distinguishes it from other dithiocarbamate compounds. The residue is extracted from crop samples with dichloromethane or acetonitrile, and after clean-up on a C18 microcolumn with methanol/water and acetonitrile/water as eluting solvents, it is analysed on a C18 reversed-phase HPLC column with UV detection at 280 nm. Quantitative recoveries were achieved from strawberries and apples down to 0.2 mg/kg.

Weber (1993) validated the method on grapes and wine. Quantitative recoveries were achieved on grape samples fortified with thiram down to 0.1 mg/kg. To improve recoveries whole grapes were extracted in the frozen state and wine was ice-cooled before extraction. Low recoveries occurred in wine fortified at 0.1 mg/kg because thiram was being degraded. Weber (1994b) also validated the method for wet and dry grape pomace and must.

The results are summarized in Table 22. The LOD for the method with the substrates tested was 0.1 mg/kg.

Table 22. Analytical recoveries of thiram from fortified samples by the method of Roland *et al.* (Weber, 1993, 1994b).

Commodity	Fortification level, mg/kg	Recovery, %
Grapes	0.1 10	86-110, n=4 92-110, n=4
Wine	0.1 10	54-69, n=4 95-99, n=4
Wet pomace	0.1 0.2 0.5 1.0	110 95 108 120
Dry pomace	0.1 0.2 0.5 1.0	90 75 72 74
Must	0.1 0.2 0.5 1.0	80 90 88 100

Gatti (1992a) described a method very similar to that of Roland *et al.* (1992), and (1992b) a GLC head-space (CS_2 evolution) method which he tested on peaches, pears, apples, plums and cherries. Recoveries from spiked samples fell in the range 88-99% by HPLC and 93-105% by GLC, with each fruit tested once at 0.1 mg/kg and once at 4.0 mg/kg.

Stability of pesticide residues in stored analytical samples

Thiram residues were shown to be stable on frozen whole plums stored in closed plastic boxes at a freezer temperature below -20°C for 500 days (Roland, 1993). At each sampling the plums were analysed by a CS_2 evolution method and on three occasions by an HPLC method specific for thiram. The methods were in good agreement. The results are summarized in Table 23.

The method of sample preparation was shown to influence the analytical result. If plums were macerated in a blender thiram was decomposed by exposure to the macerate. Roland (1993) recommended that fruit for residue analysis should be stored whole, subjected to a minimum of cutting (into halves and quarters) while still frozen, and analysed immediately.

Table 23. Stability of thiram in whole plums in closed plastic boxes stored below -20°C (Roland, 1993).

Storage period, days	Thiram, mg/kg	
	By CS_2	By HPLC
0	7.9	7.1
35	8.3	7.9
90	8.8	-
183	7.6	-
363	6.9	-
500	8.1	7.8

The frozen storage stability of thiram was tested by fortifying apple juice and pomace at 1 mg/kg in extraction vials and storing them in a freezer at -20 ± 5°C (Leppert, 1995). Thiram was determined by a CS_2 evolution method. The residues were stable for the periods tested, up to 49 weeks (Table 24).

Table 24. Frozen storage stability of thiram residues in processed apple fractions fortified at 1 mg/kg and stored at -20 ± 5°C (Leppert, 1995).

Storage period	% thiram remaining after storage		
	Apple juice	Wet pomace	Dry pomace
0 day	90, 90	84, 89	95, 88
2 weeks	94, 90	84, 118	104, 83
35 weeks	94, 84		
49 weeks		80, 72	80, 82

Residue definition

The Meeting considered the definition of thiram residues in terms of the crop metabolism studies, the supervised trials where residues had been determined by both a CS_2 evolution method and an HPLC method, and the needs of enforcement agencies.

The metabolism studies suggest that thiram is the major part of the CS_2-evolving residue, particularly when the residue is reasonably fresh and at the higher levels. Analyses of samples in supervised trials by the HPLC and CS_2 methods are usually in good agreement, which also suggests that thiram itself is the main residue.

Thiram is different from the other dithiocarbamates because it can be determined by a specific method that measures the intact molecule and MRLs could be established for thiram separately. It would, however, be confusing for analysts and enforcement agencies interpreting analytical data for one compound to appear under two different residue definitions.

The 1995 JMPR (Report, Section 2.8.1), in explaining the current basis for the definition of residues in general, stated "Preferably no compound, metabolite or analyte should appear in more than one residue definition."

The Meeting agreed that thiram should be included in the definition of dithiocarbamate residues:

The MRLs refer to total dithiocarbamates, determined as CS_2 evolved during acid digestion and expressed as mg CS_2/kg.

For dietary intake estimations the supervised trials median residue (STMR) will be expressed as thiram because estimated intakes need to be in terms of thiram itself for comparison with its ADI. For acute intake estimations a residue, such as an MRL, expressed in terms of CS_2 must be multiplied by a molecular weight correction factor of 1.58 for comparison with its ADI.

USE PATTERN

Thiram is a protective fungicide and is used as a seed treatment to control a number of fungi that cause "damping off" in seedlings and to control seedling blights. It is also widely used as a foliar treatment on fruits, vegetables and ornamentals to control *Botrytis* species, rust, scab and storage diseases.

Thiram formulations are registered for use in many countries. The Meeting was provided with information on registered uses on fruits, vegetables and other crops (Table 25).

Table 25. Registered uses of thiram.

Crop	Country	Form	Application				PHI, days
			Method	Max rate per applic.	Spray conc. kg ai/hl	No.	
Alfalfa	Germany	DS	seed treat	0.26[1]		1	
Alfalfa	Netherlands	WG	seed treat	0.20		1	
Apple	Australia	WG WP	foliar spray		0.12		7
Apple	Canada	WP	foliar spray		0.075-0.17		1
Apple	Netherlands	WP WG	foliar	1.6-2.4	0.16		Note[2]
Apple	Netherlands	WP WG	foliar	2.0-3.0	0.20		Note[3]
Apple	Netherlands	WP WG	foliar	1.2-1.5	0.10	3 Note[4]	7
Apple	UK	WG	foliar	2.4	0.16		7
Artichoke	Italy	WG	spray		0.13-0.24	1-2	10
Asparagus	Belgium	WG	soaking		1.6	1	root
Asparagus	Italy	WG	spray		0.13-0.24	1-2	10
Asparagus	Netherlands	WP WG	foliar	1.6		5	
Asparagus	Netherlands	WG	spray	1.6	0.16	1	
Asparagus	Netherlands	WP WG	drench		0.16	1	Applied at planting
Barley	Poland	FS	seed treat	0.06-0.07			
Barley	Poland	WS	seed treat	0.075-0.11			
Barley	UK	SC	seed treat	0.06			
Bean	Denmark	SC	seed treat	0.21		1	
Bean	France	WG	spray			1-3	
Bean	Germany	DS FS	seed treat	0.20		1	
Bean	Netherlands	WG	seed treat	0.15		1	
Bean	Spain	WG WP	spray		0.08-0.19	1-3	15
Bean, broad	Poland	FS	seed treat	0.08		1	
Bean, bush	Poland	FS	seed treat	0.08		1	

[1] Rate refers to kg ai/ha for foliar treatments; kg ai/100 kg seed for seed treatments

[2] Applied during and just after blossom

[3] Applied before blossom

[4] Later applications

Crop	Country	Form	Application				PHI, days
			Method	Max rate per applic.	Spray conc. kg ai/hl	No.	
Bean, field	UK	SC	seed treat	0.06			
Beet	Denmark	SC	seed treat	0.53		1	
Beet	France	WG	seed treat	0.48		1	
Beet	Germany	WP	seed treat	0.39		1	
Beet, red	Poland	FS	seed treat	0.08		1	
Beetroot	Netherlands	WP	seed treat	0.40			
Beetroot	UK	SC	seed treat	0.30			
Bilberry	UK	WG	foliar		0.16		7
Blackberry	Belgium	WG	spray		0.2	1-2	14
Blackberry	Netherlands	WG	spray		0.20	1-2	14
Blackberry	Netherlands	WP WG	foliar	2.0, 2.4	0.20	10	28
Blackberry	UK	WG	foliar		0.16		7
Brassica veg	Netherlands	WP	seed treat	0.23			
Broccoli	UK	SC	seed treat	0.30			
Brussels sprouts	UK	SC	seed treat	0.30			
Cabbage	Germany	WP	seed treat	0.40		1	
Cabbage	Italy	WG	spray		0.13-0.24	1-2	10
Cabbage	UK	SC	seed treat	0.30			
Carrot	Italy	WG	spray		0.13-0.15	1-2	10
Carrot	Netherlands	WP	seed treat	0.24			
Carrot	UK	SC	seed treat	0.30			
Cauliflower	UK	SC	seed treat	0.30			
Celeriac	Netherlands	WP	seed treat	0.32			
Celeriac	UK	SC	seed treat	0.30			
Celery	Australia	WG WP	foliar spray		0.12		7
Celery	Canada	WP	foliar spray		0.11		7
Celery	Italy	WG	spray		0.13-0.24	1-2	10
Celery	Netherlands	WG	seed treat	0.40		1	
Celery	Netherlands	WP WG	seed treat	0.32			
Celery	Spain	WG WP	spray		0.08-0.19	1-3	15
Celery	UK	SC	seed treat	0.30			
Chard	Italy	WG	spray		0.13-0.24	1-2	10
Chard	Netherlands	WP	seed treat	0.20			
Cherries	Netherlands	WP WG	foliar	3.0	0.20	4	14
Chicory	Belgium	WG	spray		0.16	1-3	28
Chicory	Italy	WG	spray		0.13-0.24	1-2	10
Chicory	UK	SC	seed treat	0.30			
Clover	Germany	DS	seed treat	0.26		1	
Courgette	Italy	WG	spray		0.13-0.24	1-2	10
Courgette	UK	SC	seed treat	0.30			
Crab apple	UK	WG	foliar	2.4	0.16		7
Cranberry	UK	WG	foliar		0.16		7
Cucumber	Belgium	WG	spray		0.2	1-2	3
Cucumber	Germany	WP	seed treat	0.33		1	
Cucumber	Germany	DS	seed treat	0.33		1	
Cucumber	Italy	WG	spray		0.13-0.24	1-2	10
Cucumber	Netherlands	WG	soaking		16	1	3
Cucumber	Netherlands	WG	watering		0.32	1-2	
Cucumber	Netherlands	WP	seed treat	0.24			
Cucumber	Netherlands	WP WG	foliar G[5]	0.8 g ai/plant		2	3
Cucumber	Spain	WG WP	spray		0.08-0.19	1-3	15
Cucumber	UK	SC	seed treat	0.30			
Currants, black, red	Denmark	WP	spray	4.8		1-2	last applic. at flowering
Currants	Netherlands	WP WG	foliar	2.0, 2.4	0.20	10	28
Currants	Netherlands	WG	spray		0.20	1-2	14
Currants, black	UK	WG	spray		0.20-0.40	1-2	
Currants, black, red, white	UK	WG	foliar		0.16		7
Dewberry	UK	WG	foliar		0.16		7
Endive	Germany	WP	dusting G	0.98 g/m^2		1	42
Escarole	Belgium	WG	spray		0.16	1-3	28

[5] Glasshouse

Crop	Country	Form	Application				PHI, days
			Method	Max rate per applic.	Spray conc. kg ai/hl	No.	
Fennel	Italy	WG	spray		0.13-0.24	1-2	10
Flax	France	WG	seed treat	0.2		1	
Flax	Germany	WP	seed treat	0.26		1	
Flax	Germany	DS	seed treat	0.26		1	
Flax	Netherlands	WG	seed treat	0.20		1	
Fodder beet	Germany	DS	seed treat	0.39		1	
Garlic	France	WG	spray	2.0		1-2	
Garlic	Italy	WG	spray		0.13-0.15	1-2	10
Gherkin	Netherlands	WP	seed treat	0.24			
Gooseberries	Netherlands	WP WG	foliar	2.0, 2.4	0.20	10	28
Gooseberry	Belgium	WG	spray		0.2	1-2	28
Grapes	Australia	WG WP	foliar spray		0.12		7
Grapes	France	WG	spray	3.2		1-3	
Grapes	Greece	WP WG	spray		0.20-0.30	1-2	
Grapes	Italy	WG	spray		0.10-0.13		
Grapes	Spain	WG WP	spray		0.16-0.24	1-3	15
Grapes, wine	Germany	WG	foliar spray	0.96-2.6	0.16	3	stage[6]
Grass	Netherlands	WG	seed treat	0.15		1	
Horseradish	Italy	WG	spray		0.13-0.15	1-2	10
Kale	UK	SC	seed treat	0.30			
Kohlrabi	UK	SC	seed treat	0.30			
Lamb's lettuce	Netherlands	WP	seed treat	0.32			
Leek	Italy	WG	spray		0.13-0.24	1-2	10
Leek	Netherlands	WP	seed treat	0.40			
Leek	UK	SC	seed treat	0.30			
Legume veg	Netherlands	WP WG SC	seed treat	0.23			
Lentil	Italy	WG	spray		0.13-0.24	1-2	10
Lettuce	Australia	WG WP	foliar spray		0.12-0.16		7
Lettuce	Belgium	WG	spray		0.16	1-3	28
Lettuce	France	WG	spray			1-2	Applied at 17 leaves
Lettuce	Italy	WG	spray		0.13-0.24	1-2	10
Lettuce	Netherlands	WP	seed treat	0.30			
Lettuce	Netherlands	WG	spray G	0.20 kg ai/m^2		1-3	28
Lettuce	Netherlands	WG	seed treat	0.40		1	
Lettuce	Netherlands	WP WG	foliar G	8.0	0.8	1	Note[7]
Lettuce	Netherlands	WP WG	foliar	0.40-0.80	0.20	4	28
Lettuce	Netherlands	DP	dusting G	10		1	28
Lettuce	UK	DP	soil treat	9.6			21
Lettuce	UK	SC	seed treat	0.30			
Lettuce	UK	DP	foliar	4.0		2-3	21
Lettuce	UK	WG	foliar	g	0.32		21
Lettuce	UK	WG	foliar		0.32		14
Lettuce, head	Germany	WP	dusting	0.98 g/m^2		1	42
Lettuce, head	Germany	DP	dusting	9.8		1	42
Linseed	Netherlands	WP WG	seed treat	0.23			
Loganberry	UK	WG	foliar		0.16		7
Lupin	Germany	DS	seed treat	0.20		1	
Lupin	Netherlands	WG	seed treat	0.15		1	
Maize	Belgium	WP	seed treat	0.19-0.25		1	
Maize	France	WG	seed treat	0.16		1	
Maize	Germany	DS FS	seed treat	0.20		1	
Maize	Germany	WP SC	seed treat	0.20		1	
Maize	Netherlands	WG	seed treat	0.15		1	
Maize	Netherlands	WP WG	seed treat	0.23			
Maize	Poland	FS	seed treat	0.05-0.06			
Maize	UK	SC	seed treat	0.11			
Marrow	Italy	WG	spray		0.13-0.24	1-2	10
Melon	Italy	WG	spray		0.13-0.24	1-2	10
Melon	UK	SC	seed treat	0.30			

[6] Spraying up to growth stage 35
[7] Applied 1 week after planting

Crop	Country	Form	Application				PHI, days
			Method	Max rate per applic.	Spray conc. kg ai/hl	No.	
Mustard	UK	SC	seed treat	0.30			
Oats	Netherlands	WG	seed treat	0.15		1	
Oats	Poland	FS	seed treat	0.06-0.07			
Oats	Poland	WS	seed treat	0.075-0.11			
Oats	UK	SC	seed treat	0.06			
Oilseed rape	Germany	DS	seed treat	0.44		1	
Onion	Canada	GR	at sowing	1.7-6.2		1	
Onion	Canada	GR	at sowing	95 g /100 m row			
Onion	France	WG	seed treat	0.06		1	
Onion	France	WG	spray	2.0		1.2	
Onion	Germany	DS WP	seed treat	0.39		1	
Onion	Italy	WG	spray		0.13-0.15	1-2	10
Onion	Netherlands	WP WG	seed treat	0.40			
Onion	UK	SC	seed treat	0.30			
Parsley	Netherlands	WG	seed treat	0.40		1	
Parsley	Netherlands	WP WG	seed treat	0.32			
Parsley	UK	SC	seed treat	0.30			
Parsnip	UK	SC	seed treat	0.30			
Pea	Denmark	SC	seed treat	0.28		1	
Pea	France	WG	spray			1-3	
Pea	Germany	DS FS	seed treat	0.20		1	
Pea	Netherlands	WG	seed treat	0.15		1	
Pea	Poland	FS	seed treat	0.08		1	
Pea	UK	SC	seed treat	0.06			
Pea, field	Germany	WP SC	seed treat	0.20		1	
Peach	Canada	WP	foliar spray		0.11-0.17	4-7	7
Peach	Netherlands	WP WG	foliar	2.4 0.8-2.4 G	0.16	4	14
Pear	Australia	WG WP	foliar spray		0.12		7
Pear	Netherlands	WP WG	foliar	2.0-3.0	0.20	Note[8]	7
Pear	Netherlands	WP WG	foliar	1.6-3.0	0.16, 0.20	3	7
Pear	Netherlands	WP WG	foliar	1.2-1.5	0.10	Note[9]	7
Pear	Netherlands	WP WG	foliar	1.6-2.4	0.16	Note[10]	7
Pear	UK	WG	spray	4.5	0.16	1-6	7
Pear	UK	WG	foliar	2.4	0.16		7
Pepper, sweet	Italy	WG	spray		0.13-0.24	1-2	10
Pepper, sweet	Netherlands	WG	spray		0.20	1-3	3
Peppers, sweet	Netherlands	WP WG	foliar G	1.0-3.0	0.20	12	3
Peppers, sweet	Netherlands	DP	dusting G	2.0-3.0		2	3
Plum	Canada	WP	foliar spray	5.1		1	dormant
Plums	Netherlands	WP WG	foliar	3.0	0.20	4	14
Pome fruit	Belgium	WG	spray		0.1-0.2	1-6	28
Pome fruit	Denmark	SC	spray	1.4-5.3		1-2	Note**Errore. Il segnalibro non è definito.** 7
Pome fruit	Denmark	WP	spray	4.8		1-2	Note**Errore. Il segnalibro non è definito.** 7
Pome fruit	Denmark	SC	spray	1.9-6.4		1-2	Note[11]
Pome fruit	Denmark	WP	spray	6.4		1-2	Note[11]
Pome fruit	France	WG	spray		0.2	1-6	
Pome fruit	Germany	WP WG	foliar spray	1.5-2.4	0.1-0.16	12	10
Pome fruit	Greece	WP WG	spray		0.20-0.30	1-5	15
Pome fruit	Italy	WG	spray		0.15-0.20	1-6	10
Pome fruit	Netherlands	WG	spray	1.4-3.0	0.10-0.20	1-6	7
Pome fruit	Portugal	WG	spray		0.12-0.16	1-6	
Pome fruit	Spain	WG WP	spray		0.16-0.24	1-5	15
Poppy	Germany	WP	seed treat	0.39		1	
Poppy seed	Germany	DS	seed treat	0.39		1	

[8] Before blossom
[9] Later applications
[10] During and just after blossom
[11] Before flowering

Crop	Country	Form	Application				PHI, days
			Method	Max rate per applic.	Spray conc. kg ai/hl	No.	
Poppy seed	Netherlands	WP WG	seed treat	0.32			
Potato	Italy	WG	spray		0.13-0.15	1-2	10
Potato seed	Denmark	WP	seed treat	0.64		1	
Pulses	Netherlands	WP WG SC	seed treat	0.23			
Quince	UK	WG	foliar	2.4	0.16		7
Radish	Germany	WP	seed treat	0.20		1	
Radish	Netherlands	WG	spray G	0.80 g ai/m^2		1	
Radish	Netherlands	WP WG	foliar	8.0	0.8	1	
Radish	Netherlands	WP	seed treat	0.24			
Radish	UK	SC	seed treat	0.30			
Rape seed	France	WG	seed treat	0.15		1	
Rape seed	Germany	WP	seed treat	0.44		1	
Rape seed	Netherlands	WG	seed treat	0.30		1	
Rape seed	Netherlands	WP WG	seed treat	0.24			
Rape seed	UK	SC	seed treat	0.015			
Raspberry	Belgium	WG	spray		0.2	1-2	14
Raspberry	Netherlands	WG	spray		0.20	1-2	14
Raspberry	Netherlands	WP WG	foliar	2.0, 2.4	0.20	10	28
Raspberry	UK	WG	spray		0.16	1-2	7
Raspberry	UK	WG	foliar		0.16		7
Rye	Netherlands	WG	seed treat	0.20		1	
Rye	Netherlands	WP WG	seed treat	0.23			
Rye	Poland	WS	seed treat	0.075-0.11			
Rye	Poland	FS	seed treat	0.06-0.07			
Rye	UK	SC	seed treat	0.06			
Shallot	France	WG	spray	2.0		1-2	
Soya bean	Poland	FS	seed treat	0.08		1	
Spinach	Germany	WP	seed treat	0.20		1	
Spinach	Italy	WG	spray		0.13-0.24	1-2	10
Spinach	Netherlands	WP WG	seed treat	0.32			
Spinach	Netherlands	WG	seed treat	0.40		1	
Spinach	UK	SC	seed treat	0.30			
Stem kale	Germany	DS	seed treat	0.44		1	
Stone fruit	Australia	WG WP	foliar spray		0.12		7
Stone fruit	Belgium	WG	spray		0.2	1-3	14
Stone fruit	France	WG	spray		0.2	1-3	
Stone fruit	Greece	WP WG	spray		0.20-0.30	1-4	15
Stone fruit	Italy	WG	spray		0.13-0.15	1-5	10
Stone fruit	Netherlands	WG	spray		0.16-0.20	1-3	14
Stone fruit	Portugal	WG	spray		0.16-0.20	1-4	
Stone fruit	Spain	WG WP	spray		0.16-0.24	1-5	15
Strawberry	Australia	WG WP	foliar spray		0.12		7
Strawberry	Belgium	WG	spray G		0.2	1-4	14
Strawberry	Canada	WP	foliar spray		0.11-0.19		3
Strawberry	Denmark	SC WP	spray	5.3-6.0		1-2	7
Strawberry	Germany	WG	foliar spray	3.2	0.16	3	blossom
Strawberry	Greece	WP WG	spray		0.20-0.30	1-2	
Strawberry	Italy	WG	spray		0.13-0.15	1-3	
Strawberry	Netherlands	WG	spray	3.0	0.20	1-3	14
Strawberry	Netherlands	WP WG	foliar	1.0, 1.2	0.20	12	14
Strawberry	Netherlands	WP WG	foliar G	1.2-2.4	0.20	6	Note[12]
Strawberry	Portugal	WG	spray		0.12-0.16	1-4	
Strawberry	Spain	WG WP	spray		0.16-0.24	1-4	15
Strawberry	UK	WG	spray		0.20-0.40	1-3	7
Strawberry	UK	WG	foliar	1.6			7
Sugar beet	Belgium	WP	seed treat	0.75		1	
Sugar beet	Germany	DS	seed treat	0.39		1	
Swede	UK	SC	seed treat	0.30			
Swedish turnips	Germany	DS	seed treat	0.44		1	
Sweet corn	Netherlands	WP	seed treat	0.20			
Sweet corn	UK	SC	seed treat	0.11			

[12] Applied until beginning of flowering

Crop	Country	Form	Application				PHI, days
			Method	Max rate per applic.	Spray conc. kg ai/hl	No.	
Sweet potato	Canada	WP	dip roots		0.11	1	
Tomato	Belgium	WG	spray		0.2		3
Tomato	Italy	WG	spray		0.13-0.24	1-2	10
Tomato	Netherlands	WG	spray		0.20	1-3	3
Tomato	Netherlands	WP WG	foliar G	1.0-3.0	0.20	12	3
Tomato	Netherlands	DP	dusting G	2.0-3.0		2	3
Tomato	UK	WG	spray	4.5-7.2	0.32	1-3	7
Tree nuts	Greece	WP WG	spray		0.20-0.30	1-4	15
Tree nuts	Portugal	WG	spray	1.6-2.4	0.16-0.20	1-4	
Tree nuts	Spain	WG WP	spray		0.16-0.24	1-5	15
Triticale	Poland	FS	seed treat	0.06-0.07			
Triticale	Poland	WS	seed treat	0.075-0.11			
Triticale	UK	SC	seed treat	0.06			
Turnip	Germany	DS	seed treat	0.44		1	
Turnip	Germany	WP	seed treat	0.44		1	
Turnip	Italy	WG	spray		0.13-0.15	1-2	10
Turnip	UK	SC	seed treat	0.30			
Vetch	Netherlands	WG	seed treat	0.15		1	
Watermelon	Italy	WG	spray		0.13-0.24	1-2	10
Wheat	France	WG	seed treat	0.16		1	
Wheat	Poland	FS	seed treat	0.06-0.07			
Wheat	Poland	WS	seed treat	0.075-0.11			
Wheat	UK	SC	seed treat	0.06			
Witloof	Italy	WG	spray		0.13-0.24	1-2	10
Witloof	Netherlands	WG	spray G	0.20 kg ai/m^2		1-3	28
Witloof	Netherlands	WP	seed treat	0.30			

RESIDUES RESULTING FROM SUPERVISED TRIALS

Residue data from supervised trials on fruit and vegetables are summarized in Tables 26-32.

Table 26	Apples. Belgium, France, Germany, Italy, Netherlands, Poland.
Table 27	Pear. Belgium, Germany, Italy, Spain.
Table 28	Peach. France, Italy, Spain.
Table 29	Plum. France, Italy. Cherries. Italy, Spain.
Table 30	Grapes. France, Germany.
Table 31	Strawberry. Belgium, France, Germany, Poland.
Table 32	Beans, dwarf French. Germany. Beans, French. France Cabbage, Savoy. Germany. Peas, green. France. Lettuce, head. Germany, Netherlands. Spinach. Germany, Netherlands. Tomato. France.

Thiram was determined by CS_2 evolution methods or by HPLC, and in some trials by both methods. Residues are reported in the Tables as thiram irrespective of the method of analysis. The molecular weight factor CS_2 x 1.58, was used to calculate the thiram residues if residues were quoted in the trial report as CS_2.

Where residues were not detected they are recorded in the Tables as below 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.

Residues in control samples are recorded only when they exceeded the LOD: this occurred in samples from 2 apple trials, 4 grape trials and 2 spinach trials analysed by the CS_2 evolution method, and in one apple and one grape sample analysed by the HPLC method.

Most of the trials were fully reported as well as being summarized. Some, especially older ones, were reported as detailed summaries rather than in full reports.

In some trials on apples, peaches, plums, cherries, strawberries and tomatoes other dithiocarbamates had been used on the crop during the growing season. Samples from such trials were considered valid for estimating thiram residue levels only if they had been analysed specifically for thiram by an HPLC method.

Thiram trials on apples were available from a number of European countries. Knapsacks were used for application in the Belgian trials of 1991-1993, where plot sizes were usually 8 or 16 trees, and in the French trial on plots of 6 trees. Thiram was applied by a tractor-mounted sprayer or axial fan blower in the German trials of 1989, and with a motorised knapsack and a wheelbarrow-mounted sprayer in the Italian trials where plot sizes were 6-12 trees.

Thiram was applied by knapsack sprayer in most of the trials on stone fruit and strawberries. Plot sizes for tree crops were 4-8 trees. Plot sizes in the strawberry trials ranged from 8 m of row to 100 m^2.

In a series of trials on grapes in France in 1992 thiram was applied with a back-pack air-blast sprayer. In each trial there were two plots whose ranged from 186-750 m^2. The plots were large enough to provide sufficient grapes for processing studies (Table 33). In a grape trial in France in 1991 thiram was applied with a knapsack atomizer to plots of 30 or 60 plants. Field samples were taken from each of 3 sub-plots.

Thiram residues in apples from 5 trials in The Netherlands in 1988 seem low by comparison with residues from other trials. Many of the residues were below the LOD (0.1 mg/kg). Details of sample storage conditions (temperature, duration, chopped or whole fruit) were not immediately available to confirm that residues had not disappeared during storage.

German trials on apples from 1971 to 1973 were reported on detailed summary sheets.

Table 26. Residues of thiram (expressed as thiram) in apples from foliar application of thiram in supervised trials in Belgium, France, Germany, Italy, The Netherlands and Poland. Underlined residues are from treatments according to GAP and are valid for estimation of maximum residue levels.

Country, year (variety)	Application				PHI, days	Residues, as thiram, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		CS_2 method	HPLC method	
Belgium, 1991 (Golden Delicious)	WG	2.4	0.80	13	83	<0.05	<0.05	CRP/92/975 GD 7D
Belgium, 1991 (Jonagold)	WG	2.4	0.80	9 dt	7	1.6	1.8	CRP 92/974 BEVJG1
Belgium, 1991 (Jonagold)	WG	2.4	0.80	11 dt	90	<0.05	<0.05	CPR/92/972 JG 7 D
Belgium, 1991 (Jonagold)	WG	2.4	0.80	12 dt	14	1.9	2.3	CRP/92/973 91/BEVJG2
Belgium, 1992 (Golden Delicious)	WG	2.4	0.80	10 dt	0 7 14		7.7, 8.5 4.9, 5.2 2.5, 3.1 c 0.77	BEWGD B.A.7792C
Belgium, 1993 (Jonagold)	WG	1.8	0.16	5 6	9 0 7 14 21	<u>2.7</u> 4.3 3.5 1.6 1.4		304735 2097/93
Belgium, 1993 (Jonagold)	WG	1.8	0.16	5 6	9 0 7	<u>4.6</u> 5.8 4.3		304670 2097/93

Country, year (variety)	Application				PHI, days	Residues, as thiram, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		CS_2 method	HPLC method	
					14 21	2.7 2.2		
France, 1993 (Starkrimson)	WG	1.6	0.16	5 6	7 0 14 21 28 35	1.9 2.8 2.4 1.9 1.7 0.4		304697 RA-2099/93
Germany, 1971 (Cox's Orange)	WP	2.9 +1.8 +1.8	0.11 +0.07 +0.07	3 dt	15	1.1		BBA 26/71 BBA 15/72 BBA 27/72
Germany, 1971 (Cox's Orange)	WP	1.8 +1.4 +2.9 +2.9	1 +0.8 +1.6 +1.6	4 dt	15	2.0		BBA 27/71 BBA 4/72 BBA 16/72
Germany, 1971 (Cox's Orange)	WP	1.8 +1.4 +2.9 +2.9	0.4 +0.32 +0.64 +0.64	4 dt	15	1.3		BBA 28/71 BBA 5/72 BBA 17/72
Germany, 1971 (Cox's Orange)	WP	2.9 +1.8 +1.8	0.64 +0.4 +0.4	3 dt	15	0.60		BBA 23/71 BBA 12/72 BBA 24/72
Germany, 1971 (Cox's Orange)	WP	2.9 +1.8 +1.8	0.32 +0.2 +0.2	3 dt	15	0.74		BBA 24/71 BBA 13/72 BBA 25/72
Germany, 1971 (Cox's Orange)	WP	2.9 +1.8 +1.8	0.16 +0.1 +0.1	3 dt	15	0.84		BBA 25/71 BBA 14/72 BBA 26/72
Germany, 1971 (Cox's Orange)	WP	1.8	0.4	3 dt	13	0.59		BBA 20/71 BBA 9/72 BBA 21/72
Germany, 1971 (Cox's Orange)	WP	1.8	0.1	3 dt	13	0.43		BBA 22/71 BBA 11/72 BBA 23/72
Germany, 1971 (Cox's Orange)	WP	1.8	0.2	3 dt	13	0.73		BBA 21/71 BBA 10/72 BBA 22/72
Germany, 1971 (Cox's Orange)	WP	1.8	1	3 dt	13	0.73		BBA 19/71 BBA 8/72 BBA 20/72
Germany, 1971 (Cox's Orange)	WP	1.8 +1.4 +2.9 +2.9	0.1 +0.08 +0.16 +0.16	4 dt	15	1.1		BBA 30/71 BBA 7/72 BBA 19/72
Germany, 1971 (Cox's Orange)	WP	1.8 +1.4 +2.9 +2.9	0.2 +0.16 +0.32 +0.32	4 dt	15	1.2		BBA 29/71 BBA 6/72 BBA 18/72
Germany, 1972 (Cox's Orange)	WP	1.4 +2.9 +2.9	0.08 +0.16 +0.16	3 dt	15	3.4		BBA 76/72 R/1800
Germany, 1972 (Cox's Orange)	WP	2.7	0.6	4 dt	12	1.2		BBA 77/72 U/450
Germany, 1972 (Cox's Orange)	WP	1.4 +2.9 +2.9	0.16 +0.32 +0.32	3 dt	15	4.7		BBA 75/72 R/900
Germany, 1972 (Cox's Orange)	WP	2.7	0.15	4 dt	12	1.1		BBA 79/72 U/1800
Germany, 1972 (Cox's Orange)	WP	2.7	0.3	4 dt	12	0.63		BBA 78/72 U/900
Germany, 1972 (Cox's Orange)	WP	2.7	0.1	4 dt	12	0.95		BBA 80/72 U/2700
Germany, 1972 (Golden	WP	1.8	1	1 dt	31	2.0		BBA 27/73

Country, year (variety)	Application				PHI, days	Residues, as thiram, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		CS_2 method	HPLC method	
Delicious)		1.8 1.8 1.8	0.4 0.2 0.1	1 dt 1 dt 1 dt	31 31 31	1.1 0.90 0.82		BBA 28/73 BBA 29/73 BBA 30/73
Germany, 1972 (Cox's Orange)	WP	1.8 1.8 1.8 1.8	1 0.4 0.2 0.1	1 dt 1 dt 1 dt 1 dt	31 31 31 31	0.71 0.87 0.66 0.47		BBA 5/73 BBA 6/73 BBA 7/73 BBA 7/73
Germany, 1972 (Cox's Orange)	WP	1.8 1.8 1.8 1.8	1 0.4 0.2 0.1	1 dt 1 dt 1 dt 1 dt	31 31 31 31	0.87 0.81 0.85 0.57		BBA 1/73 BBA 2/73 BBA 3/73 BBA 4/73
Germany, 1972 (Cox's Orange)	WP	1.4 +2.9 +2.9	0.32 +0.64 +0.64	3 dt	15	4.7		BBA 74/72 R/450
Germany, 1972 (Cox's Orange)	WP	1.8	1	3 dt	15	2.5		BBA 69/72 D/180
Germany, 1972 (Golden Delicious)	WP	1.8 1.8 1.8 1.8	1 0.4 0.2 0.1	1 dt 1 dt 1 dt 1 dt	31 31 31 31	0.68 1.0 0.66 0.52		BBA 31/73 BBA 32/73 BBA 33/73 BBA 34/73
Germany, 1972 (Cox's Orange)	WP	1.8	0.1	3	15	0.81		BBA 72/72 D/1800
Germany, 1972 (Cox's Orange)	WP	1.8	0.4	3	15	1.0		BBA 70/72 D/450
Germany, 1972 (Cox's Orange)	WP	1.4 +2.9 +2.9	0.8 +1.6 +1.6	3 dt	15	3.5		BBA 73/72 R/180
Germany, 1972 (Cox's Orange)	WP	1.8	0.2	3	15	0.49		BBA 71/72 D/900
Germany, 1973 (Cox's Orange)	WP	0.45	0.1	2	18	0.35		BBA 85/73 U/IV
Germany, 1973 (Cox's Orange)	WP	0.45	0.1	2	14	0.81		BBA 84/73 D/IV
Germany, 1989 (Gloster)	WG	2.4	0.16	4	0 5 7 10 14 10 10	4.0 3.0 2.2 1.7 1.9 s 0.21 j 0.19		0318/89
Germany, 1989 (Golden Delicious)	WG	2.4	0.80	4 dt	0 5 7 10 14	2.8 1.3 1.7 1.7 1.0		0320/89
Italy, 1991 (Golden Delicious)	WG	2×2.6 +2×3.3 +1×3.5	0.15	5	0 7 10 15 20	4.9 1.5 2.5 1.6 1.1	5.2 - - - 1.0	58/F/91-6012
Italy, 1992 (Weime)	WG	2.6	0.15	5 dt	0 7 10 15 20	10.0 7.4 4.2 3.4 2.9	10.2 7.6 4.1 3.3 2.9	UCB 205
Italy, 1993 (Perleberg)	WG	2.3	0.15	4 5	7 0 10 21 28 35	6.0 6.5 0.7 1.1 1.1 0.7		304700 RA-2099/93
Netherlands, 1986 (Golden Delicious)	WP	2.4	1.6	6	7 7 14	11 9.8 17 21 c <0.2 0.2		KvW257 (1987)

Country, year (variety)	Application				PHI, days	Residues, as thiram, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		CS_2 method	HPLC method	
					14	(3) 5.4 15 c 23 14		
Netherlands, 1986 (Golden Delicious)	WP	3.0	2.0	6	7 7 14 14	19 16 12 16 c <0.2 0.2 (3) 12 18 c 23 14		KvW257 (1987)
Netherlands, 1986 (Golden Delicious)	WP	4.5	2.0	6	7 14	6.8 6.0 6.3 8.9 5.7 7.3 3.8 6.8		KvW257 (1987)
Netherlands, 1986 (Golden Delicious)	WP	3.6	1.6	6	7 14	7.1 7.6 5.2 4.0 4.9 7.1 2.8 4.7		KvW257 (1987)
Netherlands, 1988 (Golden Delicious)	WP	3.0	0.20	5	7 14		0.46 0.12 0.40 0.33 <0.1 0.1 <0.1 0.22	RIVM 638201015
Netherlands, 1988 (Golden Delicious)	WP	2.2	0.16	5	7 14		<0.1 (4) 0.28 <0.1 (3)	RIVM 638201015
Netherlands, 1988 (Golden Delicious)	WP	2.4	1.6	5	7 14		0.47 1.8 0.56 0.27 0.33 0.14 0.76 0.34	RIVM 638201015
Netherlands, 1988 (Golden Delicious)	WP	3.1	2.0	5	7 14		1.6 6.3 4.0 0.4 1.2 0.88 0.41 0.25	RIVM 638201015
Netherlands, 1988 (Golden Delicious)	WP	2.3	0.16	5	7 14		<0.1 (4) <0.1 (4)	RIVM 638201015
Netherlands, 1988 (Golden Delicious)	WP	2.2	2.8	5	7 14		<0.1 (3) 0.25 <0.1 (4)	RIVM 638201015
Netherlands, 1988 (Golden Delicious)	WP	2.9	0.20	5	7 14		<0.1 (4) <0.1 (4)	RIVM 638201015
Netherlands, 1988 (Golden Delicious)	WP	1.8	1.6	5	7 14		<0.1 (4) <0.1 (4)	RIVM 638201015
Poland, 1994 (Starkrimson)	WG	2.4		4	7 14	7.0 3.8		
Poland, 1994 (Starkrimson)	WG	2.4		2	7 14	6.5 3.2		
Poland, 1994 (Starkrimson)	WG	3.6		4	7 14	8.5 8.1		
Poland, 1994 (Starkrimson)	WG	3.6		2	14	3.6		
Poland, 1994 (Spartan)	WG	3.6		2	7 14	2.9 1.5		
Poland, 1994 (Spartan)	WG	2.4		2	7 14	1.6 0.87		
Poland, 1994 (Spartan)	WG	2.4		4	7 14	3.2 1.7		
Poland, 1994 (Spartan)	WG	3.6		4	7 14	6.0 4.0		

dt: other dithiocarbamates also applied during the growing season.
c: control sample
s: sauce
j: juice

Table 27. Residues of thiram (expressed as thiram) in pears from foliar application of thiram in supervised trials in Belgium, Germany, Italy and Spain. Underlined residues are from treatments according to GAP and are valid for estimation of maximum residue levels.

Country, year (variety)	Application				PHI, days	Residues, as thiram, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		CS_2 method	HPLC method	
Belgium, 1991 (Conference)	WG	2.4	0.80	12	14	0.69	0.68	CRP/92/977 Bevecon 2
Belgium, 1991 (Conference)	WG	2.4	0.80	12	6	0.77 1.1	0.73 1.1	CRB/92/978 Bevecon 1
Belgium, 1991 (Conference)	WG	2.4	0.80	13	56	<0.05	<0.05	91/CONF 10D CRP/92/976
Belgium, 1992 (Conference)	WG	2.4	0.80	14	1 3 6 13		4.6 1.4 1.4 1.6	BEWCON B.A.7792b
Germany, 1989 (Alexander Lucas)	WP	2.4	0.16	4	0 5 7 10 14	3.8 3.0 2.7 1.9 1.0		0319/89
Germany, 1989 (Alexander Lucas)	WP	2.4	0.80	4	0 5 7 10 14	1.5 1.1 1.1 0.90 0.90		0321/89
Italy, 1991 (Kaiser)	WG	2.3-2.9	0.15	12	0 7 10 15 20 30	1.9 0.56 0.54 0.45 0.23 0.17	2.0	57/F/91-6010
Italy, 1991 (Abate Fetel)	WG	2.6	0.15	12	0 7 10 15 20 30	5.7 4.0 6.1 3.2 3.3 3.1 2.8 2.6 4.3 1.6 0.70 2.6 0.37 0.51 0.39 0.33 0.24	5.7 4.3 6.5 - - - 0.53 0.55 0.39 0.33 0.36	UCB 912
Italy, 1992 (William)	WG	2.6	0.15	8	0 7 10 15 20 30	5.6 5.9 5.1 2.9 2.0 1.2	4.8 6.4 4.8 2.3 2.0 1.1	UCB 202[1]
Spain, 1994 (Ercolini)	WG	2.4	0.24	6	0 14 21		2.9 3.2 2.8 1.9 2.2 3.0 1.5 1.4 1.1	94020/01-FPBI

[1] No report for HPLC method but summary data supplied

Table 28. Residues of thiram (expressed as thiram) in peaches from foliar application of thiram in supervised trials in France, Italy and Spain. Underlined residues are from treatments according to GAP and are valid for estimation of maximum residue levels.

Country, year (variety)	Application				PHI, days	Residues, as thiram, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		CS_2 method	HPLC method	
France, 1993 (Red Top)	WG	1.0-1.2	0.20	4 dt 5 dt	10 0 7 14 21	2.5 4.7 2.1 1.0 0.9		304654 RA-2083/93
Italy, 1991 (Dorata Tardiva Morettini)	WG	2.3	0.15	5	0 7 10 15	5.1 3.3 4.9 1.3 2.5 4.8 2.7 2.7 2.4 1.1 2.2 2.1	4.8 3.2 4.2 - - -	UCB 913

Country, year (variety)	Application				PHI, days	Residues, as thiram, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		CS_2 method	HPLC method	
					20	1.2 2.5 1.6	0.93 2.5 1.5	
Italy, 1991 (Glohaven)	WG	1.8	0.15	5 dt	0 8 12 15 20	4.8 1.3 1.3 0.62 0.51	4.3	56-F-91/6009
Italy, 1992 (Red Haven)	WG	2.3	0.15	5	0 10 15 20 30	5.7 3.6 1.4 0.70 0.70	5.6 3.5 1.3 0.60 0.60	UCB 203
Spain, 1994 (Maria Serena)	WG	2.4	0.24	3	0 10 14		f 1.5 f 0.43 f 0.26	94020/01-FPPF
Spain, 1994 (Baby Gold)	WG	2.4	0.24	3	0 10 14		f 3.4 f 1.3 f 0.70	94020/01-FPPF

dt: other dithiocarbamates also applied during the growing season.
f: residues in fruit without stone

Table 29. Residues of thiram (expressed as thiram) in plums and cherries from foliar application of thiram in supervised trials in France, Italy and Spain. Underlined residues are from treatments according to GAP and are valid for estimation of maximum residue levels.

CROP Country, year (variety)	Application				PHI, days	Residues, as thiram, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		CS_2 method	HPLC method	
PLUMS								
France, 1994 (Ente 707)	WG	2.4	0.24	3	0 9 14		f 1.0 1.7 0.9 f 1.2 0.8 0.9 f 0.6 1.0 0.8	94020/02-FPPL
Italy, 1991 (San Alberto)	WG	2.6	0.15	3	0 7 10 15 20	4.5 2.4 2.8 0.78 0.52 0.45 0.83 0.69 0.75 0.33 0.24 0.28 0.18 0.31 0.25	4.3 2.3 2.4	UCB 911
Italy, 1992 (Stanley)	WG	2.6	0.15	3 dt	0 7 10 15 20	2.9 0.70 0.75 0.33 0.24	2.8 0.65 0.62 0.27 0.25	UCB 201
CHERRIES								
Italy, 1991 (Durone di Vignola)	WG	9.3-10.9	0.15	3 dt	0 7 14 18 23	2.1 1.4 2.8 1.5 1.9 4.2 0.16 0.26 0.12 0.34 0.39 0.32 0.40 0.069 0.074	1.7 1.5 2.7	59/F/91/6011
Italy, 1991 (Bigareau/Moreau)	WG	1.1-1.5	0.15	3	0 7 10 15 20	0.94 2.3 1.9 0.22 0.23 0.22 0.21 0.17 0.37 0.24 0.22 0.19 0.11 0.10 0.16	0.80 2.1 1.8	UCB 914
Italy, 1992 (Roma)	WG	1.5	0.15	3	0 7 10 15 20	6.6 1.6 0.41 0.90 0.70	6.8 1.5 0.40 1.1 0.76	UCB 204
Spain, 1994 (Sunburst)	WG	2.4	0.24	3	0 10 14		6.1 8.2 5.7 0.4 0.2 0.2 0.1 0.1 0.1	94020/01-FPKI

dt: other dithiocarbamates also applied during the growing season.

f: residues in fruit without stone

Table 30. Residues of thiram (expressed as thiram) in grapes from foliar application of thiram in supervised trials in France and Germany.

Country, year (variety)	Application				PHI, days	Residues, as thiram, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		CS_2 method	HPLC method	
France, 1991 (Pinot Noir)	WG	3.2	0.8	1 dt 2 dt	70 44	0.26 0.33	<0.05 0.11	FR7/91, CRP/94/1295
France, 1992 (Merlot)	WG	3.2	1.6	3 4	32 21	9.3 11.7	2.6 5.6	28203 B001
France, 1992 (Pinot)	WG	3.2	1.2	3 4	33 21	2.4 2.5	1.1 2.2	28203 B004
France, 1992 (Pinot Noir)	WG	3.2	0.6-1.1	3 4	48 21	3.0 7.4	2.7 4.5	28203 B003
France, 1992 (Cot)	WG	3.2	1.4	3 4	32 21	0.8 4.3	0.7 2.3	28203 B002
France, 1994 (Cinsault)	WG	3.2	1.1	3	0 10 21 28 35		1.9 4.3 0.9 1.5 0.8 1.2 0.4 0.6 0.8 0.1 0.6 0.4 0.2 0.2 0.1	9402/01-FPWE loc 1
France, 1994 (Merlot)	WG	3.2	1.1	3	0 10 21 28 35		4.1 5.4 7.2 6.4 5.6 5.6 1.8 4.0 2.4 1.3 1.3 1.2 0.2 0.5 <0.2	9402/01-FPWE loc 2
Germany, 1992 (Riesling)	WG	2.4	0.24	3	0 21 28 35	1.7 c 5.1 3.0 3.3 3.0 c 1.7	c 1.9 1.1 0.8 0.7	DE/FR/03/92, DU3/30/92
Germany, 1992 (Portugieser)	WG	2.4	0.24	3	0 21 28 35	2.2 c 0.17 1.6 1.5 1.0 c 0.38	2.0 1.0 0.9 0.6	DE/FR/03/92, DU3/29/92
Germany, 1992 (Schwarzriesling)	WG	2.4	0.24	3	0 21 28 35	7.0 c 1.9 2.5 3.0 3.5 c 0.76	3.4 1.0 0.6 0.9	DE/FR/03/92, DU/60/92
Germany, 1992 (Muller Thurgau)	WG	2.4	0.24	3	0 20 28 34	3.8 c 0.76 1.9 2.9 2.7 c 0.51	2.5 1.2 0.5 1.2	DE/FR/03/92, DU/61/92

c: control sample.
dt: other dithiocarbamates also applied during the growing season.

Table 31. Residues of thiram (expressed as thiram) in strawberries from foliar application of thiram in supervised trials in Belgium, France, Germany and Poland. Underlined residues are from treatments according to GAP and are valid for estimation of maximum residue levels.

Country, year (variety)	Application				PHI, days	Residues, as thiram, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		CS_2 method	HPLC method	
Belgium, 1991 (Elsanta, June bearing)	WP	1.6	0.23	8	0 7 14 21	3.0 <u>2.4</u> 0.44 0.05	3.3 <u>2.8</u> 0.45 0.06	CRP/92/863A
Belgium, 1991 (Selva Everbearing)	WP	1.6	0.23	3 4 5 6 7 8 9 10 11 12	11 7 6 7 8 7 8 6 8 7	0.32 0.85 1.0 1.0 1.2 <u>1.4</u> 1.1 1.0 1.8 1.2	0.32 0.72 0.97 0.93 1.3 1.3 1.1 0.99 0.92 1.5	CRP/92/865
Belgium, 1991 (Vicoda, June bearing)	WG	1.6	0.23	7	0 7 14 21	2.8 <u>1.2</u> 0.82 0.72	2.7 <u>1.4</u> 0.87 0.79	CRP/92/862A
Belgium, 1991 (Selva, Everbearing)	WG	1.6	0.23	3 4 5 6 7 8 9 10 12 12	11 7 6 7 8 7 8 6 1 7	0.85 0.97 0.85 1.9 0.91 1.4 0.89 1.6 1.2 1.3	1.0 1.1 1.0 <u>2.1</u> 0.84 1.2 0.92 2.0 1.1 1.6	CRP/92/864A
Belgium, 1992 (Selva, Everbearing)	WG	1.6 dt	0.23	5 6 7 8 9 10 11 12 13 14	7 7 7 6 7 7 7 6 7 7		2.3 <u>2.4</u> 0.64 2.2 1.4 1.3 1.2 1.5 4.5 5.8	BCSEL BA 7792a
Belgium, 1992 (Irvine, Everbearing)	WG	1.6	0.23	5 6 7 8 9 10 11 12 13 14	7 7 7 7 8 7 6 6 7 7		<u>3.1</u> 2.5 1.0 1.5 1.1 3.8 2.3 4.3 6.3 11	BCIRV BA 7792d
Belgium, 1993 (Elsanta)	WG	1.6	0.29-0.41	6	0 3 7 10 14		16 1.7 <u>2.1</u> 1.7 0.45	UCB B 93-3 B27-93 9301-RU-010-1
France, 1988 (Elvira)	WG	2.2	0.22	2	14	0.70		FR5 552/6
France, 1988 (Gorella)	WG	2.2	0.29	3 dt	13	0.92		FR8 850/6
France, 1988 (Gorella)	WG	3.2	0.42	3 dt	13	1.4		FR8 850/7
France, 1988 (Elvira)	WG	3.2	0.32	2	14	0.85		FR5 552/7
France, 1988 (Gorella)	WG	3.2	0.32	2	27	1.1		FR5 551/7
France, 1988 (Gorella)	WG	2.2	0.22	2	27	0.68		FR5 551/6
France, 1989 (Gorella)	WG	2.2	0.22	2	32	<0.2		F 869/89/547
France, 1989 (Gorella)	WG	2.2	0.22	2	21	0.8		F 869/89/548

Country, year (variety)	Application				PHI, days	Residues, as thiram, mg/kg		Ref.
	Form	kg ai/ha	kg ai/hl	No.		CS_2 method	HPLC method	
France, 1989 (Selva)	WG	2.2	0.22	2 dt	19	1.1		F 869/89/847
France, 1992 (Selva)	WG	3.2 PCT[13]	0.32	2	0 2 4 8 10 14	1.3 3.7 2.8 2.1 1.6 1.5 1.4		SPV 33 02
France, 1992 (Seascape)	WG	3.2 PCT	0.32	2	0 2 4 10 14	1.2 4.1 2.3 1.7 0.79 0.71		SPV 33 01
France, 1993 (Elsanta)	WG	2.4	0.35-0.54	4	0 3 7 10 14		7.7 4.1 2.3 1.0 0.54	UCB B93-10, 93101-RU-010-3, B93501
France, 1994 (Chandler)	WG	2.4	0.24	3	0 3 7 10 14		7.4 2.4 2.8 2.9 1.5	94020/01-FPEB loc 2
France, 1994 (Chandler)	WG	2.4	0.24	3	0 3 7 11 15		2.8 3.6 5.7 2.8 3.4 3.8 0.9 1.5 1.4 0.6 0.8 1.3 0.4 0.4 0.6	94020/01-FPEB loc 1
Germany, 1962 (Senga Sengana)	WP	0.96	0.16	1	0 3 7 14	0.2 <0.2 <0.2 <0.2		BBA
Germany, 1963 (Senga Sengana)	WP	2.4	0.4	1	3	<0.2		BBA
Germany, 1964 (Senga Sengana)	WP	0.49	0.025	3	10	0.79		BBA
Germany, 1964 (Senga Sengana)	WP	4 4 4 4	0.16 0.16 0.16 0.16	4 3 2 1	12 19 25 31	2.2 1.7 0.79 0.32		BBA
Germany, 1964 (Senga Sengana)	WP	2.4	0.4	3	10	1.7		BBA
Poland, 1994 (Senga Sengana)	WG	3.2		3	0 3 14	8.1 4.4 1.2		

dt: other dithiocarbamates also applied during the growing season

Table 32. Residues of thiram (expressed as thiram) in vegetables from seed treatment and foliar application of thiram in supervised trials in France, Germany and The Netherlands. Underlined residues are from treatments according to GAP and are valid for estimation of maximum residue levels.

VEGETABLES Country, year (variety)	Application	PHI, days	Residues, as thiram, mg/kg	Ref.

[13] Outdoor, plastic covered tunnel

	Form	kg ai/ha	kg ai/hl	No.		CS_2 method	HPLC method	
BEANS, DWARF FRENCH								
Germany, 1981 (Saxa)	DS	0.2 kg ai/100 kg seed			67	<0.1		BBA 3781
BEANS, FRENCH								
France, 1987 (Faria)	WG	2.2	0.45	1	20	0.32		42308 F/18/5
France, 1987 (Faria)	WG	2.2	0.45	2	7	0.22		42308 F/18/8
France, 1987 (Carlyn)	WG	2.2	0.45	1	28	0.25		FR2 52/5
France, 1987 (Carlyn)	WG	2.2	0.45	2	17	0.44		FR2 52/8
France, 1988 (Contender)	WG	2.2	0.45	2	16	0.32		F972/88/B4
France, 1988 (Contender)	WG	2.2	0.22	1	26	<0.2		F972/88/C4
CABBAGE, SAVOY								
Germany, 1981 (Marner Frühkopf)	DS	0.20 kg ai/ 100 kg seed			90	0.06		BBA 3681
LETTUCE, HEAD								
Germany, 1970 (Rapide)	DP	9.8	dust	g 1	59	<0.5		BBA 415/70
Germany, 1970 (Kordaat)	DP	9.8	dust	g 1	70	<0.5		BBA 1a/71
Germany, 1970 (Rapide)	WP	16	dust	g 2	45	<0.5		BBA 414/70
Germany, 1973 (Marty)	WP	0.96	0.16	g 1	21	4.0		BBA 26/73
Netherlands, 1975 (Zwart-duits)	WP	2.0	0.20	2	14 21 28	3.4 6.0 3.3 2.6 0.37 0.62 1.1 0.67 ≤0.2 (4)		KvW194 (1976)
Netherlands, 1975 (Zwart-duits)	WP	2.0	0.20	2	14 21	1.5 0.88 0.56 0.78 0.25 0.24 ≤0.2 (2)		KvW194 (1976)
Netherlands, 1980 (Desi minor)	WP	2.0	0.18	g 1	28 42	0.9 0.7 0.7 0.9 <0.3 (2) 0.3 0.4		KvW225 (1982)
Netherlands, 1980 (Desi minor)	WP	2.0	0.18	g 1	28 42	2.4 1.3 0.7 3.0 <0.3 (4)		KvW225 (1982)
PEAS, GREEN								
France, 1987 (Zorba)	WG	2.2	0.45	1 2	29 16	<0.2 0.22		42308 F87/INRA
SPINACH								
Germany, 1981 (Atlanta)	DS	0.20 kg ai/ 100 kg seed			40	leaves <0.03		BBA 3581
Netherlands, 1975 (Bergola)	WP	2.0	0.20	g 1	28 28 31 31	7.2 9.6 10 10 c <0.2 (2) 1.1 0.96 0.92 0.85 c 0.29 0.27		KvW189 (1975)
Netherlands, 1975 (Bergola)	WP	2.0		g 1	28	14 5.4 4.2 4.8 c <0.2 (2) 0.28 0.26 0.43 0.38 c 0.29 0.27		KvW189 (1975)
TOMATO								
France, 1988 (Rio Grande)	WG	2.2	0.45	4 dt	11	0.17 0.36		549/4 549/5
France, 1988 (Apla)	WG	1.3	0.67	4	19	<0.2 <0.2		C 16/4 C 16/5
France, 1989 (Trésor)	WG	2.2	0.22	4	8	<0.2		F885/89/4
France, 1989 (Trésor)	WG	3.2	0.32	4	8	<0.2		F885/89/5
France, 1989 (Donna)	WG	2.2	0.22	4	10	0.95		F885/89/50/4
France, 1989 (Donna)	WG	3.2	0.32	4	10	1.1		F885/89/50/5
France, 1989 (Donna)	WG	2.2	1.1	3	28	<0.2		F985/89/4
France, 1989 (Donna)	WG	3.2	1.6	3	28	<0.2		F985/89/5

g: glasshouse c: control sample dt: other dithiocarbamates also applied during the growing season.

FATE OF RESIDUES IN STORAGE AND PROCESSING

In storage

No information was available on the fate of thiram residues in commodities in commercial storage.

In processing

Information was provided on the fate of thiram during the processing of apples to juice and pomace and during the production of wine.

In an apple processing study in the USA thiram was applied 13 times at 11.6 kg ai/ha during the growing period in 1993 to Twenty-ounce apples on a farm in New York State (Armstrong, 1993; Leppert, 1995; Tufts, 1995). Apples (50 kg) were harvested 7 days after the final application and converted to juice and pomace according to a simulated commercial process. There was no washing step; the trial was designed to represent a "worst case" for residues. Thiram residues in apples, juice and pomace were measured by a CS_2 evolution GLC head-space method. The thiram residue level in the juice was about 30% of that in the apples, while the level in wet pomace was the same as in the apples. Wet pomace was heated at 77-88°C until the weight had decreased to 20-25% to produce dry pomace. The increase in the thiram level suggested that little of the residue had been lost during the drying process. The residues in the analysed commodities were as shown below.

	Thiram residue
apples	11.5 mg/kg
juice	3.3 mg/kg
wet pomace	11.8 mg/kg
dry pomace	42 mg/kg

In grape processing studies in France field-sprayed grapes were processed into juice, wine and raisins (Blaschke, 1995a,b) and only wine (Blaschke, 1995c,d). The thiram results are summarized in Table 33. Samples other than wine had been stored for about 18 months at ≤18°C before analysis. Wine was kept at 4-8°C.

Approximately 50 kg of grapes were crushed to produce wine, while 3 kg and 6 kg were used for juice and raisin production respectively. Crushing took place in September and the wine was bottled (for analysis) about 6 months later. The champagne in trial 28203B004 (Blaschke, 1995d) was matured in the bottles for an additional year. Wet pomace was dried in an oven for 5 to 8 days and lost 50-60% of its weight.

Grapes were stemmed manually and crushed to produce juice, which was heated to 85-88°C for 5 minutes and then refrigerated for up to 5 days. The supernatant clear juice was bottled and sterilized by heating at 100°C for 20 minutes.

In raisin production, the grape bunches were dried in an oven at 60°C for about 6 days and lost 84-91% of their weight. After drying, the stems were manually removed.

Although no other dithiocarbamate fungicides had been used on the crops (except in trial 28203B004 where mancozeb had been used at a very early stage approximately 17 weeks before harvest) the residue levels of thiram calculated from the CS_2 evolution method were substantially higher than thiram measured by HPLC. CS_2 residues were not detected in wine produced from untreated grapes and in only four grape samples from untreated plots (trial 28203B002, 0.08 and 0.07 mg/kg as CS_2, and trial 28203B002, 0.10 and 0.08 mg/kg as CS_2) which suggests the production of thiram metabolites containing the CS_2 group and with sufficient persistence and water-solubility to enter the wine. An unconfirmed possibility is that the liberated CS_2 was derived from rubber tubing or gloves.

Table 33. Residues of thiram (expressed as thiram) in grapes, juice, pomace, must, wine and raisins after foliar application of thiram to grapes in supervised trials in France. The application conditions in the trials are listed in Table 30 (supervised trials on grapes).

Commodity	Residues, as thiram, mg/kg							
	Blaschke, 1995b (France, 28203B001)		Blaschke, 1995a (France, 28203B002)		Blaschke, 1995c (France, 28203B003)		Blaschke, 1995d (France, 28203B004)	
HPLC method								
Grapes (PHI, days)	1.6 (33)	4.3 (22)	1.2 (33)	1.2 (22)	1.4 (49)	1.9 (22)	1.2 (34)	3.0 (22)
Juice	<0.1		<0.1	<0.1				
Must	<0.1	0.2	<0.1	<0.1	1.3	1.7	<0.1	0.3
Wet pomace	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Dry pomace	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Wine	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Raisin	5.7	4.9	<0.1	<0.1				
CS_2 method								
Grapes (PHI, days)	9.3 (32)	12 (21)	1.3 (32)	4.3 (21)	3.0 (48)	7.4 (21)	2.4 (33)	2.5 (21)
Wine	0.9	0.9	0.22	0.25	0.98	0.74	0.12	0.19

Residues in the edible portion of food commodities

The thiram level in apple juice was about 30% of the level in the apples.

Thiram residues in grape juice and wine, by an HPLC method, were undetectable (<0.1 mg/kg).

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

Monitoring data for residues of dithiocarbamates (including thiram) in fruit and vegetable commodities in trade were available from The Netherlands, Belgium and Denmark (Tables 34-37).

Table 34. Residues of thiram (expressed as thiram) in domestic fruit and vegetable commodities in trade in The Netherlands, 1976.

Commodity	Number of samples				
	Analysed	No residues (≤0.05 mg/kg)	Residues >0.05 ≤1, mg/kg	Residues >1 ≤3, mg/kg	residues >3 mg/kg
Endive	100	98	0	1	1
Lettuce	590	582	6	1	1
Parsley	8	8	0	0	0
Celery	29	29	0	0	0
Spinach	52	52	0	0	0
Cauliflower	22	22	0	0	0
Brussels sprouts	48	48	0	0	0
Butter beans and French beans	16	15	1	0	0
Chicory	19	18	1	0	0
Leek	30	30	0	0	0
Strawberry	45	4	6	19	16[1]

[1] Residues of thiram exceeded the MRL in 12 strawberry samples. Levels were 4.2, 4.8, 5.0, 5.1, 5.5, 6.1, 7.8, 8.8, 8.9, 14, 14 and 15 mg/kg.

Table 35. Netherlands monitoring of food in commerce for dithiocarbamate residues for 1991-1994.

Commodity	Number of samples			
	Analysed	No residues (1991-93 LOD 0.2 mg/kg, 1994 LOD 0.05 mg/kg)	Residues detected, <MRL (NL)	Residues >MRL (NL)
Citrus fruit	64	59	4	0
Pome fruit	609	522	87	0
Stone fruit	404	362	42	0
Berries and small fruit	2478	2152	324	2[1]
Fruit, trop and sub-trop	180	164	16	0
Root and tuber veg.	332	310	19	3[2]
Bulb veg.	36	36	0	0
Fruiting veg.	731	656	75	0
Brassica veg.	167	126	41	0
Leaf and herb veg.	4356	3755	576	25[3]
Stem veg.	948	911	36	1[4]
Mushrooms	81	79	2	0
Pulses	89	83	6	0

[1] Fruits exceeding the MRL were grapes (1 of 549 samples) and "other small fruit" (1 of 39 samples)
[2] Residues in celeriac exceeded the MRL in 3 of 119 samples
[3] Vegetables with residues exceeding the MRLs were lamb's lettuce (8 of 267 samples), head lettuce (2 of 1517 samples), other lettuce (6 of 377 samples), endive (6 of 744 samples), spinach (2 of 204 samples) and parsley (1 of 359 samples)
[4] The MRL was exceeded in a sample of leeks (1 of 405 samples)

Dejonckheere *et al.* (1996) published a report on pesticide residues in fresh vegetables, fruits, and other selected food items in Belgium for 1991-1993. The survey included dithiocarbamate residues, which were measured by a CS_2 evolution colorimetric method (Table 36). The residues were detected more often and at higher levels in leafy vegetables.

Table 36. Dithiocarbamate residue monitoring data for Belgium for 1991-1993 (Dejonckheere *et al.*, 1996).

Commodity	Number of samples		LOD, mg/kg	Max detected residue, mg/kg
	Analysed	Residues detected		
Celery leaves	100	16	0.2	41
Endive	75	13	0.2	19
Grapes	108	0	0.2	<0.2
Lamb's lettuce	100	16	0.2	55
Leeks	108	2	0.2	2.8
Lettuce	112	16	0.2	33
Strawberries	73	1	0.2	5.2

Juhler *et al.* (1996) included dithiocarbamate residues in the 1994 survey of pesticide residues in Danish food by the National Food Agency of Denmark (Table 37). The samples were analysed by a colorimetric CS_2 evolution method. The LOD for each crop was not explicitly stated, but for this method would be expected to be close to 0.1 mg/kg. In many commodities dithiocarbamates were detected in 10-35% of the samples.

Table 37. Dithiocarbamate residue monitoring data for Denmark for 1994 (Juhler *et al.*, 1994).

Commodity	Domestic or import	Number of samples		Max detected residue, mg/kg (as CS_2)
		Analysed	Residues detected	
Apples	D	39	3	0.16
Apples	I	43	10	0.65
Apricots	I	2	1	0.12
Carambolas	I	6		
Celery	D	20		
Celery	I	20		
Cherries	D	13	4	0.14
Cherries	I	15	4	0.75
Cucumbers	I	16	5	0.35
Currants, black	D	10		
Currants red/white	D	10	1	0.4
Currants red/white	I	6	2	1.1
Gooseberries	D	7	3	0.37
Grapefruit	I	19	1	0.13
Grapes	I	63	10	0.75
Kiwifruit	I	37		
Lemons, limes	I	7		
Lettuce, iceberg/head	D	35	7	0.48
Lettuce, iceberg/ head	I	40	6	1.7
Mandarins/ clementines	I	23	2	0.33
Mangos	I	8	1	0.1
Nectarines	I	19	7	0.75
Oranges	I	27	2	0.18
Papayas	I	5	2	0.20
Passion fruit	I	2	1	1.6
Peaches	I	17	12	0.72
Pears	D	16	2	0.21
Pears	I	24	7	0.53
Peppers	I	27		
Plums	D	20	7	0.56
Plums	I	31	4	0.55
Potatoes	D	42		
Potatoes	I	25	2	0.19
Raspberries	D, I	12		
Strawberries	D	25	7	0.3
Strawberries	I	31	7	0.65
Tomatoes	I	26	5	0.29

NATIONAL MAXIMUM RESIDUE LIMITS

The Meeting was aware of the following national MRLs (see explanatory notes on next page).

Country (residue definition)	Commodity and MRL
Argentina (CS)	apples 2; beans 0.5; carrots 0.1; celery 3; cherries 1; grapes 5; lettuce 3; melons 1; peaches 3; pears 2; plums 1; potatoes 0.1; tomatoes 3; turnips 0.1
Australia (CS)	fruits 7; vegetables 7
Austria (DT)	fruits 2; vegetables 2
Belgium (T)	apples 3; apricots 3; bananas 3; cherries 3; grapes 3.8; other fruits 3; peaches 3; pears 3; plums 3; potatoes 0.05; strawberries 3.8; vegetables 3
Brazil (T)	bananas 1; beans 7; peas 7
Canada (T)	apples 7; bananas 1; beans 7; celery 7; peaches 7; pears 7; strawberries 7; tomatoes 7
Chile (DT)	apples 3; carrots 0.5; cereals 0.2; cherries 1; grapes 5; lettuce 1; peaches 3; pears 3; plums 1; potatoes 0.1; tomatoes 3
Denmark (DT)	apples 2; apricots 2; bananas 2; bulb vegetables 0.5; carrots 0.5; cherries 2; foliar vegetables 2; grapes 2; other fruits 2; peaches 2; pears 2; plums 2; potatoes 0.1; strawberries 2; vegetables 1
EEC (T)	apples 3; apricots 3; bananas 3; cherries 3; grapes 3.8; other fruits 3; peaches 3; pears 3; plums 3; strawberries 3.8; vegetables 3
France (DT)	apples 1; apricots 1; bananas 2; beans 0.5; bulb vegetables 0.5; cherries 1; grapes 1; lettuce 4; other fruits 2; peaches 1; pears 1; plums 1; potatoes 0.05; tomatoes 1; vegetables 2
Germany (T)	apples 3; apricots 3; bananas 3; cherries 3; grapes 4; other fruits 3; peaches 3; pears 3; plums 3; potatoes 0.2; strawberries 4; tomatoes 1; vegetables 3
Greece (T)	apples 3; apricots 3; bananas 3; cherries 3; grapes 3.4; other fruits 3; peaches 3; pears 3; plums 3; strawberries 3.4; vegetables 3
Ireland (T)	apples 3; apricots 3; bananas 3; cherries 3; grapes 4; other fruits 3; peaches 3; pears 3; plums 3; strawberries 4; vegetables 3
Israel (DT)	apples 3; bananas 1; beans 0.2; carrots 0.2; celery 5; cherries 1; grapes 5; lettuce 0.5; melons 0.5; peaches 3; pears 3; plums 1; potatoes 0.1; strawberries 3; tomatoes 3
Italy (T)	apples 3; apricots 3; bananas 3; cherries 3; grapes 3.8; other fruits 3; peaches 3; pears 3; plums 3; potatoes 2; strawberries 3.8; vegetables 3
Japan (T)	apples 1; peaches 0.5; pears 0.5
Luxembourg (DT)	apples 2; apricots 2; beans 2; carrots 0.5; celery 0.2; cherries 1; garlic 0.5; grapes 2; lettuce 5; onions 0.5; peaches 2; pears 2; peas 2; plums 1; strawberries 2; tomatoes 2
Mexico (T)	apples 7; celery 7; peaches 7; strawberries 7; tomatoes 7
Netherlands (NL)	apricots 2; beetroot[14] 2; Brassica vegetables[14] 2; bulb vegetables 0.5; cane fruit (other than wild) 3; carrots[14] 2; celeriac[15] 2; celery[14] 2; cherries[16] 2; courgettes[14] 2; cucumbers[14] 1; currants (red black white) 3; gherkin 2; globe artichokes[14] 2; gooseberries 3; hops 25; leek[14] 2; legume vegetables (fresh)[14] 2; maize and rice[17] 0.5; melons[14] 1; nectarines 2; nuts[18] 2; oil seeds 0.1*; oranges 2; other cereals 0.5; other citrus fruit[14] 2; other cucurbits (inedible peel)[14] 2; other food commodities 0.05*; other Solanaceae 2; parsnips[14] 2; peaches 2; plums[16] 2; pome fruits[14] 2; radish[14] 2; salsify[14] 2; spinach and similar[17] 2; strawberries (other than wild)[19] 3; sweet corn[17] 2; table and wine grapes[19] 3; tea 0.1*; tomatoes[14] 2; water cress[14] 2; witloof[15] 2
New Zealand (DT)	fruits 7; vegetables 7
Poland (CS)	cereal grains 0.05; cucumber 1; eggs 0.05; fruits 2; leafy vegetables 5; meat and meat products (in fat) 0.05; milk and milk products 0.05; stalk and stem vegetables 5,; tomato 1; vegetables other 2
Singapore (T)	fruits 3; vegetables 3
South Africa (CS)	apples 3; apricots 3; grapes 3; peaches 3; pears 3; plums 3
Spain (DT)	apples 3; apricots 3; bananas 3; cherries 3; grapes 4; other fruits 3; peaches 3; pears 3; plums 3; potatoes 0.2; strawberries 4; vegetables 3
Sweden (DT)	carrots 0.5; fruits 1; potatoes 0.1; vegetables 1
Switzerland (DT)	foliar vegetables 2; fruits 2; potatoes 0.05; vegetables 2
UK (DT)	apples 3; apricots 3; bananas 1; beans 0.5; carrots 0.5; cherries 3; grapes 5; peaches 3; pears 3; plums 1; potatoes 0.1; strawberries 3; tomatoes 3
USA (T)	apples 7; banana 7; celery 7; cereals 0.5; onions 0.5; peaches 7; strawberries 7; tomatoes 7

* At or about the LOD

Residue definitions:
(T) thiram
(CS) mg CS_2/kg

[14] Residues from mancozeb, maneb, metiram, propineb and zineb, total with a maximum of 0.5 mg CS_2/kg.
[15] Residues from mancozeb, maneb, metiram, propineb and zineb, total with a maximum of 0.2 mg CS_2/kg.
[16] Residues from mancozeb, maneb, metiram, propineb and zineb, total with a maximum of 1 mg CS_2/kg.
[17] No residues from mancozeb, maneb, metiram, propineb and zineb (LOD 0.05 mg CS_2/kg).
[18] No residues from mancozeb, maneb, metiram, propineb and zineb (LOD 0.1 mg CS_2/kg).
[19] Residues from mancozeb, maneb, metiram, propineb and zineb, total with a maximum of 2 mg CS_2/kg.

(DT) sum of dithiocarbamates (mg CS_2/kg)
(NL) all compounds that give CS_2, sum of dithiocarbamates, expressed as CS_2 in mg/kg

APPRAISAL

Thiram was originally evaluated in 1965 (toxicology) and 1967 (toxicology and residues) and is included in the dithiocarbamate group of compounds. It was evaluated at the present Meeting within the CCPR periodic review programme.

Thiram is a protective dithiocarbamate fungicide used as a foliar treatment on fruits, vegetables and ornamentals to control *Botrytis* species, rust, scab and storage diseases, and as a seed treatment to control seedling blights and a number of fungi that cause "damping off" in seedlings. Thiram formulations are registered for use in many countries. The Meeting was provided with information on registered uses on fruits, vegetables and other crops.

The Meeting received extensive information on the metabolism of thiram in rats, farm animals, apples, grapes, soya beans, cotton, wheat and sugar beet, its environmental fate in soil and water/sediment systems, methods of residue analysis, the stability of residues in stored analytical samples, approved use patterns, supervised residue trials and the fate of residues during processing.

When rats were dosed orally with [*thiocarbonyl*-^{14}C]thiram much (40-60%) of the ^{14}C was eliminated as volatile compounds in exhaled air, 25-35% was excreted in the urine and 2-5% in the faeces. The volatiles were collected in traps suggesting the presence of CS_2, CO_2 and COS. Five polar metabolites and conjugates were identified in the urine: 2-thioxo-4-thiazolidinecarboxylic acid, dimethyldithiocarbamoyl glucuronide, dimethyldithiocarbamoylsulfenic acid, methyl dimethyldithiocarbamate, and dimethyldithiocarbamoylalanine.

A major part of the ^{14}C was eliminated in respiration gases from lactating goats dosed with [*thiocarbonyl*-^{14}C]thiram equivalent to 2.5, 3.3 and 23 ppm in the feed for 4 consecutive days. Most (90% or more) of the ^{14}C in the expired air was present in CO_2, with the remainder in CS_2 and COS. The levels of ^{14}C in the milk were quite low and reached their plateaux within 1.5 to 3 days of the first dose. The total ^{14}C in the milk constituted 1.0-1.8% of the administered dose. The levels of ^{14}C were much higher in the liver than in the other tissues.

The metabolism of thiram in goats was quite extensive and much of the ^{14}C in the milk and tissues was present as very polar extractable material or as bound residues. It is likely that thiram is rapidly converted to dimethyldithiocarbamic acid and then to dimethylamine and CS_2. CS_2 is converted to COS and carbonate. [^{14}C]carbonate then enters fat, protein and carbohydrates.

When laying hens were dosed with [*thiocarbonyl*-^{14}C]thiram equivalent to 0.6 and 6.0 ppm in the feed for 4 consecutive days approximately 1% of the dose was present in the liver, which had higher levels than the other tissues. Levels of ^{14}C in egg white and egg yolk were quite low throughout the 4 days, with approximately 0.15% of the administered ^{14}C appearing in the eggs.

About half of the ^{14}C in the liver was incorporated into natural products such as acids, amino acids, peptides and proteins. Three metabolites constituting only a small percentage of the ^{14}C were identified as dimethyldithiocarbamoylornithine, 2-thioxo-4-thiazolidinecarboxylic acid and dimethyldithiocarbamoyl glucuronide.

Apples and leaves on apple trees were treated with [*thiocarbonyl*-^{14}C]thiram and examined for ^{14}C periodically after treatment. Initially most of the ^{14}C was on the fruit and leaf surfaces, but by day 14 only half of the remaining residue was on the surface. By day 101 only 2.7% of the residue was on the surface with 38% in the peel and 60% in the pulp. Thiram itself was not detected within the fruit except on day 0. The residue incorporated in the fruit contained only a small percentage of the dimethyldithiocarbamoyl moiety as demonstrated by the release of small amounts of CS_2 by acid digestion, equivalent to 1-3.5% of the incorporated ^{14}C.

When grapes and vine leaves were treated with [*thiocarbonyl*-^{14}C]thiram the ^{14}C residues on and within the fruit were quite persistent. The residues on the leaf surfaces disappeared more quickly. In grapes harvested 27 days after the final thiram application 35% of the remaining ^{14}C was in surface washings, 44% in juice and 21% in press cake.

Most of the ^{14}C in the surface washings from grapes after 27 days was in thiram itself, but HPLC showed the presence of two metabolites both more polar than thiram. Approximately 5% of the ^{14}C residue within the grapes (harvested 0, 14 or 27 days after the final treatment) liberated CS_2 on acid digestion, which demonstrated that very little of it contained the dimethyldithiocarbamoyl moiety. Most of the ^{14}C residue in the juice was shown to have a molecular weight below 500, but could not be positively identified. Much of the ^{14}C in the grapes was very polar or unextractable and had probably become incorporated into natural products.

When soya bean, cotton and wheat plants were grown from [*thiocarbonyl*-^{14}C]thiram-treated seed, the ^{14}C levels in the cotyledons and roots of the seedlings were higher than in the leaves and stems. In mature plants the highest ^{14}C levels were in the roots and the lowest in the seeds.

The major metabolites in soya bean, cotton and wheat seedlings were identified as dimethyldithiocarbamoyl and dimethylthiocarbamoyl glycosides. When an aqueous wheat extract was treated with hot acid only 3.4% of the ^{14}C was liberated as CS_2, suggesting that if any remaining metabolites contained the dithiocarbamoyl moiety they were largely unextractable.

In further studies on soya beans and wheat produced from thiram-treated seed it was shown that much of the ^{14}C had been incorporated into endogenous natural products such as sugars, fatty acids and citric acid, but some of the dimethyldithiocarbamoyl moiety had conjugated with amino acids and sugars. Thiram itself was not detected.

The main metabolite identified was 2-dimethylamino-4-thiazolinecarboxylic acid, apparently produced from dimethyldithiocarbamoylalanine. When homogenized soya bean tissue (forage, straw, pod and seed) was digested with acid 3-9% of the ^{14}C in each tissue were released as CS_2 and 3-24% as CO_2. In straw, chaff and wheat grain the corresponding figures were 2-9% and 8-21%.

^{14}C levels in sugar beet tops and roots were generally very low in plants produced from [*thiocarbonyl*-^{14}C]thiram-treated seed. As in the soya bean and wheat studies ^{14}C was detected in control plants growing nearby, suggesting that $^{14}CO_2$ had been produced. Only 3.2% of the ^{14}C in the roots was released as CS_2 when the homogenized tissue was digested with acid, demonstrating that very little of the incorporated ^{14}C contained the dithiocarbamoyl moiety.

Thiram, [*thiocarbonyl*-^{14}C]-labelled, disappeared rapidly when incubated in a sandy loam soil under aerobic conditions at 20°C and 75% of field moisture capacity, with an initial half-life of about 2 days and 85% disappearance in 7 days. Mineralization was also rapid with 9% of the applied ^{14}C evolved as $^{14}CO_2$ in 2 days and 50% within 21 days. The major metabolite was identified as dimethylcarbamoperoxothioic acid, which reached its maximum concentration on day 4 of the incubation. There were a number of other minor metabolites, three of which were identified.

Labelled thiram disappeared with a half-life of 3.7 days when exposed on a thin layer of sandy loam soil to simulated sunlight. After 21 days the volatile ^{14}C amounted to 57%, 37% as CO_2 and 20% corresponding to CS_2 in an HPLC system, but not fully identified.

The adsorption and desorption properties of thiram were measured on four soils, a sandy loam, a loamy sand, a silt loam and a loam. Thiram was judged to be slightly mobile to immobile in the soils tested.

When [*thiocarbonyl*-^{14}C]thiram was incubated in aquatic systems of river or pond water and sediment in the dark at 20°C under aerobic conditions for 101 days the initial half-life of thiram was about 2 days with more than 90% disappearance within 7 days. CS_2, CO_2 and methyl dimethyldithiocarbamate were identified as metabolites. CS_2 and the ester reached their peak concentrations in the water on day 4.

The analytical methods for dithiocarbamates which rely on CS_2 evolution may be used for the determination of thiram residues. Such methods have been reviewed previously for mancozeb, maneb and propineb (1993 JMPR) and metiram (1995 JMPR). Methods where the generated CS_2 is measured by colorimetry or by head-space GLC have been shown to be suitable for thiram, as for the other dithiocarbamates. Limits of determination in various commodities are usually 0.05-0.1 mg/kg (as CS_2).

An HPLC method has been developed for thiram residues in crops that measures thiram as

the intact molecule and distinguishes it from other dithiocarbamates. The residue is extracted with solvent from crop samples and, after clean-up on a C18 microcolumn, determined on a C18 reversed-phase HPLC column with UV detection at 280 nm. Quantitative recoveries were achieved from fruit and processed fruit fractions down to 0.1-0.2 mg/kg. Low recoveries occurred in wine fortified at the 0.1 mg/kg level because thiram was being degraded.

Data were available on the frozen storage stability of thiram residues on plums and in apple juice and apple pomace.

Thiram residues were shown to be stable on frozen whole plums stored at a freezer temperature below -20°C for 500 days. At three of the samplings the plums were analysed by a CS_2 evolution method and by an HPLC method specific for thiram. The results were in good agreement. If plums were macerated in a blender thiram was decomposed by exposure to the macerate. It was recommended that fruit for residue analysis should be stored whole, subjected to a minimum of cutting (into halves and quarters) while still frozen, and analysed immediately.

Thiram residues in apple juice, wet pomace and dry pomace fortified at 1 mg/kg were stable at -20 ± 5°C for the intervals tested, 35 to 49 weeks.

The Meeting considered the definition of thiram residues in terms of the crop metabolism studies, the supervised trials where residues had been determined by both a CS_2 evolution method and an HPLC method, and the needs of enforcement agencies.

The metabolism studies suggest that thiram is the major part of the CS_2-evolving residue, particularly when the residue is reasonably fresh and at the higher levels. Analyses of samples in supervised trials by the HPLC and CS_2 methods are usually in good agreement, which also suggests that thiram itself is the main residue.

The 1995 JMPR (Report, Section 2.8.1), in explaining the current basis for the definition of residues, stated "Preferably no compound, metabolite or analyte should appear in more than one residue definition."

The Meeting agreed that thiram should be included in the definition of dithiocarbamate residues:

> *The MRLs refer to total dithiocarbamates, determined as CS_2 evolved during acid digestion and expressed as mg CS_2/kg.*

For dietary intake estimations the supervised trials median residue (STMR) will be expressed as thiram because intakes need to be in terms of thiram itself for comparison with its ADI. For estimates of acute intake a residue such as an MRL, which is expressed in terms of CS_2, must be multiplied by a factor of 1.58 for comparison with an acute reference dose expressed in terms of thiram.

The Meeting received data on thiram residues from supervised trials on apples (Belgium, France, Germany, Italy, The Netherlands, Poland), pears (Belgium, Germany, Italy, Spain), peaches (France, Italy, Spain), plums (France, Italy), cherries (Italy, Spain), grapes (France, Germany), strawberries (Belgium, France, Germany, Poland), dwarf French beans (Germany), French beans (France), Savoy cabbage (Germany), green peas (France), head lettuce (Germany, The Netherlands), spinach (Germany, The Netherlands), and tomatoes (France).

In the trials thiram was determined by CS_2 evolution methods or by HPLC, and in some trials by both methods. Residues are expressed as thiram in the following discussion.

In some trials other dithiocarbamates had been used on the crop during the growing season. Samples from such trials were considered valid for estimating thiram residue levels only if they had been analysed specifically for thiram by an HPLC method.

Thiram is registered for application up to 12 times on pome fruit in Germany at a spray concentration of 0.1-0.16 kg ai/hl or 1.5-2.4 kg ai/ha, with a 10-day PHI. Decline curves from pome fruit trials suggest that thiram residues decrease with a typical half-life of about 10 days and that data from 7-15 days, equivalent to a concentration range of ±30%, are within acceptable range of a 10

days PHI. The decline curves also suggest that applications more than 30-40 days before the final application will contribute less than 10% to the final residues, so they should not be increased by more than 4 or 5 applications.

Apples. Thiram residues in two Belgian trials (spray concentration 0.16 kg ai/hl, PHI 9 days) were 2.7 and 4.6 mg/kg, and from a French trial (spray concentration 0.16 kg ai/hl, PHI 14 days) 2.4 mg/kg. In two German trials, one at 0.2 kg ai/hl, 15 days PHI (which exceeds 14 days but the spray concentration is slightly higher than 0.16 kg ai/hl), and the other at 0.16 kg ai/hl with a 14-day PHI, thiram residues were 0.49 and 1.9 mg/kg. In three Italian trials according to the registered use pattern of 0.15-0.20 kg ai/hl with a PHI of 10 days thiram residues were 2.5, 4.1 and 1.1 mg/kg.

The pome fruit registration in The Netherlands permits thiram application rates of 1.4-3.0 kg ai/ha or spray concentrations of 0.10-0.20 kg ai/hl with a 7-day PHI. The registration is for a WG formulation, but the trials with WP formulations are considered comparable. The highest thiram residues in apples at rates of 2.4-3.1 kg ai/ha at a 7-day PHI in three trials in The Netherlands were 0.46, 1.8 and 6.3 mg/kg. In four other trials thiram residues were generally not detected (<0.1 mg/kg) at a PHI of 7 days, but the data could not be used because sample storage conditions before analysis were not available.

Four Polish trials in which apples were treated at 2.4 kg ai/ha and harvested 14 days later were evaluated according to the German use pattern on pome fruit (1.5-2.4 kg ai/ha and 10 days PHI). Thiram residues were 3.8, 3.2, 0.87 and 1.7 mg/kg.

In summary thiram residues in apples from trials according to GAP were Belgium 2.7, 4.6 mg/kg, France 2.4 mg/kg, Germany 0.49, 1.9 mg/kg, Italy 1.1, 2.5, 4.1 mg/kg, The Netherlands 0.46, 1.8, 6.3 mg/kg and Poland 1.7, 3.2, 3.8 mg/kg. The 15 residues in rank order (median underlined) were 0.46, 0.49, 0.87, 1.1, 1.7, 1.8, 1.9, 2.4, 2.5, 2.7, 3.2, 3.8, 4.1, 4.6 and 6.3 mg/kg.

Pears. Two trials in Belgium (2.4 kg ai/ha, 13 and 14 days PHI) and two in Germany (2.4 kg ai/ha, 10 days PHI) were evaluated against German GAP for pome fruit (2.4 kg ai/ha and 10 days PHI). The thiram residues were 0.69, 1.6, 1.9 and 0.90 mg/kg. Residue levels resulting from 12 and 14 applications (0.69 and 1.6 mg/kg) were similar to those from 4 applications (0.90 and 1.9 mg/kg).

Three trials in Italy (0.15 kg ai/hl, 10 days PHI) were according to Italian GAP for pome fruit (0.15-0.20 kg ai/hl, 10 days PHI). The thiram residues were 0.54, 4.3 and 5.1 mg/kg. A trial in Spain (0.24 kg ai/hl 14 days PHI) was according to Spanish GAP for pome fruit (0.16-0.24 kg ai/hl, 15 days PHI). The thiram residue was 3.0 mg/kg.

The thiram residues in pears from each of the eight trials in rank order (median underlined) were 0.54, 0.69, 0.90, 1.6, 1.9, 3.0, 4.3 and 5.1 mg/kg.

The Meeting noted that the registered use patterns were for pome fruits, that the use patterns in the apple and pear trials were similar and that the residue levels in the two fruits overlapped. The Meeting therefore agreed to evaluate the combined apple and pear data as applying to pome fruits. The thiram residues in apples and pears taken together in rank order (median underlined) were 0.46, 0.49, 0.54, 0.69, 0.87, 0.90, 1.1, 1.6, 1.7, 1.8, 1.9, 1.9, 2.4, 2.5, 2.7, 3.0, 3.2, 3.8, 4.1, 4.3, 4.6, 5.1 and 6.3 mg/kg. The highest residue, 6.3 mg/kg as thiram, is equivalent to 4.0 mg/kg dithiocarbamates as CS_2.

The Meeting estimated a maximum residue level of 5 mg/kg for dithiocarbamates in pome fruits arising from the use of thiram, and noted that this value was the same as the current recommendation for dithiocarbamates in pome fruits. The Meeting estimated an STMR of 1.9 mg/kg for thiram (as thiram) in pome fruit.

Stone fruits. The Italian registered use for thiram on stone fruit permits a spray concentration of 0.15 kg ai/hl and a PHI of 10 days. Thiram residues in peaches in 2 Italian trials matching these conditions were 2.7 and 3.6 mg/kg, and in two Spanish trials under conditions close to Spanish GAP for stone fruit (0.16-0.24 kg ai/hl, 15 days PHI, 14 days in the trials) they were 0.26 and 0.70 mg/kg.

In a French trial on plums which was also according to Spanish GAP the highest residue 14 days after the last of 3 applications was 1.0 mg/kg. In two Italian trials on plums according to Italian GAP for stone fruit the highest residues on day 10 were 0.83 and 0.62 mg/kg.

In two Italian trials on cherries according to Italian GAP the highest residues at the recommended PHI of 10 days were 0.37 and 0.41 mg/kg, but in the second trial a residue of 1.1 mg/kg occurred 15 days after the final treatment. Thiram residues of 0.1 mg/kg were found in cherries from a Spanish trial where the conditions were close to Spanish GAP.

The residue levels in peaches from 2 of the 4 relevant trials appeared to be outside the general population of the stone fruit residues, but 4 trials on peaches were in any case insufficient to support an MRL. The Meeting concluded that the residues in plums (0.62, 0.83 and 1.0 mg/kg) and cherries (0.1, 0.37 and 1.1 mg/kg) from the valid trials could be evaluated together. The residues in rank order (medians underlined) were 0.1, 0.37, 0.62,0.83, 1.0 and 1.1 mg/kg. The highest residue, 1.1 mg/kg as thiram, is equivalent to 0.69 mg/kg dithiocarbamates as CS_2.

The Meeting estimated a maximum residue level of 1 mg/kg for dithiocarbamates (as CS_2) in plums and cherries arising from the use of thiram, and an STMR of 0.72 mg/kg for thiram (as thiram) in plums and cherries.

Berries and other small fruits. Thiram trials on grapes in France and Germany could not be evaluated because corresponding GAP information was not available.

Thiram trials on strawberries in France could not be evaluated because corresponding information on GAP was not available. Full details of sample storage and handling were not available for the German trials.

The UK use pattern on strawberries allows thiram application of 1.6 kg ai/ha beginning at white bud burst, with repeats at 7-10 day intervals and a PHI of 7 days. In commercial practice there will be no more than 4 or 5 applications in a season. Seven strawberry trials with multiple applications in Belgium were evaluated against the UK use pattern. In four of the trials samples had been taken for analysis just before each application. Generally the number of applications did not seem to influence the level of the residues, although the highest residues were found in two of the trials after 13 and 14 applications. The highest thiram residues (median underlined) in each trial within the range of the UK use pattern resulting from up to 8 applications were 1.4, 1.4, 2.1, 2.1, 2.4, 2.8 and 3.1 mg/kg. The highest residue, 3.1 mg/kg as thiram, is equivalent to 2.0 mg/kg dithiocarbamates as CS_2.

The Meeting estimated a maximum residue level of 5 mg/kg for dithiocarbamates arising from the use of thiram, and an STMR of 2.1 mg/kg for thiram (as thiram), in strawberries.

Residue data on beans, Savoy cabbage, green peas, head lettuce and spinach could not be evaluated because there was no matching GAP or because the number of trials was too small.

The UK registration for thiram on tomatoes allows a spray concentration of 0.32 kg ai/hl and a PHI of 7 days. Four tomato trials in France at 0.22 and 0.32 kg ai/hl and 8 and 10 days PHI produced thiram residues of <0.2, <0.2, 0.95 and 1.1 mg/kg. The Meeting agreed that four trials in one year were inadequate to estimate a maximum residue level for tomatoes.

Information on the fate of thiram during the processing of apples and grapes was made available to the Meeting.

The levels of thiram in apple juice, wet pomace, and dry pomace were 0.29, 1.02 and 3.65 times the level in the apples, suggesting that little of the thiram was lost during the drying process.

In four studies in France field-sprayed grapes were processed to juice, wine and raisins. Thiram residues were not detected (<0.1 mg/kg) in wine by an HPLC method, but were found at 0.12-0.98 mg/kg (as thiram) by a CS_2 evolution method. The thiram residues measured in the grapes by the CS_2 method were also somewhat higher than by the HPLC method. The results obtained by the HPLC method were considered more reliable and were used for estimating processing factors. Since thiram residues were not detected (<0.1 mg/kg) in wine, juice, wet pomace or dry pomace by the HPLC method, the processing factors for grapes to wine for the 4 trials (2 sampling intervals) by the HPLC method were <0.023, <0.033, <0.053, <0.062, <0.071 (median) and <0.083 (3).

In two of the trials thiram residue levels were determined in raisins. In one they were 3.6 and

1.1 times those in the grapes and in the other they were not detected (<0.1 mg/kg). Because of the inconsistency the Meeting could not draw any conclusions about likely residues in raisins.

Monitoring data for dithiocarbamate residues on commodities in trade were provided from The Netherlands, Belgium and Denmark. In most commodities dithiocarbamates were detected in fewer than 15-20% of the samples.

RECOMMENDATIONS

On the basis of the data from supervised trials with thiram the Meeting concluded that the residue levels listed below are suitable for establishing MRLs. Consolidated recommendations for MRLs for dithiocarbamates are listed in the monograph on dithiocarbamates.

Thiram residues, as thiram, may be calculated from dithiocarbamate residues expressed as CS_2 from the relation thiram = $CS_2 \times 1.58$.

Definition of the residue

For compliance with MRLs: The MRLs refer to total dithiocarbamates, determined as CS_2 evolved during acid digestion and expressed as mg CS_2/kg.

For estimation of dietary intake: thiram.

Commodity		Recommended MRL[1], mg/kg		Based on PHI, days	STMR[2], mg/kg	STMR-P[2], mg/kg
CCN	Name	new	current			
FS 0013	Cherries	1		10-15	0.72	
FS 0014	Plums	1		10-14	0.72	
FP 0009	Pome fruits	5	5	7-15	1.9	
FB 0275	Strawberry	5		7	2.1	
	Apple juice					0.55
	Apple pomace, wet					1.9
	Apple pomace, dry					6.93

[1] Expressed as CS_2
[2] Expressed as thiram

FURTHER WORK OR INFORMATION

Desirable

The rates of hydrolysis of thiram at various pH values should be clarified. Full copies of the reports of the studies should be made available for review.

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ZIRAM (DITHIOCARBAMATES, 105)

EXPLANATION

Ziram was originally evaluated in 1965 (toxicology) and 1967 (toxicology and residues) and is included in the dithiocarbamate group of compounds. It is a contact fungicide with protective action and is registered for use on fruit, vegetables, tree nuts and ornamentals in many countries. Ziram, applied to dormant fruit trees is also used to repel hares and rabbits.

The compound was evaluated at the present Meeting within the CCPR periodic review programme.

IDENTITY

ISO common name: ziram

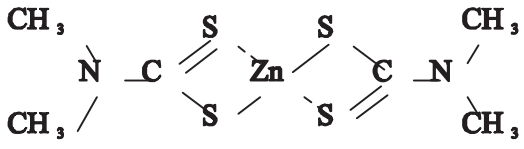
Chemical name

IUPAC: zinc bis(dimethyldithiocarbamate)

CA (T-4)-bis(dimethylcarbamodithioato-*S*,*S*)zinc

CAS No.: 137-30-4
CIPAC No.: 31
EEC No.: 205-288-3

Structural formula:



Ziram

Molecular formula: $C_6H_{12}N_2S_4Zn$

Molecular mass: 305.81

Physical and chemical properties

Pure active ingredient

Vapour pressure:	$<2.5\times10^{-4}$ Pa at 25°C (Lemal, 1987)
Melting point:	246°C
Octanol/water partition coefficient:	log P_{OW} 1.086 (Lemal and Debondue, 1984)
Solubility:	water: 18 mg/l at 20°C water: 10.3 mg/l (Lemal and Debondue, 1984) acetone: 0.8 g/l at 20°C (quoted - Wyss-Benz, 1994) chloroform: 6.4 g/l at 20°C (quoted - Wyss-Benz, 1994) petroleum ether 40-60: 21 mg/l at 20°C (quoted - Wyss-Benz, 1994)
Specific gravity:	1.66 at 25°C
Hydrolysis:	half-life at 25°C (Heasook 1995) 10 min at pH 5 18 hours at pH 7 6.3 days at pH 9
Photolysis:	not photosensitive

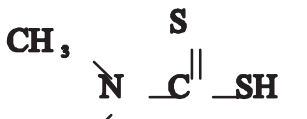
Lemal (1987) measured the vapour pressure of ziram by a gas- saturation method. Nitrogen gas was passed through ziram coated on a support material with a very high surface area and maintained at 25°C, then through a cotton wool dust filter followed by traps containing water. The contents of the absorption traps were acidified with nitric acid and analysed for zinc by atomic absorption spectrophotometry. No zinc was detected in the traps. The vapour pressure of ziram at 25°C did not exceed 2.5×10^{-4} Pa.

Lemal and Debondue (1984) measured the octanol-water partition coefficient of ziram (98% phyto quality) according to OECD Guideline 107 (OECD 1981a). In a series of tests the values of log P_{OW} ranged from 0.954 to 1.196, with a median value of 1.086.

Heasook (1995) measured the rate of hydrolysis of [^{14}C]ziram in sterile aqueous buffer solutions in the dark at pH 5, 7 and 9 and identified the products of hydrolysis.

In the rate experiments the ziram concentration was 2.8 and 2.9 mg/l and the buffer solutions contained 1% acetone to help solubility. Measurements were made for 1 hour, 72 hours and 30 days in the experiments at pH 5, 7 and 9 respectively. In the experiments designed to identify reaction products ziram concentrations were 10 mg/l and acetone was present at 3.9%.

Ziram half-life at 25°C	
pH 5	10.4 mins
pH 7	17.7 hours
pH 9	6.3 days



Dimethyldithiocarbamic acid

The major hydrolysis product at pH 5 and 7 was CS_2, which was also produced at pH 9. In total 11 products were observed but the short-lived ones were eventually converted to CS_2 and could not be isolated and identified. Dimethyldithiocarbamic acid, carbon oxysulfide, isothiocyanic acid or thiocyanic acid and *N*,*N*-dimethylformamide were identified as hydrolysis products at pH 9.

Technical material

Purity:	typically 970 g/kg
Melting range:	240-244°C
Stability:	very stable in test at 54°C for 14 days
Water solubility:	3 mg/l at 20°C (Christiaens and Verberckt, 1987)

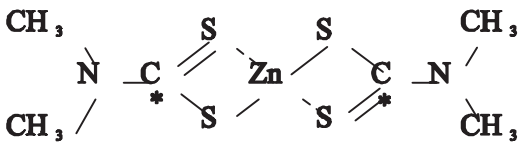
Christiaens and Verberckt (1987) stirred technical ziram with bi-distilled water in flasks at 30°C for 24, 48 or 72 hours. The flasks were then moved to a thermostatted bath at 20°C for 24 hours and shaken from time to time. The supernatant liquid was then centrifuged and the zinc content of the solution was measured by atomic absorption spectrophotometry. Measurements were made on 10 batches of technical ziram, with measured solubilities falling in the range 2.9-3.7 mg ziram/l (mean 3.35 mg/l). The procedure followed the OECD Guideline 105 (OECD, 1981b).

Formulations

WG 900 g/kg, WP 900 g/kg, WG 810 g/kg, WG 760 g/kg and WP 750 g/kg

METABOLISM AND ENVIRONMENTAL FATE

Animal metabolism



^{14}C label

Information was made available to the Meeting on studies of ziram metabolism in lactating goats.

Residues in the tissues, milk and excreta residues were measured in 2 lactating goats each weighing about 40 kg, dosed orally once daily after morning milking for 6 consecutive days by capsule with [*thiocarbonyl*-^{14}C]ziram at 11.9 and 13.2 mg/kg bw/day, equivalent to 300 ppm ziram in the feed (Bodden, 1993). The feed consumption was 1 kg/animal/day of a grain-based milking ration as well as alfalfa grass hay provided *ad libitum*; the mean total daily feed consumption was 1.7 kg. The goats were milked twice daily, producing 1.9-2.7 kg per day. Milk and excreta were collected throughout, and the animals were slaughtered 6 hours after the final dose for tissue collection.

A large part of the administered ^{14}C was not accounted for (54% and 34%). By analogy with the animal metabolism of thiram losses as CS_2 and CO_2 in expired air would be expected, but ^{14}C was not measured in the expired air. More of the ^{14}C dose was in the faeces (42% and 61%) than in the

urine (3%), tissues (0.93% and 0.78%) or milk (0.28% and 0.51%).

The levels of ^{14}C in the milk increased for the first 2 or 3 days of feeding and then reached a plateau (Table 1). The levels of ^{14}C were higher in the liver than in other tissues (Table 2).

Tissues, milk and urine were analysed for dithiocarbamate residues by a colorimetric CS_2 evolution method with a detection limit of 0.5 mg/kg as CS_2. In the liver approximately 10% of the ^{14}C was present as CS_2-liberating compounds (1.1 and 1.2 mg/kg as CS_2). CS_2 was not detected in the other tissues and milk, but the total ^{14}C levels were generally too low to expect its detection. In the urine samples on day 5 from the 2 goats 14% and 27% of the ^{14}C was present as CS_2-liberating compounds.

Nitrosodimethylamine was not detected in the milk, tissues or urine (LOD 1 □g/kg).

The ^{14}C residues in milk, liver, kidney, muscle and fat were not extractable with a chloroform/methanol/water mixture until after protease treatment. The liberated ^{14}C was present in polar water-soluble compounds. ^{14}C was present in lactose and casein isolated from milk and urea isolated from urine, showing that some of the ^{14}C had been incorporated into natural products.

Table 1. Levels of ^{14}C in milk produced by 2 goats dosed daily with [*thiocarbonyl*-^{14}C]ziram equivalent to 300 ppm in the feed (Bodden, 1993).

Day	^{14}C (as ziram), mg/kg milk			
	Goat 1, am milking	Goat 1, pm milking	Goat 2, am milking	Goat 2, pm milking
1		0.44		1.02
2	0.25	0.56	0.41	1.34
3	0.61	0.74	1.85	1.74
4	0.92	0.95	1.54	1.47
5	0.86	0.87	1.74	1.75
6	0.74	0.78	1.66	1.44

Table 2. Levels of ^{14}C in samples from 2 goats dosed daily with [^{14}C]ziram equivalent to 300 ppm in the feed and slaughtered 6 hours after the final dose (Bodden, 1993).

Sample	^{14}C as ziram, mg/kg,	
	Goat 1	Goat 2
Bile	3.1	2.3
Blood	0.87	1.6
Fat (omental)	0.16	0.20
Fat (renal)	0.17	0.18
Kidney	2.9	3.4
Liver	28.0	22.0
Muscle	0.45	0.81

Plant metabolism

Information was made available to the Meeting on ziram metabolism in apples.

Apples and apple leaves on trees were treated once by hand-spraying with [*thiocarbonyl*-^{14}C]ziram at a rate equivalent to 34 kg ai/ha, which is 5 times the label rate (Wyss-Benz, 1994). The apples were 3.5-5.0 cm at the time of treatment. Leaves and apples were sampled for analysis at intervals of 0, 14, 28, 56 and 80 days after treatment, the final occasion at apple maturity. The distribution and levels of ^{14}C in the apples and leaves at the various sampling intervals are shown in Table 3. Residues on the surface of the apples and leaves (found in washings) disappeared more quickly than incorporated residues.

Extracts of apple peel and pulp were analysed by a head-space GLC CS_2 evolution procedure. No CS_2-related residues were detected in the extracts of apple pulp (LOD ~ 0.02 mg/kg as CS_2); they constituted 3.6%, 1.6% and 5.4% of the total ^{14}C residues in apple peel on days 0, 14 and 80, but were not detected in the peel sampled on days 28 and 56. On a whole-apple basis the highest level of CS_2-related residue was 0.016 mg/kg CS_2 in the day 80 sample.

Parent ziram was detected in washings from apples and leaves sampled on days 0, 14 and 28 after treatment. The levels became too low for identification at later samplings. TLC showed that ^{14}C was present in more polar fractions than ziram. [^{14}C]ziram was detected by HPLC in apple pulp (0.014 mg/kg) from apples sampled on the day of treatment, but not at later sampling times. The extractable incorporated ^{14}C at the various sampling times was in polar material. Reference compounds which were possible metabolites did not correspond to any of the radioactive fractions.

Table 3. Distribution and levels of ^{14}C (as ziram) in apples and leaves after treatment with [*thiocarbonyl*-^{14}C]ziram at a rate equivalent to 34 kg ai/ha (Wyss-Benz, 1994).

Days after applic.	Apples						Leaves			
	washings		peel		pulp		washings		washed leaves	
	mg/kg	%[1]	mg/kg	%[1]	mg/kg	%[1]	mg/kg	%[2]	mg/kg	%[2]
0	94	*97*	2.5	*2.6*	0.63	*0.7*	5930	*98*	117	*1.9*
14	2.4	*24*	4.0	*40*	3.5	*36*	93	*45*	114	*55*
28	1.0	*19*	2.3	*41*	2.2	*40*	37	*26*	107	*74*
56	0.15	*4.6*	1.3	*40*	1.8	*56*	3.4	*6.6*	48	*93*
80	0.11	*4.2*	1.2	*46*	1.3	*50*	2.9	*6.1*	45	*94*

[1] Of total ^{14}C in apples
[2] Of total ^{14}C in leaves

METHODS OF RESIDUE ANALYSIS

Analytical methods

The methods rely on acid hydrolysis to release CS_2, which is then measured colorimetrically or by head-space gas chromatography. They are the same as those for other dithiocarbamates.

In the method of the Dutch Method Manual dithiocarbamates are converted to CS_2 by treatment with hydrochloric acid in the presence of stannous chloride (Ministry of Welfare, Health and Cultural Affairs, The Netherlands, 1988). The CS_2 in the head space is determined by GLC with either an ECD or FPD in the sulphur mode. Wyss-Benz (1994) used a similar head-space method to measure levels of CS_2-generating compounds in apples in the metabolism study.

Wyss-Benz (1994) separated [^{14}C]ziram from [^{14}C]CS_2 in the apple metabolism study on a styrene-divinylbenzene column with an EDTA tetrabutylammonium hydroxide aqueous mobile phase. The compounds were detected with a ^{14}C scintillation detector.

Brielbeck and Marx (1994a) described the CS_2 evolution photometric method used in many of the ziram residue trials. Samples were cut up, treated with a stannous chloride hydrochloric acid mixture and heated for one hour. The evolved gases were carried by a stream of nitrogen from the reaction flask through traps to remove H_2S and the CS_2 was then collected in a methanolic potassium hydroxide trap. Measurement of the UV absorbance of the solution at 302 nm gave the concentration of xanthogenate formed. Standard solutions of CS_2 dissolved in methanol were used to prepare a calibration curve. The LOD for peaches was 0.1 mg CS_2/kg. Recoveries were satisfactory in the range 0.25-4 mg ziram /kg, but tended to be elevated at the lowest level tested.

Ohs (1994b) described the CS_2 evolution colorimetric method used in ziram residue trials. CS_2 was released by reacting samples with a stannous chloride-hydrochloric acid mixture. It was collected in a trap and determined colorimetrically after reaction with copper acetate and diethanolamine. The LOD was 0.05 mg CS_2/kg. The method has been used for dithiocarbamate residue analysis for many years. Ziram recoveries were satisfactory in the 0.05-5.0 mg CS_2/kg range. Balluff (1995g) reported essentially the same method for ziram residues in supervised trials, although the LOD in this case was 0.5 mg ziram/kg. A method based on the same chemistry was used in the goat metabolism study to measure the levels of CS_2-generating compounds in milk and tissues (Bodden, 1993).

Holstege and Westberg (1987) described the CS_2 evolution head-space GLC procedure used in the US trials on ziram. The sample was reacted with stannous chloride-hydrochloric acid reagent at 100°C in a sealed reaction flask. An aliquot of the head-space gas was analysed by GLC and compared with ziram standards similarly reacted and injected. Recoveries were satisfactory over the range 0.05-7 mg ziram/kg. The LOD was 0.05 mg ziram/kg. The same method was used in the apple processing study (Meikle, 1992), where recoveries on apples, juice and pomace were found to be satisfactory. Koch (1996) used a similar method in the frozen storage stability study of ferbam and ziram in apples. Satisfactory recoveries were recorded for apples fortified at 0.2 and 2 mg ziram/kg.

Samples in the apple trial in Belgium (66/09) were analysed by a polarographic method, but no summary or details were available (Vervier and Cigot, 1966).

Stability of pesticide residues in stored analytical samples

The Meeting received information on the frozen storage stability of ziram in apples, peaches, almond kernels and almond hulls.

Koch (1996) tested the stability of ziram in macerated apples fortified at 1 mg/kg and stored in head-space bottles at -20°C for 18 weeks. Samples were analysed by a CS_2 evolution GLC head-space method. Ziram was stable under these conditions for the duration of the experiment.

Table 4. Stability of ziram in macerated apples fortified at 1 mg/kg and stored at -20°C (Koch, 1996).

Storage period	Ziram, mg/kg (as ziram)	Method recovery, %, at time of stored sample analysis
0	0.87 0.86	86 88
2 weeks	0.86 0.82	87 81
4 weeks	0.80 0.83	85 83
18 weeks	1.05 1.05	100 107

Bookbinder (1989j) showed that ziram was stable for limited periods in apples, peaches, almond kernels and almond hulls during freezer storage. The sample (4 g ground with dry ice) was fortified with ziram at 2 mg/kg and stored in glass reaction vessels (160 ml) in a freezer at -20±2°C for intervals up to 6 months (Table 5). Samples were analysed by a CS_2 evolution GLC head-space method. Analytical method recoveries were tested on each occasion for each commodity and the ranges were almond kernels 77-91%, hulls 73-93%, apples 80-98%, and peaches 84-104%.

The data suggest that ziram residues in apples and peaches are not sufficiently stable to allow storage of samples in a freezer longer than 3 months.

Table 5. Freezer storage stability of ziram on almond kernels and hulls, apples and peaches fortified at 2 mg/kg and stored at -20±2°C for intervals up to 6 months (Bookbinder, 1989j).

Storage interval	% of original ziram remaining			
	almond kernels	almond hulls	apples	peaches
0 days	76 81	76 79	87 89	96 94
2 weeks	91 86	81 78	90 97	98 100
1 month	91 94	84 82	87 80	97 106
3 months	86 94	74 76	69 69	70 71
4 months			53 45	58 62
6 months	84 91		46 43	56 54

Residue definition

Ziram residues are measured as evolved CS_2 by the methods that are used for the other dithiocarbamates. All samples from the supervised trials on ziram have been analysed by these methods. The Meeting agreed that ziram should be included in the definition of the dithiocarbamate residues: *The MRLs refer to total dithiocarbamates, determined as CS_2 evolved during acid digestion and expressed as mg CS_2/kg.*

For dietary intake purposes and comparison of calculated intakes with the ADI it is preferable to express the residues as ziram because the ADI is expressed in terms of ziram (ziram = $CS_2 \times 2.01$).

USE PATTERN

Ziram is a contact fungicide with protective action. Ziram formulations are registered for use on fruit, vegetables, tree nuts and ornamentals in many countries. Ziram, applied to dormant fruit trees, is also used to repel hares and rabbits.

The Meeting was provided with information on registered uses on fruits, vegetables, tree nuts and cereals Table 6).

Table 6. Registered uses of ziram.

Crop	Country	Form	Application				PHI, days, or application stage
			Method	Max rate per applic., kg ai/ha	Spray conc., kg ai/hl	No.	
Almond	UK	PA	spray	3.2			stage PB
Almond	USA	WG	foliar	6.8	0.72	3	stage 5PF
Apple	Australia	WG	foliar		0.11		7
Apple	Belgium	WG	foliar	1.8	0.080-0.16	6[1]	28
Apple	France	WG	foliar	2.2	0.18	6	14
Apple	Greece	WG	foliar	2.3	0.23	6	15
Apple	Italy	WG	foliar	2.3	0.23	6	10
Apple	Netherlands	WP	foliar	0.75-1.7	0.075-0.11	4	14
Apple	Netherlands	WP	foliar	2.3	0.15	4	14
Apple	UK	PA	spray	3.2			stage PB
Apple	UK	PA	spray	8.0			W
Apple	USA	WG	foliar	5.2-6.8	0.54-0.72		14
Apricot	Greece	WG	foliar	2.3	0.15	4	15
Apricot	USA	WG	foliar	6.8	0.72	4	30
Barley	Germany	SC	spray	3.3	2.2	1	stage 4L
Bean	Greece	WG	foliar		0.20-0.23	3	15
Bean	Italy	WG	foliar		0.15-0.20	1	10
Bean	Portugal	WG	foliar		0.13-0.18	3	14
Berries	Netherlands	WP	foliar	1.9-3.2	1.0-1.6	1	60
Berry fruit except strawberries	Germany	SC	paint	13		2	W
Berry fruit except strawberries	Germany	SC	spray	13	16	2	W
Blackberry	Netherlands	WP	foliar	2.3-2.7	0.23	3	stage[2]
Broccoli	Germany	SC	spray	3.3	0.54-0.81	1	stage PE10
Brussels sprouts	Germany	SC	spray	3.3	0.54-0.81	1	stage PE10
Cabbage, red	Germany	SC	spray	3.3	0.54-0.81	1	stage PE10

[1] Maximum of 4 applications after flowering

[2] Until beginning of flowering.

Crop	Country	Form	Application				PHI, days, or application stage
			Method	Max rate per applic., kg ai/ha	Spray conc., kg ai/hl	No.	
and white							
Cauliflower	Germany	SC	spray	3.3	0.54-0.81	1	stage PE10
Celery	Australia	WP WG	foliar		0.11		7
Celery	Italy	WG	foliar		0.15-0.20	1	10
Cherry	Italy	WG	foliar	2.3	0.23	3	10
Cherry	Netherlands	WP	foliar	2.0	0.13	2	
Cherry	UK	PA	spray	3.2			stage PB
Cherry	UK	PA	spray	8.0			W
Cherry	USA	WG	foliar	3.4-4.3	0.36-0.46	5	7 west
Cherry	USA	WG	foliar	3.4-4.3	0.36-0.46	5	14 east
Chestnut	UK	PA	spray	3.2			stage PB
Chinese cabbage	Germany	SC	spray	3.3	0.54-0.81	1	stage PE10
Citrus fruit	Italy	WG	foliar		0.15-0.20		10
Climbing French beans	Germany	SC	spray	3.3	0.54-0.81	1	stage PE10
Crab apple	UK	PA	spray	8.0			W
Crab apple	UK	PA	spray	3.2			stage PB
Cucumber	Italy	WG	foliar		0.15-0.20	1	10
Cucumber	Portugal	WG	foliar		0.18	2	14
Currant	France	WG	foliar		0.19	2	
Damson	UK	PA	spray	8.0			W
Damson	UK	PA	spray	3.2			stage PB
Dwarf French beans	Germany	SC	spray	3.3	0.54-0.81	1	stage PE10
Fodder beet	Germany	SC	spray	3.3	2.2	1	stage PE14
Garden peas	Germany	SC	spray	3.3	0.54-0.81	1	stage PE10
Garlic	Portugal	WG	foliar		0.18	2	14
Grape	Australia	WG	foliar		0.11		7
Grape	Spain	WG WP	foliar		0.25-0.35	4	7
Hazelnut	UK	PA	spray	8.0			W
Hazelnut	UK	PA	spray	3.2			stage PB
Kale	Germany	SC	spray	3.3	0.54-0.81	1	stage PE10
Kohlrabi	Germany	SC	spray	3.3	0.54-0.81	1	stage PE10
Lettuce	Spain	WG WP	foliar		0.25-0.35	4	7
Maize	Germany	SC	spray	3.3	2.2	1	stage 4L
Medlar	Portugal	WG	foliar		0.13-0.18	3	28
Melon	Greece	WG	foliar		0.20-0.23		
Nectarine	USA	WG	foliar	6.8	0.72		14 east
Nectarine	USA	WG	foliar	6.8	0.72		30 west
Oats	Germany	SC	spray	3.3	2.2	1	stage 4L
Olive	France	WG	foliar		0.19	2	
Olive	Portugal	WG	foliar		0.23	2	
Onion	Italy	WG	foliar		0.15-0.20	1	10
Onion	Portugal	WG	foliar		0.13-0.18	2	14

Crop	Country	Form	Application				PHI, days, or application stage
			Method	Max rate per applic., kg ai/ha	Spray conc., kg ai/hl	No.	
Pea	Italy	WG	foliar		0.15-0.20	1	10
Pea	Portugal	WG	foliar		0.18	2	14
Peach	France	WG	foliar	2.2	0.18	3	14
Peach	Greece	WG	foliar	2.3	0.15	4	15
Peach	Italy	WG	foliar	2.3	0.23	3	10
Peach	Italy	WG	foliar	5.3	0.53	2	stage[1]
Peach	Netherlands	WP	foliar	1.3-1.6	0.13	3	stage[2]
Peach	Netherlands	WP	foliar	2.0	0.13	2	
Peach	USA	WG	foliar	6.8	0.72		14 east
Peach	USA	WG	foliar	6.8	0.72		30 west
Pear	Australia	WG	foliar		0.11		7
Pear	Belgium	WG	foliar	1.8	0.080-0.16	6 Errore. Il segnalibro non è definito.	28
Pear	Greece	WG	foliar	2.3	0.23	6	15
Pear	Italy	WG	foliar	2.3	0.23	6	10
Pear	Netherlands	WP	foliar	2.3	0.15	4	14
Pear	Netherlands	WP	foliar	0.75-1.7	0.075-0.11	4	14
Pear	UK	PA	spray	8.0			W
Pear	UK	PA	spray	3.2			stage PB
Pear	USA	WG	foliar	5.2-6.8	0.54-0.72		5 west
Pear	USA	WG	foliar	5.2-6.8	0.54-0.72		14 east
Pecan	USA	WG	foliar	6.8	0.72	5	55
Plum	Italy	WG	foliar	2.3	0.23	3	10
Plum	Netherlands	WP	foliar	2.0	0.13	2	
Plum	UK	PA	spray	8.0			W
Plum	UK	PA	spray	3.2			stage PB
Pome fruit	Australia	WP	foliar		0.11		7
Pome fruit	France	WG	foliar		0.14-0.19	6	
Pome fruit	Germany	SC	spray	13	16	2	W
Pome fruit	Germany	SC	paint	13		2	W
Pome fruit	Greece	WG	foliar		0.17-0.20	6	15
Pome fruit	Italy	WG	foliar	2.4	0.15-0.20	6	10
Pome fruit	Portugal	WG	foliar		0.13-0.18	5	28
Pome fruit	Spain	WG WP	foliar		0.25-0.35	5	7
Potato	Greece	WG	foliar		0.20-0.23	3	15
Potato	Italy	WG	foliar		0.15-0.20	1	10
Quince	UK	PA	spray	3.2			stage PB
Quince	UK	PA	spray	8.0			W
Raspberry	France	WG	foliar		0.19	2	

[1] Application after harvest and before regrowth.

[2] From bud burst until beginning of flowering.

Crop	Country	Form	Application				PHI, days, or application stage
			Method	Max rate per applic., kg ai/ha	Spray conc., kg ai/hl	No.	
Rye	Germany	SC	spray	3.3	2.2	1	stage 4L
Savoy cabbage	Germany	SC	spray	3.3	0.54-0.81	1	stage PE10
Stone fruit, except apricot	Australia	WG	foliar		0.11		7
Stone fruit, except apricot	Australia	WP	foliar		0.18		7
Stone fruit	France	WG	foliar		0.19	3	
Stone fruit	Germany	SC	spray	13	16	2	W
Stone fruit	Germany	SC	paint	13		2	W
Stone fruit	Greece	WG	foliar		0.15-0.20	4	15
Stone fruit	Italy	WG	foliar	2.4	0.15-0.20	3	10
Stone fruit	Portugal	WG	foliar		0.13-0.18	3	28
Stone fruit	Spain	WG WP	foliar		0.25-0.35	4	7
Strawberries	Spain	WG WP	foliar		0.25-0.27	3	7
Sugar beet	Germany	SC	spray	3.3	2.2	1	stage PE14
Tomato	Greece	WG	foliar		0.20-0.23	g 3	7
Tomato	Greece	WG	foliar		0.20-0.23	3	15
Tomato	Italy	WG	foliar		0.15-0.20	1	10
Tomato	Spain	WG WP	foliar		0.25-0.35	4	7
Tree nuts	Portugal	WG	foliar		0.13-0.18	3	14
Tree nuts	Spain	WG WP	foliar		0.25-0.35	4	7
Triticale	Germany	SC	spray	3.3	2.2	1	stage 4L
Vines	Australia	WP	foliar		0.11		7
Walnut	UK	PA	spray	3.2			stage PB
Walnut	UK	PA	spray	8.0			W
Water melon	Portugal	WG	foliar		0.18	2	14
Watermelon	Greece	WG	foliar		0.20-0.23		
Wheat	Germany	SC	spray	3.3	2.2	1	stage 4L

5PF: Up to 5 weeks after petal fall.
W: Treat woody parts in winter to repel hares and rabbits.
PE10: Up to 10 days post-emergence or after planting.
4L: Up to 4th leaf stage.
PE14: Up to 14 days post-emergence.
PB: Before bud burst.

RESIDUES RESULTING FROM SUPERVISED TRIALS

Residue data from supervised trials on fruit and tree nuts are summarized in Tables 7-16.

Table 7. Apples. Belgium, France, Italy, Netherlands, Spain, USA.
Table 8. Pears. Belgium, France, Italy, Netherlands, Spain, USA.
Table 9. Apricots. USA.
Table 10. Cherries. Spain, USA.
Table 11. Nectarines. Italy, USA.

Table 12. Peaches. France, Italy, Spain, USA.
Table 13. Plums. France, Spain.
Table 14. Almonds. USA.
Table 15. Pecans. USA.
Table 16. Almond hulls. USA.

Where residues were not detected, the results are recorded in the Tables as below 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. Only when residues were detected in control samples are they recorded in the Tables. Dithiocarbamate (CS_2) residues were detected in control samples in 3 apple trials, 4 pear trials, and 1 trial each on nectarines, peaches, plums and almonds (hulls).

The trials were generally fully reported and well documented. Ziram residues had been expressed as CS_2 in some cases and as ziram in others. All the residues in the supervised trials in this monograph are expressed as CS_2 irrespective of the mode of expression in the original. The theoretical factor 0.497 was used to calculate CS_2 levels from ziram levels.

In some trials on tree crops a treated plot within the trial was divided for sampling purposes into sub-plots. In such cases the separate analytical results for each subplot are recorded in the Tables and provide some information on the variation of residue levels which can occur even from within one sprayed plot.

Knapsack air sprayers, mistblowers and wheelbarrow sprayers were use to apply ziram to apple trees in the European trials. Plot sizes ranged from 8 to 20 trees. Freezer storage stability studies suggest that the duration of freezer storage can affect ziram residue levels. No direct information was available on the duration of sample storage in trials B 93-2, B 93-7, B 93-8, B 93-1 or B 93-5. However, an upper limit can be calculated from the dates of sampling and the final date for each study. The maximum periods of sample storage in these five apple trials were in the range 5-9 months. The duration of sample storage before analysis was 2-3 months for trial 304662 and 1½3 months for 94021/01-FPAP. In the 5 ground application trials on apples in the USA ziram was applied by airblast sprayers and backpacks with handgun sprayers. Aerial application in one trial was by helicopter. Plot sizes were 24-120 m^2 for ground application and 2200 m^2 for aerial application. The intervals between harvest and analysis were 1-2 months.

In the pear trials in Europe plot sizes were in the range 8 to 20 trees. In some trials in France and Spain plots were samples as 3 sub-plots. Ziram was applied by knapsack mistblower and wheelbarrow sprayers. Only a summary was available for a Belgian trial in 1966. No direct information was available on the duration of sample storage before analysis in 4 trials (2-37, B 93-12, B 94-1 and B 94-2), but the upper limits could be calculated as before, giving estimated intervals of 5-20 days in trial 2-37, 9.5-11 months in B 93-12, 7-8 months in B 94-12, and 7.5-8.5 months in trial B 94-2. Intervals between harvest and analysis in other trials were 2-3 months in trial 304727, 3½4 months in trial 94021/02, 3½5 months in trial 94021/01, and 6-14 weeks in trials 90A-88, 89B-88 and 90C-88. In the US pear trials ziram was applied by airblast sprayer; plot sizes ranged from 35 m^2 to 1.1 ha.

In the US trials on apricots ziram was applied by aircraft and by airblast sprayer from the ground. Plot sizes were 1100-3700 m^2 for the ground applications and 5 ha for aerial spraying. Samples were stored in a freezer for 3-4 months between harvest and analysis.

Cherry trees were sprayed with a plot knapsack sprayer in the Spanish trials where the plot of

8 trees was treated as 3 sub-plots for sampling. Intervals between harvesting and analysis of the samples were 4-6 months, which may have allowed some decrease in residue levels. In the 5 cherry trials in the USA the trees in 3 (plot sizes 300-1100 m^2) were sprayed with airblast sprayers and in 2 (plot sizes 1900-3000 m^2) from fixed-wing aircraft. The periods of sample storage in a freezer before analysis were mostly 3-4 months, but 2 samples were stored for 5 months.

In the Italian trial on nectarines ziram was applied by a compressed air operated sprayer. The plot size was 9 trees. About 2-4 months elapsed between sampling and analysis. In the US trials on nectarines ziram was applied by airblast sprayer and by aircraft. Plot sizes were 300 and 1100 m^2 for ground application and 2000 m^2 for aerial application. Samples were stored between harvest and analysis for 2-3 months.

Ziram was applied to peach trees in trials in France, Italy and Spain with a backpack airblast sprayer, a wheelbarrow sprayer and a plot knapsack sprayer respectively. Plot sizes ranged from 8 to 14 trees. In the US trials ziram was applied with airblast or back-pack sprayers in 6 trials and by fixed-wing aircraft in the seventh trial. Plot sizes in the US ground spraying trials ranged from 15 to 1100 m^2; the plot size in the aerial spraying trial was 2000 m^2. No direct information was available on the duration of sample storage before analysis in trials UCB 211 and B 93-6, but from the sampling and report dates it could be calculated that samples were stored no longer than 3.5 months in UCB 211 and 11-12 months in B 93-6. Periods of sample storage in other trials were 2-3 months in trial 304646, 2½5 months in trial 94021 and 1½3 months in the US trials. No field report was available for trial UCB 211 so there was no information about the sprayer, plot sizes or whether other dithiocarbamates were used.

In the French and Spanish trials on plums plots of 8 trees were sprayed with knapsack sprayers and plots were sampled as 3 sub-plots. Samples were held in a freezer for 3-3½and 5-6 months before analysis in the French and Spanish trials respectively.

Airblast sprayers were used for ground application in 5 almond trials (plot sizes 40-5000 m^2) in the USA while in the other two trials (plot sizes 1400 and 12000 m^2) ziram was applied by helicopter and fixed-wing aircraft. Samples from 2 trials were stored in a freezer for 3-4 months and those from 5 trials for 6-6½months before analysis.

Pecan trees were treated with ziram with an airblast sprayer or a hand-held wand sprayer. Plot sizes ranged from 80 to 1300 m^2. The duration of freezer storage between harvest and analysis was 119-164 days.

In all trials (Tables 7-16) underlined residues are from treatments according to GAP and are valid fro the estimation of maximum residue levels.

Table 7. Residues of ziram (as CS_2) in apples from foliar application of ziram in supervised trials in Belgium, France, Italy, The Netherlands, Spain and the USA.

Country, year (variety)	Application				PHI, days	Ziram residues as CS_2, mg/kg	Ref.
	Form	kg ai/ha	kg ai/hl	No.			
Belgium, 1966 (Reine des Reinettes)	WP	7×3.6 +4×2.7	7×0.18 +4×0.15	11	0 14 21	0.60 [1] <0.2 0.50	66/09

Country, year (variety)	Application				PHI, days	Ziram residues as CS_2, mg/kg	Ref.
	Form	kg ai/ha	kg ai/hl	No.			
Belgium, 1966 (Ellison's Orange)	WP	7×3.6 +4×2.7	7×0.18 +4×0.15	11	0 2 7 14 21	1.9 [1] 0.50 0.45 <0.2 <0.2	66/09
Belgium, 1993 (Jonagold)	WG	2.4	0.23-0.37	6	0 7 14 21 28 35	0.90 c 0.92 1.2 0.58 0.53 0.61 0.45	UCB B 93-1 93101-RU-010-1
France, 1993 (Wellspur)	WG	2.4	0.28-0.42	6	0 7 14 21 28 35	4.0 2.0 1.4 1.3 0.78 0.83 c0.39	UCB B 93-7 93101-RU-010-4
France, 1993 (Golden Delicious)	WG	2.4	0.24	6	0 6 13 20 27 34	1.5 1.2 0.53 0.21 0.29 0.33	UCB B 93-5 93101-RU-010-3
France, 1993 (Golden Delicious)	WG	2.4	0.28-0.42	6	0 7 14 21 28 35	5.2 5.6 1.9 1.1 2.4 2.0	UCB B 93-8 93101-RU-010-5
Italy, 1993 (Perleberg 3)	WG	2.4	0.16	4 5	7 0 10 21 28 35	2.5 4.5 0.90 0.55 0.36 0.29	304662 RA-2095/93
Netherlands, 1993 (Golden Delicious)	WG	2.4	0.25-0.37	6 dt[2]	0 7 14 21 28 35	2.1 0.71 0.70 0.54 0.44 0.23	UCB B 93-2 93101-RU-010-2
Spain, 1994 (Granny Smith)	WG	2.3	0.23	4	0 21 28	2.6 4.3 4.4 2.0 3.0 1.5 1.9 1.2 1.6	94021/01-FPAP
USA (CA), 1988 (Newton Pippin)	WG	6.8	0.83	4	5 14	3.1 2.3 4.0 4.2 2.4 1.6 2.4 2.3	83A-88

[1] Residues in this trial were measured as ziram by a polarographic method. Residues in the Table are calculated and expressed as CS_2.

[2] Metiram used in May, approximately 4 months before harvest

Country, year (variety)	Application				PHI, days	Ziram residues as CS_2, mg/kg	Ref.
	Form	kg ai/ha	kg ai/hl	No.			
USA (GA), 1988 (Red Delicious)	WG	6.8	1.5	7	14 21	0.38 1.8	82G-88
USA (IL), 1988 (Lodi, Red Delicious, Jonathan)	WG	6.8-7.9	1.45-1.7	7	14 21	1.9 3.5	82F-88
USA (MI), 1988 (Macspur)	WG	6.8	1.45	7	14 21	1.1 0.52 c0.05	82E-88
USA (NY), 1988 (Twenty Ounce)	WP	6.8	2.0	7	14 21	0.98 0.48	82A-88
USA (NY), 1988 (Twenty Ounce)	WG	6.8	2.0	7	14 21	1.2 1.4	82A-88
USA (NY), 1988 (Twenty Ounce)	SC	6.8	2.0	7	14 21	0.97 0.84	82A-88
USA (WA), 1988 (Red Delicious, Red Chief)	WG	6.8	8.1	4 a	5 14	0.36 0.16	83D-88

dt: other dithiocarbamates also used during the growing period.
c: control sample.
a: application by aircraft.

Table 8. Residues of ziram (as CS_2) in pears from foliar application of ziram in supervised trials in Belgium, France, Italy, The Netherlands, Spain and USA. Underlined residues are from treatments according to GAP and are valid data for MRL estimation.

Country, year (variety)	Application				PHI, days	Ziram residues as CS_2, mg/kg	Ref.
	Form	kg ai/ha	kg ai/hl	No.			
Belgium, 1966 (Conference)	WP	7×3.6 +4×2.7	7×0.18 +4×0.15	11	0 2 7 14 21	0.60 [1] 0.45 0.80 0.60 <0.2	66/09
Belgium, 1994 (Conference)	WG	2.3	0.23	4	0 15 20	3.8 c 0.13 0.66 c 0.09 0.54 c 0.14	UCB B 94-1 93101-RU-010-8
France, 1992 (Conference)	WG	2.4	0.20	14	0 15 32	1.3 0.78 0.26	2-37
France, 1992 (Conference)	WG	4.8	0.40	14	0 15 32	2.3 0.78 0.27	2-37

[1] Residues in this trial were measured as ziram, by a polarographic method. Residue data in the table are calculated and expressed as CS_2.

Country, year (variety)	Application				PHI, days	Ziram residues as CS_2, mg/kg	Ref.
	Form	kg ai/ha	kg ai/hl	No.			
France, 1993 (Williams)	WG	2.9[1]	0.54	6	0 6 13 20 27 34	4.9 4.3 3.6 2.4 3.8 1.5	UCB B 93-12 93101-RU-010-7
France, 1994 (Guyot)	WG	2.3	0.23	4	0 7 14 21 28	1.8 3.4 2.5 2.7 1.0 1.2 c 0.2 0.94 0.65 1.5 0.80 1.0 1.6 0.50 1.1 0.40	94021/02-FPBI
Italy, 1993 (William)	WG	2.8	0.16	4 5	7 0 10 21 28 35	1.8 3.3 0.64 0.47 0.33 0.23	304727 RA-2095/93
Netherlands, 1994 (Conference)	WG	2.4	0.23	4	0 14	1.6 c 0.15 0.58 c 0.07	UCB B 94-2 93101-RU-010-9
Spain, 1994 (Ercolini)	WG	2.3	0.23	4	0 7 14 21 28	1.3 1.6 3.3 1.1 1.6 1.9 1.1 0.85 1.1 1.0 0.50 0.85 0.80 1.4 0.50	94021/01-FPBI
USA (CA), 1988 (Bartlett)	WG	6.8	1.8	4 dt	5 14	1.8 1.1 0.86 1.1 0.53 0.67 0.49 0.64 c 0.12 c 0.14	90A-88
USA (NY), 1988 (Bartlett)	WG	6.8	1.8	7	14 21	0.42 0.84 0.94 0.62 0.21 0.59 0.04 0.29	89B-88
USA (WA), 1988 (Red D'Anjou)	WG	6.8	1.45	4	5 14	2.0 1.2	90C-88

dt: other dithiocarbamates also used during the growing period.
c: control sample.
a: application by aircraft.

Table 9. Residues of ziram (as CS_2) in apricots from foliar applications of ziram in supervised trials in the USA.

State, year (variety)	Application				PHI, days	Ziram residues as CS_2, mg/kg	Ref.
	Form	kg ai/ha	kg ai/hl	No.			
CA, 1988 (Modesto)	WG	5.1	1.2	5	30 45 60	0.97 1.4 1.3 1.5 1.0 1.4 1.6 0.90 0.89 0.75 0.78 0.90	84D-88
CA, 1990 (Royal	WG	6.8	1.45	5	30	2.8 4.8	90101

[1] Nominal application rate 2.4 kg ai/ha, measured 2.9 ± 0.2 kg ai/ha.

State, year (variety)	Application				PHI, days	Ziram residues as CS_2, mg/kg	Ref.
	Form	kg ai/ha	kg ai/hl	No.			
Blenheim)					45 60	3.0 1.2 2.4 1.0	
CA, 1990 (Royal Blenheim)	WG	6.8	7.3	5 a	30 45 60	5.3 0.88 0.80 0.55 1.9 0.46 0.37 0.32 1.4 0.34 0.37 0.33	90101
WA, 1990 (Tilton)	WG	6.8	1.45	5	30 45 60	3.7 3.6 3.4 2.8 2.4 2.4 1.5 1.5 1.1	90102

a: application by aircraft.

Table 10. Residues of ziram (as CS_2) in cherries from foliar applications of ziram in supervised trials in Spain and the USA.

CHERRY Country, year (variety)	Application				PHI, days	Ziram residues as CS_2, mg/kg	Ref.
	Form	kg ai/ha	kg ai/hl	No.			
Spain, 1994 (Sunburst)	WG	2.3	0.23	3	0 7 14 21 28	2.9 0.70 3.1 0.60 0.45 0.70 0.35 0.25 0.35 0.35 0.20 0.30 0.20 0.20 0.25	94021/01-FPKI Loc 1
Spain, 1994 (Stark Hardi)	WG	2.3	0.23	3	0 7 14 21 28	2.5 0.65 2.9 0.85 0.85 0.55 0.45 0.65 0.40 0.35 0.30 0.40 0.30 0.30 0.50	94021/01-FPKI Loc 2
USA (CA), 1988 (Bing and Black Tartarian)	WG	5.1	1.1	5	30 45 60	1.1 1.4 0.61 0.95 1.3 1.3 0.95 0.57 0.95 1.2 0.91 0.81	86A-88
USA (CA), 1988 (Bing and Black Tartarian)	WG	5.1	5.5	5 a	30 45 60	0.12 0.22 0.24 0.20 0.15 0.25 0.23 0.11 0.08 0.10 0.08 0.09	86B-88
USA (MI), 1988 (Montmorency)	WG	5.1	1.1	8	7 14 21	1.6 0.79 1.3	85A-88
USA (MI), 1988 (Montmorency)	WG	5.1	5.5	8 a	7 14 21	1.1 0.84 0.80	85B-88
USA (WA), 1988 (Bing)	WG	5.1	1.1	5	30 45 60	1.0 0.53 0.34	86E-88

a: application by aircraft.

Table 11. Residues of ziram (as CS_2) in nectarines from foliar applications of ziram in supervised trials in Italy and the USA.

Country, year (variety)	Application				PHI, days	Ziram residues as CS_2, mg/kg	Ref.
	Form	kg ai/ha	kg ai/hl	No.			
Italy, 1993 (Famtalade)	WG	2.4	0.16	2 3	7 0 7 10 14 21 28 35	0.54 0.71 c 0.12 0.35 <u>0.27</u> 0.23 <u>0.28</u> 0.12 c <0.05 0.11	304719 RA-2082/93
USA (GA), 1988 (Red Gold)	WG	6.8	1.5	10	7 14 21	1.5 <u>1.1</u> 0.91	87G-88
USA (CA), 1988 (Sun Red)	WG	6.8	1.5	7	30 45 60	0.04 0.10 0.07 <u>0.12</u> 0.04 0.05 0.05 0.06 0.03 0.06 0.04 0.03	88C-88
USA (CA), 1988 (Sun Red)	WG	6.8	7.3	a 7	30 45 60	0.07 0.08 <u>0.20</u> 0.18 0.07 0.04 0.11 0.03 0.08 0.12 0.14 0.07	88H-88

c: control sample.
a: application by aircraft.

Table 12. Residues of ziram (as CS_2) in peaches from foliar applications of ziram in supervised trials in France, Italy, Spain and the USA.

Country, year (variety)	Application				PHI, days	Ziram residues as CS_2, mg/kg	Ref.
	Form	kg ai/ha	kg ai/hl	No.			
France, 1993 (Red Haven)	WG	2.4	0.24	3 dt[1]	0 7 14 21 28	fs 2.1 2.5 1.9 fs 2.5 1.3 2.6 fs <u>1.1</u> 0.43 0.45 fs 0.24 0.47 0.55 fs 0.69 0.32 0.70	UCB B 93-6 B93508 93101-RU-010 RU 0593
Italy, 1992 (Red Haven)	WG	2.4	0.16	3	0 7 10 15 20	5.1 2.1 <u>0.96</u> <u>1.8</u> 0.64	UCB 211
Italy, 1993 (Red Haven)	WG	2.4	0.16	2 3	7 0 10 21	1.0 2.0 c 0.06 <u>1.4</u> 1.3	304646 RA-2082/93

[1] Thiram applied in March.

Country, year (variety)	Application				PHI, days	Ziram residues as CS_2, mg/kg	Ref.
	Form	kg ai/ha	kg ai/hl	No.			
					28 35	1.0 c0.14 0.53	
Spain, 1994 (Baby Gold 8)	WG	2.3	0.23	3	0 14 21	fs 3.4 2.0 1.4 fs 0.89 0.40 0.65 fs 0.89 0.94 0.55	94021/01-FPPF Loc1
Spain, 1994 (Maria Serena)	WG	2.3	0.23	3	0 14 21	fs 2.5 1.7 2.1 fs 1.8 0.80 0.80 fs 1.4 0.70 0.75	94021/01-FPPF Loc2
USA (CA), 1988 (Fairtimes)	WG	6.8	1.5	7	30 45 60	<0.03 (2) 0.05 0.03 0.63 0.34 0.41 0.58 0.08 0.77 0.67 0.12	88B-88
USA (CA), 1988 (Ryanson)	WG	6.8	1.45	8	30 45 60	0.49 0.44 0.42 0.50 0.11 0.22 0.27 0.26 0.12 0.34 0.29 0.31	88A-88
USA (CA), 1988 (Fairtimes)	WG	6.8	7.7	7 a	30 45 60	0.07 0.04 0.03 0.03 0.07 0.03 0.08 0.08 0.03 0.03 0.03 0.04	88G-88
USA (MI), 1988 (Harbelle)	WG	6.7	0.40	10	7 14	3.4 2.3	87C-88
USA (NJ), 1988 (Red Haven)	WG	6.8	1.2	10	7 14 21	<0.03 0.43 0.34	87B-88
USA (SC), 1988 (Crest Haven)	SC	6.8	1.45	10	7 14 21	12 5.3 4.8	87A-88
USA (WA), 1988 (Delp Hale)	WG	6.8	1.45	9	30 45 60	0.72 0.58 0.67	88F-88

dt: other dithiocarbamates also used during the growing period.
fs: fruit without stone.
c: control sample.
a: application by aircraft.

Table 13. Residues of ziram (as CS_2) in plums from foliar applications of ziram in supervised trials in France and Spain.

Country, year (variety)	Application				PHI, days	Ziram residues as CS_2, mg/kg	Ref.
	Form	kg ai/ha	kg ai/hl	No.			
France, 1994 (Ente 707)	WG	2.3	0.23	3	0 7 14 21	0.65 1.8 0.70 0.80 0.45 1.7 1.7 0.25 1.0 c0.15 0.50 0.30 0.45	94021/02-FPPL
Spain, 1994 (Red Beaut)	WG	2.3	0.23	3	0 8 14	0.65 0.20 0.30 <0.2 0.2 <0.2 2.5 <0.2 0.3	94021/01-FPPL

Country, year (variety)	Application				PHI, days	Ziram residues as CS_2, mg/kg	Ref.
	Form	kg ai/ha	kg ai/hl	No.			
					21 28	0.40 0.2 <0.2 <0.2 <0.2 <0.2	

c: control sample.

Table 14. Residues of ziram (as CS_2) in almond kernels from foliar applications of ziram on almond orchards in supervised trials in the USA.

State, year (variety)	Application				PHI, days	Ziram residues as CS_2, mg/kg	Ref.
	Form	kg ai/ha	kg ai/hl	No.			
CA), 1988 (Nonpareil)	WG	6.8	1.45	4 3	149 184	<0.02 <0.02	80A-88
CA), 1988 (Nonpareil)	SC	6.8	1.45	4 3	149 184	<0.02 <0.02	80A-88
CA), 1988 (Nonpareil)	WP	6.8	1.45	4 3	149 184	<0.02 <0.02	80A-88
CA), 1988 (Monterey)	SC	6.8	1.45	4 3	167 205	0.03 (3) <0.02 <0.02 (4)	80B-88
CA), 1988 (Monterey)	WG	6.8	1.45	4 3	167 205	<0.02 (4) <0.02 (4)	80B-88
CA), 1988 (Monterey)	WP	6.8	1.45	4 3	167 205	<0.02 (4) <0.02 (4)	80B-88
CA), 1988 (Nonpareil)	WG	6.8	1.45	4 3	143 178	<0.02 (4) <0.02 (4)	80C-88
CA), 1988 (Nonpareil)	SC	6.8	1.45	4 3	143 178	<0.02 (4) <0.02 (4)	80C-88
CA), 1988 (Nonpareil)	WP	6.8	1.45	4 3	143 178	<0.02 (4) <0.02 (4)	80C-88
CA), 1988 (Nonpareil)	SC	8.2	8.7	a 4 a 3	142 177	<0.02 (4) <0.02 (4)	80E-88
CA), 1988 (Monterey and Carmel)	WG	6.8	7.3	a 4	176	<0.02 (4)	80F-88
CA), 1988 (Nonpareil)	WG	6.8	2.4	4 3	156 191	<0.02 (4) <0.02 (4)	80G-88
CA), 1988 (Nonpareil)	WG	6.8	1.4-1.8	4	139	<0.02 (4)	80H-88

a: aerial application

Table 15. Residues of ziram (as CS_2) in pecan kernels from foliar applications of ziram in the USA.

State, year (variety)	Application				PHI, days	Ziram residues as CS_2, mg/kg	Ref.
	Form	kg ai/ha	kg ai/hl	No.			
GA, 1988 (Stuart)	SC WG WP	6.8 6.8 6.8	0.77 0.77 0.77	8 8 8	51 51 51	<0.02 <0.02 <0.02	91A-88
TX, 1988 (Wichita)	SC WG	6.8 6.8	0.40 0.40	8 8	57 57	0.03 <0.02	91B-88
OK, 1988 (indigenous)	SC	6.8	1.0	8	63	<0.02	91D-88
NM, 1988 (Western Schley)	SC	5.6	0.30	8	83	<0.02	91E-88

Table 16. Residues of ziram (as CS_2) in almond hulls from foliar applications of ziram on almond orchards in supervised trials in the USA.

State, year (variety)	Application				PHI, days	Ziram residues as CS_2, mg/kg	Ref.
	Form	kg ai/ha	kg ai/hl	No.			
CA, 1988 (Nonpareil)	WG	6.8	1.45	4 3	149 184	2.8 0.24	80A-88
CA, 1988 (Nonpareil)	SC	6.8	1.45	4 3	149 184	4.6 0.60	80A-88
CA, 1988 (Nonpareil)	WP	6.8	1.45	4 3	149 184	2.0 0.43	80A-88
CA, 1988 (Monterey)	SC	6.8	1.45	4 3	167 205	8.8 6.2 6.0 4.9 0.28 0.60 0.35 0.34	80B-88
CA, 1988 (Monterey)	WG	6.8	1.45	4 3	167 205	4.9 4.3 5.3 4.2 0.17 0.18 0.19 0.17	80B-88
CA, 1988 (Monterey)	WP	6.8	1.45	4	167	5.0 5.0 5.8 3.6	80B-88
CA, 1988 (Nonpareil)	WG	6.8	1.45	4 3	143 178	5.4 6.1 5.7 5.0 0.47 0.33 0.23 0.25	80C-88
CA, 1988 (Nonpareil)	SC	6.8	1.45	4 3	143 178	8.4 8.4 5.0 9.3 0.71 0.71 1.1 0.64	80C-88
CA, 1988 (Nonpareil)	WP	6.8	1.45	4 3	143 178	6.4 6.7 6.1 5.3 0.42 0.52 0.42 0.86	80C-88
CA, 1988 (Nonpareil)	SC	8.2	8.7	4 a 3 a	142 177	3.0 3.5 4.5 3.1 1.3 1.3 1.1 1.3	80E-88
CA, 1988 (Monterey and Carmel)	WG	6.8	7.3	4 a 3 a	176 211	1.8 3.0 0.85 1.5 0.20 0.18 0 14 0.18	80F-88
CA, 1988 (Nonpareil)	WG	6.8	2.4	4 3	156 191	1.3 1.0 0.92 0.86 0.31 0.46 0.20 0.21 c 0.12	80G-88
CA, 1988 (Nonpareil)	WG	6.8	1.4-1.8	4 3	139 174	0.02 0.62 6.9 6.0 0.38 0.40 0.37 0.34	80H-88

c: control sample.

a: aerial application

FATE OF RESIDUES IN STORAGE AND PROCESSING

In storage

No information was available.

In processing

Information was made available on the fate of ziram residues during the processing of apples.

In a 1989 US processing study Monroe apples in an orchard in New York were treated 7 times by airblast spray with ziram at an exaggerated rate of 34 kg ai/ha (9.1 kg ai/hl) and harvested 5 days after the final treatment for processing (Meikle, 1992). Lots of approximately 60 kg apples were processed into juice and pomace. The processing equipment included a hammer-mill, a crusher-stemmer, a press and a drier. Apples and processed commodities were analysed for ziram by a CS_2 evolution method.

Commodity	Ziram (as CS_2), mg/kg
Apple, whole fruit	8.4
Apple juice	0.79 0.84
Wet pomace	10.8 11.7
Dry pomace	15.1 15.4

The process did not include washing the fruit. Juice had no additional filtering after the press cloth. Wet pomace was dried at 77-88°C for 1-4 hours to achieve a moisture level below 10% (dry pomace).

Ziram residue levels in the juice were about 10% of those in the apples, while levels in the wet pomace were slightly higher than in the apples. Levels in dry pomace were 30-40% higher than in the wet pomace. Much of the ziram residues must be lost during the drying process because dry pomace is 20-25% by weight of wet pomace.

Residues in the edible portion of food commodities

Ziram residue levels in apple juice were about 10% of the levels in the apples.

RESIDUES IN FOOD OR AT CONSUMPTION

Monitoring data on dithiocarbamates are included in the monograph on thiram.

NATIONAL MAXIMUM RESIDUE LIMITS

National MRLs for dithiocarbamates are included in the monograph on thiram.

APPRAISAL

Ziram was originally evaluated in 1965 (toxicology) and 1967 (toxicology and residues) and is included in the dithiocarbamate group of compounds. It was evaluated at the present Meeting within the CCPR periodic review programme.

Ziram is a dithiocarbamate contact fungicide with protective action and is registered for use on fruit, vegetables, tree nuts and ornamentals in many countries. Ziram, applied to dormant fruit trees, is also used to repel hares and rabbits.

The Meeting received information on the metabolism of ziram in goats and apples, methods of residue analysis, the stability of residues in stored analytical samples, approved use patterns, supervised residue trials and the fate of residues during the processing of apples.

Ziram is hydrolysed very quickly at pH 5 (half-life 10.4 minutes) and more slowly at pH 7 (half-life 17.7 hours) and pH 9 (half-life 6.3 days). The major hydrolysis product at pH 5 and 7 is CS_2. At pH 9 CS_2 was produced, but dimethyldithiocarbamic acid, carbon oxysulphide, isothiocyanic or thiocyanic acid and *N*,*N*-dimethylformamide were also identified.

When two lactating goats were dosed for 6 days with [*thiocarbonyl*-^{14}C]ziram at a rate equivalent to 300 ppm ziram in the feed the levels of ^{14}C in milk increased for the first 2 or 3 days of feeding and then reached a plateau. A large part of the administered ^{14}C was not accounted for (39% and 58%). By analogy with the animal metabolism of thiram losses as CS_2 and CO_2 in expired air would be expected, but ^{14}C was not measured in the expired air. More of the ^{14}C dose was in the faeces (42% and 61%) than in the urine (3%), tissues (0.93% and 0.78%) or milk (0.28% and 0.51%).

The levels of ^{14}C were higher in the liver than in other tissues and approximately 10% of the ^{14}C in the liver was liberated as CS_2 when treated with hot acid. Liberated CS_2 was not detected in the other tissues and milk but the total ^{14}C levels were generally too low to expect its detection. The ^{14}C residues in the milk and tissues were not extractable until after protease treatment. The liberated ^{14}C was present in polar water-soluble compounds. Since lactose and casein containing ^{14}C were isolated from milk some ^{14}C had evidently been incorporated into natural products.

Because of the fairly rapid hydrolysis of ziram the parent compound would not be expected to occur in animal tissues, which generally agrees with the findings of the goat metabolism study. Dimethyldithiocarbamic acid, identified as a hydrolysis product of ziram at pH 9, was the major intermediate in the metabolism of thiram. It is probably also a hydrolysis product of ziram at lower pHs, but would disappear too quickly for identification. The presence of dimethyldithiocarbamic acid as a hydrolysis product suggests that the metabolites of ziram are likely to be the same as those of thiram.

When apples and apple leaves on a tree were treated with [*thiocarbonyl*-^{14}C]ziram the parent compound was detected in washings from the apples and leaves 0, 14 and 28 days after treatment. Residues on the surface disappeared more quickly than incorporated residues. The parent compound was detected in the pulp from apples sampled on the day of treatment, but not at later sampling times. The extractable incorporated ^{14}C was in polar material and was not identified because the reference compounds which had been chosen as possible metabolites did not correspond to the ^{14}C fractions.

Extracts of apple peel and pulp were analysed by a CS_2 evolution head-space GLC procedure. CS_2-related residues were not detected in the extracts of pulp, but constituted up to 5% of the total ^{14}C

residues in the peel. In whole-apples the highest level of CS_2-related residue was 0.016 mg/kg as CS_2.

Ziram residues are essentially on the surface. Most of the residue which becomes incorporated into tissue no longer contains the CS_2 structure. As in animal metabolism, because dimethyldithiocarbamic acid is a hydrolysis product of ziram it is quite likely that the plant metabolites of ziram are the same as those of thiram.

Studies of environmental fate were not provided for review by the FAO Panel, but the Meeting was informed that studies were available and had been supplied to the Environmental Core Assessment Group. They would be supplied for future evaluation by the FAO Panel. The Meeting agreed that the recommended MRLs would be temporary pending the review of data on environmental fate by the FAO panel.

The analytical methods for ziram residues rely on acid hydrolysis to release CS_2, which is then measured colorimetrically or by head-space gas chromatography. The methods are the same as those for other dithiocarbamates (see also the monograph on thiram). The methods used in the trials gave satisfactory recoveries and LODs were about 0.05-0.1 mg/kg.

The Meeting received information on the frozen storage stability of ziram residues in apples, peaches, almond kernels and almond hulls.

Ziram in macerated apples fortified at 1 mg/kg and stored at -20°C was stable for the duration of the test (18 weeks).

Ziram in macerated apples and peaches fortified at 2 mg/kg stored at -20±2°C was of marginal stability (about 70% remaining) after 3 months storage and had decreased to approximately half the fortification level after 6 months. Ziram residues were stable in almond kernels and almond hulls at -20±2°C for the intervals tested, 6 months and 3 months respectively.

The Meeting was informed that storage stability studies are in progress on ziram residues in whole peaches, whole apples, almond kernels and almond hulls. Summary data for 12 months storage generally showed adequate stability but the Meeting agreed to await full reports of the studies.

Ziram residues are measured as evolved CS_2 by the methods that are used for the other dithiocarbamates. All samples from supervised trials on ziram have been analysed by these methods. The Meeting agreed that ziram should be included in the definition of the dithiocarbamate residues.

For estimates of dietary intake and comparison of calculated intakes with the ADI it is preferable to express the residues as ziram because the ADI is expressed in terms of ziram.

Because the residues in the supervised trials are expressed as CS_2 it is convenient to discuss them in this form and convert them to a ziram basis (ziram = $CS_2 \times 2.01$) for recommendations for STMRs.

The Meeting received data from supervised residue trials on, apples (Belgium, France, Italy, The Netherlands, Spain, USA), pears (Belgium, France, Italy, The Netherlands, Spain, USA), apricots (USA), cherries (Spain, USA), nectarines (Italy, USA), peaches (France, Italy, Spain, USA), plums (France, Spain), almonds (USA), pecans (USA) and almond hulls (USA).

The residues are expressed as CS_2 in the following discussion. In some trials other dithiocarbamates had been used on the crop; if this occurred during the growing season the trials were

not considered valid for ziram.

Because the frozen storage stability studies had shown that ziram residues in fruit had decreased to about 70% of the initial level after 3 months freezer storage, trials on fruit were considered invalid where no information was provided on the storage conditions or duration or where the duration of storage was excessive. Residues would be expected to be more stable when samples were stored as whole unchopped fruit, as was demonstrated by the storage stability of thiram on whole plums. The stability of ziram and thiram in frozen storage should be comparable.

In France ziram is registered for use on apples at 2.2 kg ai/ha with a PHI of 14 days. The residues in apples in a trial in Belgium where ziram was used at 2.4 kg ai/ha were 0.58 and 0.61 mg/kg as CS_2 14 and 28 days after the final application respectively. In 3 trials in France also close to the registered use pattern dithiocarbamate residues as CS_2 were 0.53, 1.4 and 2.4 mg/kg. The residue as CS_2 was 0.70 mg/kg in an apple trial in The Netherlands which closely followed French GAP.

In Italy ziram is registered for use on apples at 2.3 kg ai/ha (0.23 kg ai/hl) with a PHI of 10 days. In a trial in Italy at 2.4 kg ai/ha the residues was 0.90 mg/kg as CS_2 10 days after the final application.

Ziram is registered in Spain for use on pome fruit at a spray concentration of 0.25-0.35 kg ai/hl with a PHI of 7 days. Residues of 3.0 mg/kg as CS_2 were recorded in apples in a Spanish trial 21 days after application at 0.23 kg ai/ha. Although this residues was obtained from an application approximating GAP and residues appeared to be decreasing quite slowly 21 days is too remote from 7 days to be considered for evaluation.

US GAP on apples permits ziram applications at 5.2-6.8 kg ai/ha with harvest 14 days after the final application. In 8 US trials according to this use pattern residues as CS_2 in apples were 0.16, 0.97, 0.98, 1.1, 1.4, 1.8, 2.4 and 3.5 mg/kg. In 3 of the trials the residues at 21 days were higher than at 14 days and are therefore included in the evaluation.

In summary, ziram residues as CS_2 in apples from 14 trials in rank order (median underlined) were 0.16, 0.53, 0.61, 0.70, 0.90, 0.97, 0.98, 1.1, 1.4, 1.4, 1.8, 2.4, 2.4 and 3.5 mg/kg.

GAP for pears in The Netherlands permits the application of 2.3 kg ai/ha (0.15 kg ai/hl) of a WP formulation and harvest 14 days after the last of 4 applications. Trials with WG formulations in Belgium, France and The Netherlands were evaluated according to this use pattern because in this situation residues from the use of WP and WG formulations would be expected to be similar. Ziram residues as CS_2 in 1 Belgian trial, 2 French trials and 1 trial in the Netherlands approximating Netherlands GAP were 0.66, 3.8, 1.6 and 0.58 mg/kg. In the French trials residues from longer PHIs were higher and replaced the residue at the GAP PHI.

In Italy ziram is registered for use on pears at 2.3 kg ai/ha (0.23 kg ai/hl) with a PHI of 10 days. In a trial in Italy at 2.8 kg ai/ha the ziram residue was 0.64 mg/kg as CS_2 10 days after the final application.

Ziram is registered in Spain for use on pome fruit at a spray concentration of 0.25-0.35 kg ai/hl with a PHI of 7 days. Ziram residues of 1.9, 1.6 and 1.1 mg/kg as CS_2 were recorded in pears in 3 sub-plots of a Spanish trial where fruit were harvested 7 days after the final ziram application at a spray concentration of 0.23 kg ai/hl, which is at the lower end of the acceptable range for evaluation purposes.

US GAP on pears allows ziram application at 5.2-6.8 kg ai/ha with a PHI of 5 days in the west and 14 days in the east. Residues as CS_2 in 2 US trials according to these 2 use patterns were 0.94 and 2.0 mg/kg. A third US trial could not be evaluated because other dithiocarbamates had been used during the growing period.

In summary, valid results on pears were available from 8 trials with residues as CS_2 or 0.58, 0.64, 0.66, 0.94, 1.6, 1.9, 2.0 and 3.8 mg/kg.

The Meeting concluded that the residues in apples and pears appeared to be from similar populations and could be combined to represent pome fruit. Ziram residues as CS_2 in pome fruit in rank order (median underlined) were 0.16, 0.53, 0.58, 0.61, 0.64, 0.66, 0.70, 0.90, 0.94, 0.97, 0.98, 1.1, 1.4, 1.4, 1.6, 1.8, 1.9, 2.0, 2.4, 2.4, 3.5 and 3.8 mg/kg.

The Meeting estimated a maximum residue level of 5 mg/kg as CS_2 for dithiocarbamates in pome fruits arising from the use of ziram and noted that this level was consistent with the MRL already established. The Meeting estimated an STMR for ziram on pome fruit of 1.04 mg/kg as CS_2, equivalent to 2.1 mg/kg as ziram.

US GAP on apricots permits application of ziram at 6.8 kg ai/ha and harvest of the fruit 30 days after the final application. The maximum ziram residues as CS_2 with GAP on apricots from 3 US trials at 6.8 kg ai/ha and 1 US trial at 5.1 kg ai/ha were 1.6, 3.7, 4.8 and 5.3 mg/kg.

The stone fruit registration in Spain permits 4 ziram applications at spray concentrations of 0.25-0.35 kg ai/hl with a 7-day PHI. In two Spanish trials on cherries where ziram was applied at 0.23 kg ai/hl and fruit were harvested 7 days after the last of 3 applications the residues as CS_2 in the three sub-plots of each trial were 0.45, 0.60 and 0.70 mg/kg and 0.55, 0.85 and 0.85 mg/kg.

US GAP for cherries permits ziram application at 3.4-4.3 kg ai/ha with 7- and 14-day PHIs in the west and east respectively. The application rate in the US trials was 5.1 kg ai/ha, but data from 3 trials could not be used because the shortest interval between the final application and harvest was 30 days, which is too remote from the registered 7 days. In the remaining 2 trials the residues as CS_2 14 days after the final application were 0.84 and 0.79 mg/kg (replaced for evaluation by residues at 14 days of 1.3 mg/kg).

In summary, the residues as CS_2 from the 4 valid trials on cherries were 0.70, 0.84, 0.85, and 1.3 mg/kg.

The stone fruit registration in Italy permits ziram applications at 2.4 kg ai/ha (spray concentrations of 0.15-0.20 kg ai/hl) with a 10-day PHI. In an Italian trial on nectarines ziram was applied at 2.4 kg ai/ha. The residues as CS_2 were 0.27 mg/kg after 10 days and 0.28 mg/kg after 21 days.

The US registration for nectarines permits ziram application at 6.8 kg ai/ha with harvest 14 and 30 days after the final application in the east and west respectively. The residues as CS_2 in 2 nectarine trials from the west and 1 from the east according to these use patterns were 0.12, 0.20 and 1.1 mg/kg.

In summary, residues as CS_2 from the 4 valid trials on nectarines were 0.12, 0.20, 0.28 and 1.1 mg/kg.

In 2 Italian trials on peaches according to GAP the residues as CS_2 were 1.4 and 0.96 mg/kg

after 10 days. In the second trial the residue at 15 days was 1.8 mg/kg and this higher residue was used for evaluation.

Ziram may be applied 3 times at 2.2 kg ai/ha to peaches with harvest 14 days after the final application according to the registration in France. One trial in France and 2 in Spain conformed to this use pattern and the maximum residues as CS_2 in each trial were 1.1, 0.94 and 1.8 mg/kg.

US GAP for peaches is the same as for nectarines. The residues as CS_2 in 3 peach trials according to the use pattern in the east were 0.43, 2.3 and 5.3 mg/kg. The highest residues as CS_2 in each of 4 trials according to the use pattern in the west were 0.08, 0.50, 0.72 and 0.77 mg/kg. In one of these trials the residue in a 60-day sample, 0.77 mg/kg, was much higher than the residues in samples from 30 days, 0.03 and 0.05 mg/kg.

In summary, the residues as CS_2 from the 12 valid trials on peaches (median underlined) were 0.08, 0.43, 0.50, 0.72, 0.77, 0.94, 1.1, 1.4, 1.8, 1.8, 2.3 and 5.3 mg/kg

In a Spanish trial on plums according to GAP for stone fruit where ziram was applied at 0.23 kg ai/hl and fruit were harvested 8 days and after the final application ziram residues as CS_2 in the three sub-plots of the trial were <0.2, <0.2 and 0.2 mg/kg. Residues in plums harvested on day 14 were <0.2, 0.3 and 2.5 mg/kg. The 2.5 mg/kg value seemed inconsistent with the other results, but the analysis had been repeated. A French trial where ziram was applied to plums at a spray concentration of 0.23 kg ai/hl and fruit were harvested 7 days after the final application was evaluated against Spanish GAP. The residues as CS_2 in the three sub-plots were 0.45, 0.80 and 1.7 mg/kg. In summary, the residues as CS_2 on plums from two valid trials were 1.7 and 2.5 mg/kg.

The use patterns for ziram on the different stone fruits within a country are generally the same and the Meeting concluded that the data from the trials on stone fruits could be combined although the residues on apricots tended to be higher than on the other fruits. The residues from the valid trials were 3.7, 4.8, 5.3 mg/kg on apricots, 0.70, 0.84, 0.85, 1.3 mg/kg on cherries, 0.12, 0.20, 0.28, 1.1 mg/kg on nectarines, 0.08, 0.43, 0.50, 0.72, 0.77, 0.94, 1.1, 1.4, 1.8, 1.8, 2.3, 5.3 mg/kg on peaches and 1.7, 2.5 mg/kg on plums. The residues as CS_2 in 26 trials on stone fruit in rank order (median underlined) were 0.08, 0.12, 0.20, 0.28, 0.43, 0.50, 0.70, 0.72, 0.77, 0.84, 0.85, 0.94, 1.1, 1.1, 1.3, 1.4, 1.6, 1.7, 1.8, 1.8, 2.3, 2.5, 3.7, 4.8, 5.3 and 5.3 mg/kg .

The Meeting estimated a maximum residue level of 7 mg/kg (as CS_2) for dithiocarbamates in stone fruit arising from the use of ziram and an STMR of 2.2 mg/kg as ziram (1.1 mg/kg as CS_2) for ziram in stone fruit.

Ziram is registered for use on almonds in the USA with an application rate of 6.8 kg ai/ha and with the last of 3 applications to be completed by 5 weeks after petal fall. The pre-harvest intervals in the supervised trials were in the range 139-167 days and the conditions were taken to comply with US GAP. In 12 of the 13 trials no residues were detected (<0.02 mg/kg as CS_2) while in one trial the residue was 0.03 mg/kg. The median residue for the 13 trials was <0.02 mg/kg.

The Meeting estimated a maximum residue level of 0.1* mg/kg CS_2 in almonds from the use of ziram. This is a practical LOD which can be achieved by regulatory methods. The Meeting estimated an STMR of 0.04 mg/kg as ziram (0.02 mg/kg as CS_2) for ziram in almonds.

The residues as CS_2 on the almond hulls from the 13 almond trials in rank order (median underlined) were 1.3, 2.0, 2.8, 3.0, 4.5, 4.6, 5.3, 5.8, 6.1, 6.7, 6.9, 8.8 and 9.3 mg/kg. No information was available on the moisture content of the almond hulls; residue levels in animal feed materials

should be expressed on a dry-weight basis.

The Meeting estimated a maximum residue level of 10 mg/kg for dithiocarbamates as CS_2 on almond hulls arising from the use of ziram and an STMR of 10.6 mg/kg as ziram (5.3 mg/kg as CS_2) for ziram on almond hulls.

Ziram is registered for use on pecans in the USA with an application rate of 6.8 kg ai/ha and a PHI of 55 days. In the 7 trials the interval between the final application and harvest was 51-83 days. In 6 of the 7 trials no residues were detected (<0.02 mg/kg as CS_2) while in one trial the residue was 0.03 mg/kg. The median residue for the 7 trials was <0.02 mg/kg.

The Meeting estimated a maximum residue level of 0.1* mg/kg CS_2 in pecans from applications of ziram, taking into account MRLs recommended by the 1993 JMPR for dithiocarbamates in almonds and peanut at a practical LOD of 0.1* mg/kg, and an STMR of 0.04 mg/kg as ziram (0.02 mg/kg as CS_2) for ziram in pecans.

In a processing study on apples sprayed with ziram at an exaggerated rate (34 kg ai/ha) ziram residue levels in the juice were about 10% of those in the apples while the residues in the wet pomace were 1.34 times those in the apples. The levels of ziram in dry pomace were 30-40% higher than in wet pomace which suggests loss of ziram during the drying process because dry pomace is only 20-25% by weight of wet pomace. The processing factors from apples to juice, wet pomace, and dry pomace were 0.097, 1.34 and 1.82 respectively. The process did not include a washing step. Because ziram residues are on the apple surface a commercial process with an initial washing and cleaning step would be expected to reduce the residue.

The supervised trials median residues (STMR-Ps) for the processed apple commodities, calculated from the processing factors and the STMR for pome fruit (2.1 mg/kg) are apple juice 0.204 mg/kg, wet apple pomace 2.81 mg/kg, dry apple pomace 3.82 mg/kg, all expressed as ziram.

Monitoring data for dithiocarbamate residues in commodities in trade are included in the monograph on thiram.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting estimated the maximum residue levels listed below, which are recommended for use as TMRLs. The estimates are temporary pending the review of data on environmental fate. Consolidated recommendations for MRLs for dithiocarbamates are listed in the monograph on dithiocarbamates.

Definition of the residue

For compliance with MRLs: The MRLs refer to total dithiocarbamates, determined as CS_2 evolved during acid digestion and expressed as mg CS_2/kg.

For estimation of dietary intake: ziram.

Residues expressed as ziram may be calculated from residues expressed as CS_2 from the relation ziram = $CS_2 \times 2.01$.

Commodity		Recommended MRL[1], mg/kg		Based on PHI, days	STMR[2], mg/kg	STMR-P[2], mg/kg
CCN	Name	New	Current			
AM 0660	Almond hulls	10 T	20		10.6	
TN 0660	Almonds	0.1* T	0.1*		0.04	
TN 0672	Pecan	0.1* T		51-83	0.04	
FP 0009	Pome fruits	5 T	5	5-15	2.1	
FS 0012	Stone fruits	7 T		7-30	2.2	
	Apple juice					0.204
	Apple pomace, wet					2.81
	Apple pomace, dry					3.82

[1] Expressed as CS_2.
[2] Expressed as ziram.

FURTHER WORK OR INFORMATION

Required (by 1997)

Information on the environmental fate of ziram in soil and in water/sediment systems.

Desirable

1. Information on the effect of washing on ziram residues on fruits.

2. Final reports of freezer storage stability studies now in progress on peaches, apples and almonds.

3. Information on attempts to develop specific methods of analysis for ziram, whether successful or not.

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ANNEX I

ACCEPTABLE DAILY INTAKES, RESIDUE LIMITS AND SUPERVISED TRIALS MEDIAN RESIDUES PROPOSED AT THE 1996 MEETING

The Table of recommendations includes maximum Acceptable Daily Intakes (ADIs) and Maximum Residue Limits (MRLs). It should be noted that MRLs include draft MRLs and Codex MRLs (CXLs). The MRLs recommended by the JMPR on the basis of its estimates of maximum residue levels enter the Codex procedure as draft MRLs. They become Codex MRLs when they have passed through the procedure and have been adopted by the Codex Alimentarius Commission.

In general, the recommended MRLs listed for compounds which have been reviewed previously are additional to, or amend, those recorded in the reports of earlier Meetings. For compounds re-evaluated in the CCPR periodic review programme however, both new and previous recommendations are listed because such re-evaluations are regarded as replacing the original evaluation rather than supplementing it.

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 response to recommendations of a Joint FAO/WHO Consultation on Guidelines for predicting the Dietary Intake of Pesticide Residues held in York, the UK, in 1995, the 1996 Meeting has extended its estimations of residues to include calculations of the median residues found in supervised trials (STMRs) in order to provide a basis for the estimation of the dietary intake of the pesticides reviewed. The estimated STMRs are included in the Table of ADIs and MRLs. Further details of the response of the Meeting to the York Consultation are given in Section 2.2.1 of this report, and information about an informal workshop held in The Hague, The Netherlands, in April 1996 to consider the implementation of its recommendations by the JMPR in Section 2.2.3. The report of this Workshop is reproduced as Annex III.

Attention is drawn to Section 3.1 of the report of the present Meeting: 'Definition of the residues of fat-soluble compounds'. Residues of such compounds are distinguished in the Table of Recommendations by the parenthetic note '(fat-soluble residue)' on a line below the residue definition.

The following qualifications and abbreviations are used.

* 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.
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 Trial Median Residue (see explanation on previous page).
STMR-P	An STMR for a processed commodity calculated by applying the mean concentration or reduction factor for the process to the STMR calculated for the raw agricultural commodity.
T following ADIs	The ADI is temporary, and due for re-evaluation in the year indicated.
T following MRLs	The MRL is temporary, irrespective of the status of the ADI, until required information has been provided and evaluated.
V following recommendations for commodities of animal origin	The recommendation accommodates veterinary uses.
W in place of an MRL	The previous recommendation is withdrawn.

If a recommended MRL is an amendment, the previous value is also recorded. The absence of a figure in the "Previous" column indicates that the recommendation is the first for the commodity or group concerned.

The Table includes the Codex Classification Numbers (CCNs) of both the compounds and the commodities listed, to facilitate reference to the Guide to Codex Maximum Limits for Pesticide Residues 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 was made to facilitate checking and comparison with the CCPR Tables of MRLs, which are in alphabetical order.

ACCEPTABLE DAILY INTAKES (ADIs), MAXIMUM RESIDUE LIMITS (MRLs) AND SUPERVISED TRIALS MEDIAN RESIDUES (STMRs) [1]

Pesticide (Codex ref. No.)	ADI (mg/kg bw)	Commodity		Recommended MRL or ERL (mg/kg)		STMR (mg/kg)
		CCN	Name	New	Previous	
Acephate	0.03	VB 0400	Broccoli	2	-[1]	0.11
(095)		VB 0041	Cabbages, Head	2	-[1]	0.33
		VB 0404	Cauliflower	2	-[1]	0.11
		VO 0448	Tomato	1	-[1]	0.38
			Tomato, canned			0.19 P[2]
			Tomato, canned juice			0.35 P
			Tomato, bulk paste			1.52 P
			Tomato, canned puree			0.68 P
			Tomato, wet pomace			0.23 P
			Tomato, dry pomace			0.38 P
		Residue	(for MRLs & STMRs): acephate			
		Notes:	[1]Previous recommendation withdrawn by 1994 JMPR [2]STMR-P			
Aldicarb	0.003	VR 0589	Potato	0.5	0.5 T	0.077
(117)			Potato chips			0.056 P[1]
			Potato fries			0.045 P
			Potato, microwaved			0.065 P
			Potato, baked			0.050 P
		Residue	(for MRLs & STMRs): Sum of aldicarb, its sulfoxide and its sulfone, expressed as aldicarb.			
		Notes:	[1]STMR-P			
Bifenthrin	0.02	GC 0654	Wheat	0.5 Po	0.05*	0.255
(178)		CM 0654	Wheat bran, unprocessed	2 PoP	-	0.89 P[1]
		CF 1211	Wheat flour	0.2 PoP	-	0.076 P
		CF 1212	Wheat wholemeal	0.5 PoP	-	0.21 P
		Residue	(for MRLs & STMRs): bifenthrin (fat-soluble residue)			
		Notes:	[1]STMR-P			
Carbaryl** (008)	0.003	Notes:	Previous ADI was 0.01 mg/kg bw. Periodic review was only for toxicology			
Carbofuran** (096)	0.002	Notes:	Previous ADI was 0.01 mg/kg bw. Periodic review was only for toxicology			
Chlorfenvinphos**	0.0005	VB 0400	Broccoli	W	0.05	

[1]See explanation on pp. xv and 617

Pesticide (Codex ref. No.)	ADI (mg/kg bw)	Commodity		Recommended MRL or ERL (mg/kg)		STMR (mg/kg)
		CCN	Name	New	Previous	
(014)		VB 0402	Brussels sprouts	W	0.05	
		VB 0041	Cabbages, head	W	0.05	
		VR 0577	Carrot	W	0.4	
		VB 0404	Cauliflower	W	0.1	
		VS 0624	Celery	W	0.4	
		FC 0001	Citrus fruits	W	1	
		SO 0691	Cotton seed	W	0.05	
		VO 0440	Egg plant	W	0.05	
		VR 0583	Horseradish	W	0.1	
		VA 0384	Leek	W	0.05	
		GC 0645	Maize	W	0.05	
		MM 0095	Meat (from mammals, other than marine mammals)	W	0.2 (fat) V	
		ML 0107	Milk of cattle, goats and sheep	W	0.008 F V	
		VO 0450	Mushrooms	W	0.05	
		VA 0385	Onion, bulb	W	0.05	
		SO 0697	Peanut	W	0.05	
		VR 0589	Potato	W	0.05	
		VR 0494	Radish	W	0.1	
		GC 0649	Rice	W	0.05	
		CM 1205	Rice, polished	W	0.05	
		VR 0497	Swede	W	0.05	
		VR 0508	Sweet potato	W	0.05	
		VO 0448	Tomato	W	0.1	
		VR 0506	Turnip, Garden	W	0.05	
		GC 0654	Wheat	W	0.05	
		Residue (for MRLs & STMRs): chlorfenvinphos, sum of *E*- and *Z*- isomers (fat-soluble residue) Notes: Periodic review was only for residues.				
2,4-D** (020)	0.01	Notes: ADI refers to acid equivalent. Previous ADI was 0.3 mg/kg bw. Periodic review was only for toxicology				
DDT (021)	0.02 (PTDI[1]	MM 0095	Meat (from mammals other than marine mammals)	5 (fat) E	1 (fat) E	
		Residue (for MRLs & STMRs): sum of *p,p*¢DDT, *o,p*¢DDT, *p,p*¢DDE, and *p,p*¢TDE (*p,p*¢ DDD) (fat-soluble residue) Notes: [1]provisional tolerable daily intake. See 1994 report, Section 2.3				
Diazinon[1]	0.002	PO 0840	Chicken, Edible offal of	0.02*	-	0
(022)		PE 0840	Chicken eggs	0.02*	-	0
		PM 0840	Chicken meat	0.02*	-	0
		MM 0814	Goat meat	2 (fat) V	-	0.3 (fat) 0.02 (whole muscle)
		MO 0098	Kidney of cattle, goats, pigs and sheep	0.03 V	-	0.01
		MO 0099	Liver of cattle, goats, pigs and sheep	0.03 V	-	0.01

Pesticide (Codex ref. No.)	ADI (mg/kg bw)	Commodity		Recommended MRL or ERL (mg/kg)		STMR (mg/kg)
		CCN	Name	New	Previous	
		MM 0097	Meat of cattle, pigs and sheep	2 (fat) V	W[1]	0.3 (fat) 0.02 (whole muscle)
		ML 0106	Milks	0.02 F V	W[1]	0.02
	Residue (for MRLs & STMRs): diazinon (fat-soluble residue) Notes: [1]Withdrawal of existing CXL proposed by 1993 JMPR.					
Dimethoate** (027)	0.002	Notes: ADI is for the sum of dimethoate and omethoate expressed as dimethoate. Previous ADI was 0.01 mg/kg bw. Periodic review was only for toxicology				
Disulfoton (074)	0.0003	Acute RfD 0.003 mg/kg bw.				
Dithiocarbamates		AM 0660	Almond hulls	20[1] mb[2], zm	20	
(105)		TN 0660	Almonds	0.1* mb, zm	0.1*	
		TN 0672	Pecan	0.1* T zm	-	
		FP 0009	Pome fruits	5 mz, mt, pb, th, zm	5	
		FS 0012	Stone fruits	7[3] T th, zm	-	
		FB 0275	Strawberry	5 th	-	
		Residue: total dithiocarbamates, determined as CS_2 evolved during acid digestion and expressed as mg CS_2/kg. Notes: MRLs refer to total residues from the use of any or each of the groups of dithiocarbamates. [1] The estimated temporary maximum residue level for dithiocarbamates arising from the use of ziram is 10 mg/kg, but the current draft MRL of 20 mg/kg recommended by the 1993 JMPR should be maintained to accommodate uses of maneb. [2] Based on trials with mb maneb, mz mancozeb, mt metiram, pb propineb, th thiram, zm ziram. Underlined compounds are those on which estimates of maximum residue levels are mainly based. [3] The estimated maximum residue level for dithiocarbamates arising from the use of thiram on plums and cherries is 1 mg/kg, but a TMRL of 7 mg/kg is recommended to accommodate uses of ziram on stone fruits.				
Fenarimol	0.01	AB 0226	Apple pomace,dry	5	5 T	
(192)		VS 0620	Artichoke, Globe	0.1	0.1 T	
		FI 0327	Banana	0.2	0.2 T	
		MO 1280	Cattle, kidney	0.02*	0.02* T	
		MO 1281	Cattle, liver	0.05	0.05 T	
		MM 0812	Cattle meat	0.02*	0.02* T	
		FS 0013	Cherries	1	1 T	
		DF 0269	Dried grapes (= Currants, Raisins and Sultanas)	0.2	0.2 T	
		FB 0269	Grapes	0.3	0.3 T	
		DH 1100	Hops, dry	5	-	
		VC 0046	Melons, except Watermelon	0.05	0.05 T	
		FS 0247	Peach	0.5	0.5 T	
		TN 0672	Pecan	0.02*	0.02* T	
		VO 0445	Peppers, Sweet	0.5	0.5 T	
		FP 0009	Pome fruits	0.3	0.3 T	
		FB 0275	Strawberry	1	1 T	

Pesticide (Codex ref. No.)	ADI (mg/kg bw)	Commodity		Recommended MRL or ERL (mg/kg)		STMR (mg/kg)
		CCN	Name	New	Previous	
	Residue (for MRLs & STMRs): fenarimol					
Ferbam** (Dithiocarbamates, 105)	0.003	Notes: group ADI for ferbam and ziram. Previous ADI was 0.02 mg/kg bw, also for ferbam and ziram.				
Flumethrin* (195)	0.004	MM 0812	Cattle meat	0.2 (fat)[1] V	-	0.01 (fat) 0.005 (whole muscle)
		ML 0812	Cattle milk	0.05 F V	-	0.01
			Honey	0.005*	-	0.005
	Residue (for MRLs & STMRs): flumethrin (fat-soluble residue) Notes: [1]maximum residue in whole meat (muscle) reflecting approved uses was 0.01 mg/kg. Recommended MRL is on carcase fat basis.					
Haloxyfop	0.0003	AL 1021	Alfalfa forage (green)	W	Prov.[1]	
(194)		FI 0327	Banana	0.05*	Prov.[1]	0
		MO 0812	Cattle, Edible offal of	W	Prov.[1]	
		MF 0812	Cattle fat	W	Prov.[1]	
		MM 0812	Cattle meat	W	Prov.[1]	
		ML 0812	Cattle milk	W	Prov.[1]	
		FM 0812	Cattle milk fat	W	Prov.[1]	
		PO 0840	Chicken, Edible offal of	0.1	Prov.[1]	0.01
		PE 0840	Chicken eggs	0.01*	Prov.[1]	0.01
		PM 0840	Chicken meat	0.01*	Prov.[1]	0.01
		FC 0001	Citrus fruits	0.05*	Prov.[1]	0
		SO 0691	Cotton seed	0.2	Prov.[1]	0.09
		OC 0691	Cotton seed oil, crude	0.5	Prov.[1]	0.1 P[2]
		AM 1051	Fodder beet	0.3	Prov.[1]	0.02
		AV 1051	Fodder beet leaves or tops	W	Prov.[1]	
		FB 0269	Grapes	0.05*	Prov.[1]	0
		SO 0697	Peanut	0.05	Prov.[1]	0.03
		VP 0063	Peas (pods and succulent = immature seeds)	0.2	-	0.02
		FP 0009	Pome fruits	0.05*	Prov.[1]	0
		VD 0070	Pulses (dry)	0.2	Prov.[1]	0.03
		VR 0589	Potato	0.1	Prov.[1]	0.04
		SO 0495	Rape seed	2	Prov.[1]	0.17
			Rape seed meal			0.15 P
		OC 0495	Rape seed oil, crude	5	Prov.[1]	0.36 P
		OR 0495	Rape seed oil, edible	5	Prov.[1]	0.28 P
		CM 1206	Rice bran, unprocessed	0.02*	Prov.[1]	0.02 P
		CM 0649	Rice, husked	0.02*	Prov.[1]	0
		CM 1205	Rice, polished	0.02*	Prov.[1]	0
			Soya bean			0.03 (Pulses (dry))
			Soya bean meal			0.03 P
		OC 0541	Soya bean oil, crude	0.2	Prov.[1]	0.02 P
		OR 0541	Soya bean oil, refined	0.2	Prov.[1]	0.02 P
		VR 0596	Sugar beet	0.3	Prov.[1]	0.02

Pesticide (Codex ref. No.)	ADI (mg/kg bw)	Commodity		Recommended MRL or ERL (mg/kg)		STMR (mg/kg)
		CCN	Name	New	Previous	
		AV 0596	Sugar beet leaves or tops	W	Prov.[1]	
			Sugar beet pressed pulp			0.008 P
			Sugar, refined			0.002 P
		SO 0702	Sunflower seed	0.2	Prov.[1]	0.05
	Residue (for MRLs & STMRs): haloxyfop esters, haloxyfop and its conjugates expressed as haloxyfop Notes: [1]Provisional estimates of maximum residue levels were made by the 1995 JMPR, but were not recommended for use as MRLs. [2]STMR-P					
Maleic hydrazide** (102)	0.3	Notes: Previous ADI was 5 mg/kg bw. Periodic review was only for toxicology				
Methamidophos	0.004	VB 0041	Cabbage, Head	0.5	-[1]	0.01
(100)		VB 0404	Cauliflower	0.5	-[1]	0.01
		FS 0247	Peach	1	-[1]	0.16
			Peach, washed fruit			0.10
			Peach, juice (100% basis)			0.11 P[2]
			Peach, jam			0.10 P
			Peach, canned fruit			0.08 P
		VO 0448	Tomato	1	-[1]	0.12
	Residue (for MRLs & STMRs): methamidophos Notes: [1]Withdrawn by 1994 JMPR [2]STMR-P Recommended MRLs are based on residues from the use of methamidophos or acephate					
Mevinphos** (053)	0.0008	Notes: Acute RfD 0.003 mg/kg bw. Previous ADI was 0.0015 mg/kg bw. Periodic review was only for toxicology				
Phorate (112)	0.0005	Notes: Previous ADI confirmed				
Propoxur	0.02	VL 0482	Lettuce, Head	0.5	3	
(075)		VR 0589	Potato	0.02*	0.1*	
	Residue (for MRLs): propoxur					
Tebufenozide*	0.02	FB 0269	Grapes	0.5	-	0.12
(196)		FP 0009	Pome fruits	1	-	0.16
		CM 0649	Rice, husked	0.1	-	0.03
		TN 0678	Walnut	0.05	-	0.003
			Apple pomace, wet			0.4 P[1]
			Apple juice			0.02 P
			Apple puree			0.04 P
			Grape pomace, wet			0.36 P
			Wine			0.03 P
	Residue (for MRLs & STMRs): tebufenozide (fat-soluble residue) Notes: [1]STMR-P					
Teflubenzuron*	0.01	VB 0402	Brussels sprouts	0.5	-	0.21
(190)		VB 0041	Cabbages, Head	0.2	-	0.05
		FS 0014	Plums (including Prunes)	0.1	-	0.04
		FP 0009	Pome fruits	1	-	0.48

Pesticide (Codex ref. No.)	ADI (mg/kg bw)	Commodity		Recommended MRL or ERL (mg/kg)		STMR (mg/kg)
		CCN	Name	New	Previous	
		VR 0589	Potato	0.05*	-	0
	Residue (for MRLs & STMRs): teflubenzuron (fat-soluble residue) Notes: First evaluation of residue and analytical aspects. Toxicology was evaluated in 1994.					
Thiram**	0.01		Apple juice			0.55 P[1]
(Dithiocarbamates, 105)			Apple pomace, wet			1.9 P
			Apple pomace, dry			6.93 P
		FS 0013	Cherries	1	-	0.72
		FS 0014	Plums (including Prunes)	1	-	0.72
		FP 0009	Pome fruits	5	5	1.9
		FB 0275	Strawberry	5	-	2.1
	Residue for MRLs: see dithiocarbamates for STMRs: thiram Notes: [1]STMR-P Periodic review was only for residues.					
Ziram**	0.003	AM 0660	Almond hulls	10 T	20	10.6
(Dithiocarbamates,		TN 0660	Almonds	0.1* T	0.1*	0.04
105)			Apple juice			0.204 P
			Apple pomace, wet			2.81 P
			Apple pomace, dry			3.82 P
		TN 0672	Pecan	0.1* T	-	0.04
		FP 0009	Pome fruits	5 T	5	2.1
		FS 0012	Stone fruits	7 T	-	2.2
		Residue for MRLs: see dithiocarbamates for STMRs: ziram Notes: group ADI for ferbam and ziram. Previous ADI was 0.02 mg/kg bw, also for ferbam and ziram.				

ANNEX II

PREVIOUS FAO AND WHO DOCUMENTS

1. FAO/WHO. 1962 — Principles governing consumer safety in relation to pesticide residues. Report of a meeting of a WHO Expert Committee on Pesticide Residues held jointly with the FAO Panel of Experts on the Use of Pesticides in Agriculture. FAO Plant Production and Protection Division Report, No. PL/1961/11; WHO Technical Report Series, No. 240.

2. FAO/WHO. 1964 — Evaluations of the toxicity of pesticide residues in food. Report of a Joint Meeting of the FAO Committee on Pesticides in Agriculture and the WHO Expert Committee on Pesticide Residues. FAO Meeting Report, No. PL/1963/13; WHO/Food Add./23.

3. FAO/WHO. 1965a — Evaluations of the toxicity of pesticide residues in food. Report of the Second Joint Meeting of the FAO Committee on Pesticides in Agriculture and the WHO Expert Committee on Pesticide Residues. FAO Meeting Report, No. PL/1965/10; WHO/Food Add./26.65.

4. FAO/WHO. 1965b — Evaluations of the toxicity of pesticide residues in food. FAO Meeting Report, No. PL/1965/10/1; WHO/Food Add./27.65.

5. FAO/WHO. 1965c — Evaluation of the hazards to consumers resulting from the use of fumigants in the protection of food. FAO Meeting Report, No. PL/1965/10/2; WHO/Food Add./28.65.

6. FAO/WHO. 1967a — Pesticide residues in food. Joint report of the FAO Working Party on Pesticide Residues and the WHO Expert Committee on Pesticide Residues. FAO Agricultural Studies, No. 73; WHO Technical Report Series, No. 370.

7. FAO/WHO. 1967b — Evaluation of some pesticide residues in food. FAO/PL:CP/15; WHO/Food Add./67.32.

8. FAO/WHO. 1968a — Pesticide residues. Report of the 1967 Joint Meeting of the FAO Working Party and the WHO Expert Committee. FAO Meeting Report, No. PL:1967/M/11; WHO Technical Report Series, No. 391.

9. FAO/WHO. 1968b — 1967 Evaluations of some pesticide residues in food. FAO/PL:1967/M/11/1; WHO/Food Add./68.30.

10. FAO/WHO. 1969a — Pesticide residues in food. Report of the 1968 Joint Meeting of the FAO Working Party of experts on Pesticide Residues and the WHO Expert Committee on Pesticide Residues. FAO Agricultural Studies, No. 78; WHO Technical Report Series, No. 417.

11. FAO/WHO. 1969b — 1968 Evaluation of some pesticide residues in food. FAO/PL:1968/M/9/1; WHO/Food Add./69.35.

12. FAO/WHO. 1970a Pesticide residues in food. Report of the 1969 Joint Meeting of the FAO Working Party of experts on Pesticide Residues and the WHO Expert Committee on Pesticide Residues. FAO Agricultural Studies, No. 84; WHO Technical Report Series, No. 458.

13. FAO/WHO. 1970b 1969 Evaluation of some pesticide residues in food. FAO/PL:1969/M/17/1; WHO/Food Add./70.38

14. FAO/WHO. 1971a Pesticide residues in food. Report of the 1970 Joint Meeting of the FAO Working Party of experts on Pesticide Residues and the WHO Expert Committee on Pesticide Residues. FAO Agricultural Studies, No. 87; WHO Technical Report Series, No. 474.

15. FAO/WHO. 1971b 1970 Evaluation of some pesticide residues in food. AGP:1970/M/12/1; WHO/Food Add./71.42.

16. FAO/WHO. 1972a Pesticide residues in food. Report of the 1971 Joint Meeting of the FAO Working Party of experts on Pesticide Residues and the WHO Expert Committee on Pesticide Residues. FAO Agricultural Studies, No. 88; WHO Technical Report Series, No. 502.

17. FAO/WHO. 1972b 1971 Evaluation of some pesticide residues in food. AGP:1971/M/9/1; WHO Pesticide Residues Series, No. 1.

18. FAO/WHO. 1973a Pesticide residues in food. Report of the 1972 Joint Meeting of the FAO Working Party of experts on Pesticide Residues and the WHO Expert Committee on Pesticide Residues. FAO Agricultural Studies, No. 90; WHO Technical Report Series, No. 525.

19. FAO/WHO. 1973b 1972 Evaluation of some pesticide residues in food. AGP:1972/M/9/1; WHO Pesticide Residues Series, No. 2.

20. FAO/WHO. 1974a Pesticide residues in food. Report of the 1973 Joint Meeting of the FAO Working Party of experts on Pesticide Residues and the WHO Expert Committee on Pesticide Residues. FAO Agricultural Studies, No. 92; WHO Technical Report Series, No. 545.

21. FAO/WHO. 1974b 1973 Evaluation of some pesticide residues in food. FAO/AGP/1973/M/9/1; WHO Pesticide Residues Series, No.3.

22. FAO/WHO. 1975a Pesticide residues in food. Report of the 1974 Joint Meeting of the FAO Working Party of experts on Pesticide Residues and the WHO Expert Committee on Pesticide Residues. FAO Agricultural Studies, No. 97; WHO Technical Report Series, No. 574.

23. FAO/WHO. 1975b 1974 Evaluation of some pesticide residues in food. FAO/AGP/1974/M/9/11; WHO Pesticide Residues Series, No.4.

24. FAO/WHO. 1976a Pesticide residues in food. Report of the 1975 Joint Meeting of the FAO Working Party of experts on Pesticide Residues and the WHO Expert Committee on Pesticide Residues. FAO Plant Production and Protection Series, No.1; WHO Technical Report Series, No. 592.

25. FAO/WHO. 1976b 1975 Evaluation of some pesticide residues in food. AGP:1975/M/13; WHO Pesticide Residues Series, No. 5.

26. FAO/WHO. 1977a Pesticide residues in food. Report of the 1976 Joint Meeting of the FAO Panel of Experts on Pesticide Residues and the Environment and the WHO Expert Group on Pesticide Residues. FAO Food and Nutrition Series, No. 9; FAO Plant Production and Protection Series, No. 8; WHO Technical Report Series, No. 612.

27. FAO/WHO. 1977b 1976 Evaluation of some pesticide residues in food. AGP:1976/M/14.

28. FAO/WHO. 1978a Pesticide residues in food - 1977. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 10 Rev.

29. FAO/WHO. 1978b Pesticide residues in food: 1977 evaluations. FAO Plant Production and Protection Paper 10 Sup.

30. FAO/WHO. 1979a Pesticide residues in food - 1978. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 15.

31. FAO/WHO. 1979b Pesticide residues in food: 1978 evaluations. FAO Plant Production and Protection Paper 15 Sup.

32. FAO/WHO. 1980a Pesticide residues in food - 1979. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 20.

33. FAO/WHO. 1980b Pesticide residues in food: 1979 evaluations. FAO Plant Production and Protection Paper 20 Sup.

34. FAO/WHO. 1981a Pesticide residues in food - 1980. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 26.

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ANNEX III

Report of an informal workshop on data evaluation in the estimation of dietary intake of pesticide residues for the JMPR

INTRODUCTION

A Joint FAO/WHO Consultation on Guidelines for predicting the Dietary Intake of Pesticide Residues was held in York, United Kingdom from 2-6 May 1995. The main objectives of the Consultation were to review the existing guidelines and to recommend feasible approaches for improving the reliability and accuracy of methods for predicting dietary intake of pesticide residues. The final published report of this Consultation[2] became available in February 1996.

An informal Workshop was convened in the Hague, Netherlands from 11th-12th April 1996. Dr W. H. van Eck, of the Netherlands Ministry of Health, Welfare and Sport served as chairman. The Workshop had been arranged at the request of the FAO Panel members in order to consider the consequences of the recommendations of the York Consultation for individual reviewers as well as for the JMPR.

The focus of the Workshop was on the issues relating to the reviews of residue data undertaken by the FAO Panel members.

A list of participants is given. The participants considered a number of working examples on quintozene, dithiocarbamates, parathion-methyl and fenpropimorph, which illustrated issues of interest to the FAO Panel.

OBJECTIVES

The chairman explained that the implementation of the York consultation recommendations would have practical consequences for the way the FAO Panel members carried out their evaluations, how those data would be presented and how consumer risk assessments would be carried out by the JMPR. Guidance was needed for the FAO Panel members as to how recommendations are to be implemented. In addition, criteria need to be established in order to ensure consistency and transparency in the work of the FAO Panel.

The Workshop focused mainly on practical considerations of the application of the York consultation recommendations to the work of the FAO Panel. Discussion centred on the following issues:

- the criteria for the selection of residues trials data used to calculate the Supervised Trials Median Residue (STMR) level.
- the presentation in the JMPR monographs of intake related information (eg. median residue levels).
- the approach for dealing with residues at the limit of determination (LOD), also referred to as the limit of quantitation (LOQ).
- practical considerations of the cases where the residue definition for consumer risk assessment is different from that recommended for enforcement
- evaluation of data on edible portion and processing (combined supervised trials data with

[2] 'Recommendations for the revision of the guidelines for predicting dietary intake of pesticide residues', Report of a FAO/WHO Consultation; World Health Organisation 1995.

processing information)

- .identification of appropriate residue values for acute intake assessments

Guidelines were developed in order to give guidance to the FAO Panel reviewers. In addition, a few general recommendations were made. The Workshop recognised that additional guidelines will need to be developed by the JMPR in the future, as experience is gained by the reviewers.

GUIDANCE TO THE FAO PANEL REVIEWERS ON THE IMPLEMENTATION OF THE YORK CONSULTATION RECOMMENDATIONS

The Workshop recommended that:

Comparability

Residues data from countries are evaluated against the GAP in the country of the trials or a neighbouring country with similar climate and cultural practices.

In identifying the STMR, the trials values selected should be comparable with the maximum registered use (ie. maximum application rate, maximum number of treatments, minimum PHI) on which the MRL is based.

In establishing comparability of uses in the residue trials to the maximum registered use, the application rates in the trials should generally be no more than ± 25 to 30% of the maximum application rate. Deviations from this should be explained in the appraisal. Similarly, ± 25 to 30% should also be used as a guide for establishing comparability of PHI; however, in this case the latitude of acceptable PHIs will also depend on the rate of decline of residues of the compound under evaluation. Consideration as to whether the number of treatments reported in trials are comparable to the registered maximum number of treatments will depend on the persistence of the compound and the interval between applications. Nevertheless, when a large number of treatments are made in the trials (more than 5 or 6) the residue level should be considered very little influenced by further treatments unless the compound is persistent or the treatments are made with unusually short intervals.

In establishing comparability of residue trials data in which more than one parameter (i.e application rate, number of treatments or PHI) deviate from the maximum registered use, consideration should be given to the combination effect on the residue value which may lead to an underestimation or overestimation of the STMR. For example, a trial result should not normally be selected for the estimation of the STMR if both the application rate is lower (perhaps 0.75 kg/ha in the trial; 1kg ai/ha GAP) than the maximum rate registered and the PHI is longer (perhaps 18 days in the trial, 14 days GAP) than the minimum registered PHI, since these parameters would combine to underestimate the residue. When results are selected for the estimation of STMRs, despite combination effects, the reasons should be explained in the appraisal.

If the residue value arising from a use considered comparable with the maximum registered use is lower than another residue value from the same trial which is within GAP, then the higher residue value should be selected in identifying the STMR. For example, if the GAP specified a minimum PHI of 21 days and the residue levels in a trial reflecting GAP were 0.7, 0.6 and 0.9 mg/kg at 21, 28 and 35 days respectively, then the residue value of 0.9 mg/kg would be selected.

Trials with more than one residue value

In identifying the STMR only one data point should be take from each trial (ie. site location)

Where several residue values have been reported from replicate plots from a single trial (ie. site location) the highest residue should be selected for the purpose of identifying the STMR.

Where several residue values have been reported from replicate analyses of the same field sample taken from a single trial (ie. site location) the mean residue should be selected for the purpose of identifying

the STMR.

Rounding of results

In identifying the STMR from a residue trial the actual residue value should be used in the estimation of dietary intake without rounding up or down. This would even be the case where the actual results were below the practical limit of determination considered appropriate for enforcement purposes. Rounding of residue values is inappropriate since the STMRs are used at an intermediate stage in the dietary intake calculation.

Residue definition

The WHO Panel consider routinely indicating in their evaluations which metabolites should be included in the dietary risk assessment.

If it is recommended that the residue definition for the risk assessment is different from that for enforcement, then this is clearly stated in the appraisal.

Close communication should be established between the FAO Panel reviewers and the respective reviewers on the Toxicological and Environmental Groups, on questions such as which metabolites are of toxicological significance, prior to the JMPR meeting.

In tabulating the residue trials data the FAO Panel reviewer should indicate the levels of relevant metabolites separately from those of the parent compound, but in a way which would allow subsequent combination, in order to ensure that changes in the residue definition can be accommodated at the JMPR meeting.

In those cases where it is not possible to finalise the risk assessment at the JMPR (September, year 1) usually because of a change in residue definition, then the MRLs would still be recommended to the CCPR (by way of Codex circular letter for comment at step 3) and the compound would be rediscussed at the following years JMPR meeting (September, year 2). The recommended MRLs together with the conclusion of the risk assessment would be available for the next CCPR (April, year 3).

If two compounds, for which STMRs can be calculated, produce the same analyte in compliance monitoring (eg. CS_2 for dithiocarbamates) it is possible to separate the intake assessments, if required, because the intake assessment is no longer based on the MRL but is based on residue data specific to the individual compounds.

Combining of populations of data for the calculation of STMRs

In identifying the STMR, residue data reflecting different countries GAPs would normally be combined. However, if the trials data reflecting different countries GAPs appear to give rise to different populations of data then these data sets should not be combined. In these cases the STMR should be calculated from the population(s) of data which is (are) driving the MRL. In deciding whether the results of trials reflecting different countries GAPs give rise to different populations of residues data, the size of the database reflecting the different countries GAPs should be taken into account.

Residues below the limit of determination

That as a general rule, where all residue trials data are <LOD, the STMR would be assumed to be at the LOD, unless there is scientific evidence that residues are "essentially zero". Such supporting evidence would include residues from related trials at shorter PHIs, exaggerated, but related, application rates or a greater number of applications, expectations from metabolism studies or data from related commodities.

Where there are two or more sets of trials with different LODs, and no determinable residues have been reported in the trials, then the lowest LOD should normally be used for the purpose of STMR selection (unless the residues can be assumed to be essentially zero as given above). The size of the trials database supporting the lowest LOD value should be taken into account in the decision.

Processing, cooking factors and edible portion residue data

In using data on the effects on residue levels of processing or cooking practices, the mean reduction or concentration factor should be applied to the STMR estimated for the raw agricultural commodity as already described. The STMR value estimated in this way for the processed commodity should be referred to as the STMR-P.

If data are available for the residues in the edible portion of the commodity (eg. banana pulp) then a STMR should be estimated directly using the edible portion residue values from maximum registered use trials (as opposed to using pesticide values for the whole commodity).

Acute dietary intake

The attention of the FAO Panel members is drawn to the recommendation that for the purpose of acute risk assessment the MRL, or the highest residue in the edible portion, should be used in estimating dietary intake.

Estimation of MRLs for products of animal origin

In estimating MRLs for products of animal origin, theoretical feed intakes for domestic animals should be calculated using the STMR for each feed item (derived from supervised trials comparable with the maximum registered use), rather than the MRL, together with the maximum feed incorporation rates. This is in conformity with past JMPR decisions.

Estimation of STMRs for commodity groups

Where there are adequate trials data the STMRs should, in principle, be identified for the individual commodities and these values used for the intake assessment. However, where the MRL has been established for a group of commodities (eg. pome fruit) a single STMR should be calculated for the group of commodities.

Presentation of STMRs in the JMPR monographs and report

The GAP(s) on which trials data have been selected for the purpose of identifying the STMR should be clearly identified in the monographs.

In tabulating trials data in the monographs the reviewer should ensure that in addition to the normal underlining of trials data that are within GAP (and therefore have been used for the MRL evaluation), the single residue values selected for the estimation of the STMR should be double underlined.

Information on the residue values on which the STMR is based should not only be identified in the tabulated trials data (see above) but a list of the residue values selected should be included in the appraisal, in numerical order, with the median residue underlined. Where the residue situation is complex (eg. a number of metabolites to be considered) these data may best be tabulated in the appraisal. In addition, the STMR values should be included in the recommendation table in the appraisal and in Annex 1 of the report.

The range for the rates and PHIs used in the selection of residue values for STMR should be clearly identified in the appraisal (eg. trials data with application rates from 1.8 - 3.0 kg ai/ha have been selected).

RECOMMENDATIONS

The Workshop recommended that:

a) The recommendations of the York Joint FAO/WHO Consultation are implemented in full into the work of the JMPR.

b) The acronym "STMR" be used in the JMPR monographs and report for the Supervised Trials Median Residue level.

c) The FAO Panel identify STMRs routinely for each commodity as part of all future evaluation of compounds in order to facilitate more realistic estimates of long-term dietary intake.

d) The guidance given in section 3 above is used by the FAO Panel reviewers in their evaluations for the 1996 JMPR.

e) The report of the York Consultation be considered by 1996 JMPR together with worked examples that demonstrate the FAO Panel guidance given in section 3.

f) GAP information when submitted by either the manufacturer or member governments, clearly identify which of the rates and PHIs are statutory conditions of use or taken directly from the product label and which are estimates made by the manufacturer or member governments (eg. whether the application rates in kg ai/ha have been calculated from the kg ai/hl application concentrations).

g) The concepts contained in the FAO Panel guidance, as given in section 3, be incorporated into the draft document currently entitled "FAO Guidelines in the evaluation of pesticide residues data and the estimation of the Maximum Residue Limits in Food and Feed".

OTHER CONSIDERATIONS

As a result of the examination of a worked example for STMR estimation, the Workshop noted that significant residues of HCB may result in commodities following applications of quintozene. When quintozene is re-evaluated by the JMPR, consideration should be given to the risk associated with the residues of the impurity HCB.

The WHO informed the Workshop that in revising the Guidelines for the prediction of dietary intake of pesticide residues, they would include hypothetical worked examples of intake calculations in order to give further guidance to member governments.

LIST OF PARTICIPANTS *(in alphabetical order)*

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ANNEX III

Report of an informal workshop on data evaluation in the estimation of dietary intake of pesticide residues for the JMPR

INTRODUCTION

A Joint FAO/WHO Consultation on Guidelines for predicting the Dietary Intake of Pesticide Residues was held in York, United Kingdom from 2-6 May 1995. The main objectives of the Consultation were to review the existing guidelines and to recommend feasible approaches for improving the reliability and accuracy of methods for predicting dietary intake of pesticide residues. The final published report of this Consultation[2] became available in February 1996.

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The focus of the Workshop was on the issues relating to the reviews of residue data undertaken by the FAO Panel members.

A list of participants is given. The participants considered a number of working examples on quintozene, dithiocarbamates, parathion-methyl and fenpropimorph, which illustrated issues of interest to the FAO Panel.

OBJECTIVES

The chairman explained that the implementation of the York consultation recommendations would have practical consequences for the way the FAO Panel members carried out their evaluations, how those data would be presented and how consumer risk assessments would be carried out by the JMPR. Guidance was needed for the FAO Panel members as to how recommendations are to be implemented. In addition, criteria need to be established in order to ensure consistency and transparency in the work of the FAO Panel.

The Workshop focused mainly on practical considerations of the application of the York consultation recommendations to the work of the FAO Panel. Discussion centred on the following issues:

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- the presentation in the JMPR monographs of intake related information (eg. median residue levels).
- the approach for dealing with residues at the limit of determination (LOD), also referred to as the limit of quantitation (LOQ).
- practical considerations of the cases where the residue definition for consumer risk assessment is different from that recommended for enforcement
- evaluation of data on edible portion and processing (combined supervised trials data with

[2] 'Recommendations for the revision of the guidelines for predicting dietary intake of pesticide residues', Report of a FAO/WHO Consultation; World Health Organisation 1995.

processing information)

- identification of appropriate residue values for acute intake assessments

Guidelines were developed in order to give guidance to the FAO Panel reviewers. In addition, a few general recommendations were made. The Workshop recognised that additional guidelines will need to be developed by the JMPR in the future, as experience is gained by the reviewers.

GUIDANCE TO THE FAO PANEL REVIEWERS ON THE IMPLEMENTATION OF THE YORK CONSULTATION RECOMMENDATIONS

The Workshop recommended that:

Comparability

Residues data from countries are evaluated against the GAP in the country of the trials or a neighbouring country with similar climate and cultural practices.

In identifying the STMR, the trials values selected should be comparable with the maximum registered use (ie. maximum application rate, maximum number of treatments, minimum PHI) on which the MRL is based.

In establishing comparability of uses in the residue trials to the maximum registered use, the application rates in the trials should generally be no more than ± 25 to 30% of the maximum application rate. Deviations from this should be explained in the appraisal. Similarly, ± 25 to 30% should also be used as a guide for establishing comparability of PHI; however, in this case the latitude of acceptable PHIs will also depend on the rate of decline of residues of the compound under evaluation. Consideration as to whether the number of treatments reported in trials are comparable to the registered maximum number of treatments will depend on the persistence of the compound and the interval between applications. Nevertheless, when a large number of treatments are made in the trials (more than 5 or 6) the residue level should be considered very little influenced by further treatments unless the compound is persistent or the treatments are made with unusually short intervals.

In establishing comparability of residue trials data in which more than one parameter (i.e application rate, number of treatments or PHI) deviate from the maximum registered use, consideration should be given to the combination effect on the residue value which may lead to an underestimation or overestimation of the STMR. For example, a trial result should not normally be selected for the estimation of the STMR if both the application rate is lower (perhaps 0.75 kg/ha in the trial; 1kg ai/ha GAP) than the maximum rate registered and the PHI is longer (perhaps 18 days in the trial, 14 days GAP) than the minimum registered PHI, since these parameters would combine to underestimate the residue. When results are selected for the estimation of STMRs, despite combination effects, the reasons should be explained in the appraisal.

If the residue value arising from a use considered comparable with the maximum registered use is lower than another residue value from the same trial which is within GAP, then the higher residue value should be selected in identifying the STMR. For example, if the GAP specified a minimum PHI of 21 days and the residue levels in a trial reflecting GAP were 0.7, 0.6 and 0.9 mg/kg at 21, 28 and 35 days respectively, then the residue value of 0.9 mg/kg would be selected.

Trials with more than one residue value

In identifying the STMR only one data point should be take from each trial (ie. site location)

Where several residue values have been reported from replicate plots from a single trial (ie. site location) the highest residue should be selected for the purpose of identifying the STMR.

Where several residue values have been reported from replicate analyses of the same field sample taken from a single trial (ie. site location) the mean residue should be selected for the purpose of identifying

the STMR.

Rounding of results

In identifying the STMR from a residue trial the actual residue value should be used in the estimation of dietary intake without rounding up or down. This would even be the case where the actual results were below the practical limit of determination considered appropriate for enforcement purposes. Rounding of residue values is inappropriate since the STMRs are used at an intermediate stage in the dietary intake calculation.

Residue definition

The WHO Panel consider routinely indicating in their evaluations which metabolites should be included in the dietary risk assessment.

If it is recommended that the residue definition for the risk assessment is different from that for enforcement, then this is clearly stated in the appraisal.

Close communication should be established between the FAO Panel reviewers and the respective reviewers on the Toxicological and Environmental Groups, on questions such as which metabolites are of toxicological significance, prior to the JMPR meeting.

In tabulating the residue trials data the FAO Panel reviewer should indicate the levels of relevant metabolites separately from those of the parent compound, but in a way which would allow subsequent combination, in order to ensure that changes in the residue definition can be accommodated at the JMPR meeting.

In those cases where it is not possible to finalise the risk assessment at the JMPR (September, year 1) usually because of a change in residue definition, then the MRLs would still be recommended to the CCPR (by way of Codex circular letter for comment at step 3) and the compound would be rediscussed at the following years JMPR meeting (September, year 2). The recommended MRLs together with the conclusion of the risk assessment would be available for the next CCPR (April, year 3).

If two compounds, for which STMRs can be calculated, produce the same analyte in compliance monitoring (eg. CS_2 for dithiocarbamates) it is possible to separate the intake assessments, if required, because the intake assessment is no longer based on the MRL but is based on residue data specific to the individual compounds.

Combining of populations of data for the calculation of STMRs

In identifying the STMR, residue data reflecting different countries GAPs would normally be combined. However, if the trials data reflecting different countries GAPs appear to give rise to different populations of data then these data sets should not be combined. In these cases the STMR should be calculated from the population(s) of data which is (are) driving the MRL. In deciding whether the results of trials reflecting different countries GAPs give rise to different populations of residues data, the size of the database reflecting the different countries GAPs should be taken into account.

Residues below the limit of determination

That as a general rule, where all residue trials data are <LOD, the STMR would be assumed to be at the LOD, unless there is scientific evidence that residues are "essentially zero". Such supporting evidence would include residues from related trials at shorter PHIs, exaggerated, but related, application rates or a greater number of applications, expectations from metabolism studies or data from related commodities.

Where there are two or more sets of trials with different LODs, and no determinable residues have been reported in the trials, then the lowest LOD should normally be used for the purpose of STMR selection (unless the residues can be assumed to be essentially zero as given above). The size of the trials database supporting the lowest LOD value should be taken into account in the decision.

Processing, cooking factors and edible portion residue data

In using data on the effects on residue levels of processing or cooking practices, the mean reduction or concentration factor should be applied to the STMR estimated for the raw agricultural commodity as already described. The STMR value estimated in this way for the processed commodity should be referred to as the STMR-P.

If data are available for the residues in the edible portion of the commodity (eg. banana pulp) then a STMR should be estimated directly using the edible portion residue values from maximum registered use trials (as opposed to using pesticide values for the whole commodity).

Acute dietary intake

The attention of the FAO Panel members is drawn to the recommendation that for the purpose of acute risk assessment the MRL, or the highest residue in the edible portion, should be used in estimating dietary intake.

Estimation of MRLs for products of animal origin

In estimating MRLs for products of animal origin, theoretical feed intakes for domestic animals should be calculated using the STMR for each feed item (derived from supervised trials comparable with the maximum registered use), rather than the MRL, together with the maximum feed incorporation rates. This is in conformity with past JMPR decisions.

Estimation of STMRs for commodity groups

Where there are adequate trials data the STMRs should, in principle, be identified for the individual commodities and these values used for the intake assessment. However, where the MRL has been established for a group of commodities (eg. pome fruit) a single STMR should be calculated for the group of commodities.

Presentation of STMRs in the JMPR monographs and report

The GAP(s) on which trials data have been selected for the purpose of identifying the STMR should be clearly identified in the monographs.

In tabulating trials data in the monographs the reviewer should ensure that in addition to the normal underlining of trials data that are within GAP (and therefore have been used for the MRL evaluation), the single residue values selected for the estimation of the STMR should be double underlined.

Information on the residue values on which the STMR is based should not only be identified in the tabulated trials data (see above) but a list of the residue values selected should be included in the appraisal, in numerical order, with the median residue underlined. Where the residue situation is complex (eg. a number of metabolites to be considered) these data may best be tabulated in the appraisal. In addition, the STMR values should be included in the recommendation table in the appraisal and in Annex 1 of the report.

The range for the rates and PHIs used in the selection of residue values for STMR should be clearly identified in the appraisal (eg. trials data with application rates from 1.8 - 3.0 kg ai/ha have been selected).

RECOMMENDATIONS

The Workshop recommended that:

a) The recommendations of the York Joint FAO/WHO Consultation are implemented in full into the work of the JMPR.

b) The acronym "STMR" be used in the JMPR monographs and report for the Supervised Trials Median Residue level.

c) The FAO Panel identify STMRs routinely for each commodity as part of all future evaluation of compounds in order to facilitate more realistic estimates of long-term dietary intake.

d) The guidance given in section 3 above is used by the FAO Panel reviewers in their evaluations for the 1996 JMPR.

e) The report of the York Consultation be considered by 1996 JMPR together with worked examples that demonstrate the FAO Panel guidance given in section 3.

f) GAP information when submitted by either the manufacturer or member governments, clearly identify which of the rates and PHIs are statutory conditions of use or taken directly from the product label and which are estimates made by the manufacturer or member governments (eg. whether the application rates in kg ai/ha have been calculated from the kg ai/hl application concentrations).

g) The concepts contained in the FAO Panel guidance, as given in section 3, be incorporated into the draft document currently entitled "FAO Guidelines in the evaluation of pesticide residues data and the estimation of the Maximum Residue Limits in Food and Feed".

OTHER CONSIDERATIONS

As a result of the examination of a worked example for STMR estimation, the Workshop noted that significant residues of HCB may result in commodities following applications of quintozene. When quintozene is re-evaluated by the JMPR, consideration should be given to the risk associated with the residues of the impurity HCB.

The WHO informed the Workshop that in revising the Guidelines for the prediction of dietary intake of pesticide residues, they would include hypothetical worked examples of intake calculations in order to give further guidance to member governments.

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